Macroleif and chemical composition of a surface of details made of stainless steels after electrochemical polishing in a non-stationary electrolysis mode

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Abstract. This paper is dedicated to study of the macroleif and the chemical composition of the surface of the details made of stainless steel after the electrochemical polishing in the nonstationary electrolysis mode with usage of a scanning electron microscope JEOL JSM-6510A. We presented images of the surface of samples under test and spectrograms of quantity distribution of chemical elements in their near surface layers after a processing in different technological modes (process phases). We made a calculation of sizes of the surface irregularities and an average number of chemical elements distribution in the near surface layers of the samples under test. We presented diagrams of a dependence of the surface irregularities sizes of the samples under test on the technological modes of the processing.

1. Introduction
Different ways of physical-technical processing particularly different technologies of the electrochemical polishing are used for a final processing of details surface for items made of stainless steels of aviation, oil, gas and medical industry. Traditional ways of the electrochemical polishing are known, but the general disadvantage of their usage is a necessity of conduction of special programs connected with provision of people and environmental safety [1].

A significant local irritation of an anode surface in an electric current switch was marked in papers of number of scientists dedicated to a technology of metals and alloys heat in electrolytic plasm with usage of a direct-current source as one of negative particularities of this process (II stage of the process) [2, 3, 4]. Having made a hypothesis about a possibility of usage of this effect with positive purposes for a development of a new way of details surface polishing and having conducted a complex of studies, a group of scientists from Tyumen state oil and gas university laboratory of functional coverages developed and patented a way of the electrochemical polishing of items made of nickel-chromium steels in the mode of non-stationary electrolysis with usage of a two-component environmental friendly electrolyte [5].

Definite problems connected with technological heredity because of influence of an electrochemical process on details surface often appear during usage of electrochemical polishing technologies in the industry. It is generally recognized that the process of electrochemical polishing can influence on condition of the surface of the processed details and particularly change the chemical composition of the surface [6]. Besides, as the process of final processing of the detail surface electrochemical polishing cannot always eliminate such defects after previous processings as deep
scratches, cracks, shears and others. That is why study of the macrorelief and chemical composition of the details dependence on current parameters and the basic phases of the process carrying in an interelectrode gap is an important task for application of correct processing modes.

2. **Object and methods of researches**

Considering that a specificity of physical and mechanical properties of stainless steels creates definite difficulties for the research of the chemical composition of the modified surface of details and technical capabilities of surface analyzers and profile recorders often do not give an opportunity to examine the macrorelief (topography) in details, the authors of the article offered to use scanning electronic microscope JEOL JSM-6510A (SEM).

The hardware and software suite of the microscope enables to receive magnified views of objects of subnanometer sizes, estimate quality and quantity composition of elements with a different error for elements which are presented in high and low concentrations, build maps of surface distribution of elements.

Formation of full view on the object under study appears as a result of a point-by-point scanning of different objects on its surface by a focused beam of electrons with a consequent detection of reflected electrons and usage of X ray energy-dispersive microanalysis (energy-dispersive analyzer).

We used a method of their sizes control on SEM shots in X and Y coordinates and by means of a micrometric bar for the analysis of micro-irregularities on the surfaces of the samples under study. We used surface analyzer SurfTest Extreme SV-3000CNC in order to determine a height of the surface irregularity of the samples under study.

We exposed samples with dimensions of 10x10x2 mm made of stainless steel 08X22H6T after the electrochemical polishing in an aqua solution of sodium carbonite (Na₂CO₃) in different technological modes and a non-treated (initial) sample in a supply condition to scrutiny. Preliminarily all the objects under study were cleaned in a hypersonic liquid installation.

In order to obtain more precise and valid data about an element composition boosting voltage on an emissive tungsten pre-centered cathode was within the range of 20-30 kV.

3. **Research of the chemical composition and topography of a surface**

As a result of the research we received images of the surface of the samples under study with a different degree of magnifying and spectrograms of quantity distribution of chemical elements in their near surface layers after the processing in different technological modes (the process phases).

3.1. **Research of chemical composition of a surface**

For the purposes of conduction of a comparison analysis of the samples chemical composition after the electrochemical polishing first we obtained spectrograms of quantity distribution of chemical elements in the near surface layers for the non-processed sample (see figure 1). Herewith capabilities of software and hardware complex SEM allowed receiving three spectrograms in one shot: 001 – for a border of wedges; 002 – for a basic surface and 003 – for a border of a cavity on the sample surface.

Considering that in order to reach the target values in the process of electrochemical polishing of stainless steels in the non-stationary electrolysis mode it is recommended to use the phase of steady electro-hydrodynamical condition of a gas-steam sheath [7]. Only the spectrograms of the samples processed in range of direct current voltage of 180-340 V are given below. Additionally during the research conduction we took into consideration the fact that if the chemical composition of the samples surface can change under the influence of electrochemical anode process, than these changes should appear at most at maximum exposure time of these processes, i.e. maximum time of processing. That is why the samples were exposed to the spectral analysis on SEM after the electrochemical polishing within 10 minutes.
Figure 1. Spectrograms of quantity distribution of chemical elements in the near surface layers of the sample under study (The initial sample in a supply condition).

The spectrograms of quantity distribution of chemical elements in the near surface layers of the samples after the electrochemical polishing under voltage on an electrolytic cell of 180 V (see figure 2), 220 V (see figure 3), 260 V (see figure 4) and 340 V (see figure 5) are given below. The preliminary visual analysis of the spectrograms by the value of peaks and energy of reflected electrons of every chemical element allow drawing of conclusion that the chemical composition of the samples surface after the electrochemical polishing within the voltages range of 180-300 V is almost identical to the chemical composition of the initial sample in the supply condition according to the basic chemical elements.

Figure 2. Spectrograms of quantity distribution of chemical elements in the near surface layers of the surface under study (U = 180 V, T = 10 min).
Figure 3. Spectrograms of quantity distribution of chemical elements in the near surface layers of the sample under study (U = 220 V, T = 10 min).

Figure 4. Spectrograms of quantity distribution of chemical elements in the near surface layers of the sample under study (U = 260 V, T = 10 min).

An exclusion is the spectrogram of the surface of the sample which was processed under voltage of 340 V (see figure 5) where peaks of other chemical elements including oxygen are marked on the basic surface of the sample (see spectrogram 009 figure 5). Presence of oxygen on the spectrogram confirms information about a fact that oxide and hydrooxides coagulates can be formed on the anode surface during an unstable electro-hydrodynamical mode of the gas-steam sheath [7, 8].
3.2. Analysis of chemical composition of a surface

An accurate analysis of the quantity distribution of chemical elements in the near surface layers of the samples under study was conducted in an automatic mode by means of software and hardware complex SEM according to standard practices of the X ray energy-dispersive micro-analysis and was printed as tables of ready values of each spectrogram.

For convenience of the general analysis of the spectrograms averaged data of all measured samples is accumulated in the single table (see table 1) and presented as a diagram of the dependence of each of basic chemical elements on the sample surface on the voltage in the electrolytic cell (see figure 6).

Table 1. Mass fraction of chemical elements on a surface of exemplars as a percentage depending on voltage in the electrolytic cell.

|       | U=0V | U=60V | U=100V | U=140V | U=180V | U=220V | U=260V | U=300V | U=340V |
|-------|------|-------|--------|--------|--------|--------|--------|--------|--------|
| Fe    | 66,42| 58,50 | 49,71  | 46,66  | 66,18  | 66,37  | 66,24  | 66,38  | 56,37  |
| Cr    | 21,99 | 22,33 | 24,20  | 18,82  | 22,41  | 22,52  | 22,58  | 22,78  | 22,68  |
| Ni    | 7,79  | 6,48  | 5,14   | 5,79   | 7,82   | 7,39   | 7,82   | 7,74   | 7,96   |
| Ti    | 0,74  | 1,07  | 1,44   | 0,46   | 0,73   | 0,69   | 0,61   | 0,58   |        |
| O₂    | 0,00  | 14,00 | 21,00  | 24,00  | 0,00   | 0,00   | 0,00   | 0,00   | 1,50   |

Figure 5. Spectrograms of the quantity distribution of chemical elements in the near surface layers of the sample (U = 340 V, T = 10 min).
We can draw a firm conclusion from the analysis of the verified data presented in table 1 and figure 6 that the chemical composition of the samples surface after the electrochemical polishing in voltages range of 180-300 V of all chemical elements has only fractional deviations from the chemical composition of the initial sample (0-7%) and it actually stays within the limit of an acceptable error of SEM.

For a range of voltages in the electrolytic cell from 0 to 140 V there appears a significant increase of a percentage rating of oxygen and a corresponding decrease of a percentage rating of ferrum. And this fact confirms the information about an activation of oxidative processes on the surface of the anode under these modes of processing and primarily about ferrum oxidation [3, 4]. Here the unstable electro-hydrodynamical mode of the gas-steam sheath leads to a periodical implosion of the gas-steam sheath and a direct contact of the electrolyte and anode happens consequently, and that leads to the oxidation of its surface by means of electrochemical processes. That is the reason, why it is recommended to use the technological modes of processing within the range of voltages of more than 160 V [7] for the electrochemical polishing of the details made of stainless steels.

Besides, results of the spectral analysis of the samples showed that the electrochemical polishing within the range of voltages of more than 300 V also leads to the significant change of chemical composition of their surface concerning ferrum and titanium.

3.3. Analysis of topography of a surface

The analysis of topography of the surface of the samples under study was conducted by means of a method of a fixation of sizes and mutual disposition of micro-irregularities on SEM shots by X and Y coordinates visually and by means of the micrometric bar. Herewith with the purposes of succession of the research results and expansion of comprehension of physical-chemical essence of the details electrochemical polishing process in the nonstationary electrolysis mode SEM shot fixed the same surface areas of the samples where the spectral analysis of their chemical composition was held. Moreover, all the samples exposed to the electrochemical processing within the range of direct current of 0-340 V and with the time variation from 2 to 10 minutes were exposed to fixation by means of SEM and the preliminary visual analysis. SEM shots analysis showed that macrorelief of “indulation with a step of 25-50 µm” (see figure 7) is clearly of evidence on the surface of samples which were processed in the recommended technological modes within 2-4 mines within the range of voltages of 180-220 V.
This macrorelief can be almost fully deleted (see figure 8) in case of increase of the processing time up to 8-10 minutes and/or voltage in the electrolytic cell to 260-300 V. It is apparent from SEM shot (see figure 8) that the relief of the samples surface after electrochemical polishing in these modes generally has only micro-irregularities (roughness). Herewith separate deep cavities and scratches are observed on the surface of this and other samples. These cavities and scratches result from the previous mechanical processing (in our case – rolling). As a result if SEM shots analysis we can make a conclusion that it is necessary to set a time period more than 10 minutes.

The analysis of SEM shots of the samples processed out of the recommended range of technological modes [7] showed that condition of macrorelief of the surface does not correspond to the
established quality standards. Oxide layers of different form and composition, cavitation segregation of the samples surface, craters and caverns (see figure 9) were observed visually on SEM shots. Herewith the craters size can vary within the range of 5-25 µm. Moreover it is necessary to note that the craters are consequence of the gas-steam sheath by micro-arc blasts or by spark discharges of high power through so called electrolytic bridges [2, 3, 8]. Upon that the author [8] included the electrolytic bridges in his mathematical model as an essential condition of electro-impulse polishing. In this case SEM shot (see figure 9) shows categorically that we can speak only about an electrochemical dimensional processing of a detail surface because as metal removal is obvious than according to the surface condition it is impossible to state that we deal with one of polishing types.

![Figure 9. SEM, U = 260 V, T = 8 min.](image)

We used surface analyzer Surftest Extreme SV-3000CNC for measurement of the height of irregularities of the samples surface under analysis. The received data was processed according to the standard methods and presented in table 2 in short form. On the basis of this data we have built a diagram of dependence of maximum height of the samples surface undulation on voltage in the electrolytic cell and processing time (see figure 10).

**Table 2.** Value of maximum height of undulation on a surface from processing time.

| Processing Time | U=0V | U=60V | U=100V | U=140V | U=180V | U=220V | U=260V | U=300V | U=340V |
|-----------------|------|-------|--------|--------|--------|--------|--------|--------|--------|
| 2 min           | 1,420| 1,382 | 1,245  | 1,364  | 1,124  | 0,483  | 0,285  | 0,205  | 0,245  |
| 4 min           | 1,420| 1,285 | 1,012  | 0,996  | 0,737  | 0,424  | 0,304  | 0,195  | 0,216  |
| 6 min           | 1,420| 1,210 | 1,186  | 0,694  | 0,547  | 0,315  | 0,212  | 0,195  | 0,206  |
| 8 min           | 1,420| 1,073 | 1,045  | 0,463  | 0,214  | 0,115  | 0,084  | 0,075  | 0,135  |
| 10 min          | 1,420| 1,120 | 0,772  | 0,363  | 0,084  | 0,068  | 0,063  | 0,126  | 0,146  |
Figure 10. The dependence of the maximum height of the samples surface undulation on voltage of the electrolytic cell and processing time.

It is following from the analysis of data presented in table 2 and figure 10 that minimal values of macro-irregularities on the surface of the details made of stainless steel 08X22H6T can be received during electrochemical polishing during 8-10 minutes in the steady electro-hydrodynamical mode of the gas-steam sheath within the range of voltages. Polishing during 2-8 minutes within the range of voltages in the electrolytic cell of 260-300 V is also acceptable.

4. General conclusions
Therefore researches of the elemental composition of the samples surface by means of SEM showed that quantity of basic chemical elements in the near surface layers almost does not change after electrochemical polishing.

In order to obtain minimal values of macro-irregularities on the surface of details made of stainless steel 08X22H6T it is necessary to follow recommendations on technological modes for provision of the steady electro-hydrodynamical condition of the gas-steam sheath within voltage range of 180-300 V [7].

Furthermore we can make a conclusion that the shots of the details surface after electrochemical polishing obtained by means of SEM allow to conduct a closer study of the topography up to the finest details which cannot be studied by devices like the surface analyzer or profilograph because of their technical specifics.

References
[1] Papilov L Y 1982 Electrophysical and electrochemical processing of materials (Moscow: Mashinostroeniye) p 400
[2] Lazarenko B R, Duradjee V N, Faktarovitch A A and Bryantsev I V 1970 A research of puncture between metallic and electrolytic electrodes Electronnaya obrabotka materialov 4 18-21
[3] Lazarenko B R, Duradjee V N, Faktarovitch A A and Bryantsev I V 1974 A research of puncture between metallic and electrolytic electrodes Electronnaya obrabotka materialov 3 37-40
[4] Duradjee V N, Bryantsev I V and. Towarkov A K 1978 Research of anode erosion during its exposure to electrolytic plasma Electronnaya obrabotka materialov 5 13-17
[5] Patent 2118412 Russian Federation. MIIP® C 25 F 3/24. A way of electrochemical polishing of items made nickel-chromium steels / Stavyshenko A.S., Nauk P.E., Skifiskiy S.V. Applicant
and owner the patent – Tyumen state oil and gas university. – 97106310/02; claim dated by 
24.04.97; published on 27.08.98, Bulletin № 24. – 11 p.: illustrations

[6] Grilihes S Y 1987 Electrochemical and chemical polishing: Theory and practice. Influence on 
metals properties (Leningrad: Mashinostroeniye, Leningrad branch) p 231

[7] Stavyshenko A S 2015 Research of processes proceeding during polishing of surfaces of details 
made of stainless steels according to technology EPSS-NE Papers of the international 
scientific-technological conferention Oil and gas of Western Siberia vol 4, ed P V Evtin 
(Tyumen: Tyumen oil and gas state university Press) pp 181–185

[8] Sinkevich Y V and Yankovskiy I N 2006 Physical-mathematical model of electro-impulsive 
polishing process Progressive technologies and machine engineering systems (Int. 
collection of scientific works) release 32 (Donetsk: Donetsk national technical university 
Press) pp 206-212