



ENHANCEMENT OF MICRO SMOOTH REFLECTIVE FINISHING OF AISI 304 STAINLESS STEEL

Ahmed M. Awad Abouelata

Department of Chemical Engineering and Pilot Plant, National Research Centre, El Bohouth St., Dokki, Giza, Egypt

E-Mail: awadxl@yahoo.com

ABSTRACT

Electropolishing (EP) process is successful for brightening and removal of roughness of AISI 304 stainless steel surface by controlling the conditions applied such as temperature, current density and time. Two electrodes system containing working electrode as anode and lead plate as cathode were used, where both electrodes were connected to DC power supply as a source of electric current. A mixture of phosphoric acid, sulfuric acid and water was prepared with constant viscosity 11 mPa and used as the electrolytic solution into a glass cell. Gloss value (G) of the surface was measured and metal loss in mili gram/cm² was determined after EP process, using different values of temperature, current density and time. SEM showed the improvement of the surface morphology. EDX analysis revealed the composition of the outer surface layer and the variation of Fe, Cr, Ni and Mn contents after EP process.

Keywords: electropolishing, stainless steel, morphology, brightness and roughness.

1. INTRODUCTION

AISI 304 stainless steel is characterized by distinct properties, e.g. thermal conductivity 16.2 W/m.K, solidus temperature 1400 °C, and liquidus temperature 1455 °C and linear thermal expansion coefficient 17.3 µm/m °C. So, it is widely used in different industries such as chemical equipment, cooking equipment, dairy equipment, evaporators, feed water tubing, flexible metal hose, food processing equipment, hospital surgical equipment, hypodermic needles, kitchen sinks, marine equipment and fasteners, nuclear vessels, oil well filter screens, refrigeration equipment, paper industry, pots and pans, pressure vessels, sanitary fittings, valves, shipping drums, spinning, textile dyeing equipment (Metals Handbook, 1990). Unfortunately, severe corrosion has been observed on structural steel exposed to mixtures of sulphur and water in several important industrial areas, especially in sour gas or oil production fields and transport systems (Postgate, 1984 and Werner, 1998).

Metal surface can be brightened and smoothed by three methods, namely mechanical, chemical and electropolishing (EP). Mechanical polishing has the disadvantage of the surface deformation, inducing residual tension and oxide incorporation, forming scratch marks and layer damage (Lee, 2000). Chemical polishing removes the surface roughness by very slow rate chemical dissolution of a thick layer of the surface by using expensive synthetic chemicals. While, a rapid regular dissolution of thin film of the surface occurred by applying EP using cheap chemicals in a simple set up (Tegart 1959 and Gabe *et al.*, 1994).

EP is generally the improvement of the surface finish of a metal by making it as anode in an appropriate solution. It can be used for different purposes, e.g. removal of deformations in metallic surfaces, cleaning of reactors, tubes, installations, valves, heat exchangers, making these materials more corrosion resistant and highly smooth surface.

Also, EP process is useful for removal of burrs from metallic machines after blanking or milling

processes, sharpening of tools, control of the dimensions of measurement instruments, smoothing of machine parts subjected to friction (Fedot'ev and Grilikhes, 1959 and Durney, 1986).

The surface after EP is highly smooth and it is characterized by the presence of billions of nearly hexagonal cells of dimensions 50-250 nm, and crystallographic grains of sample are not revealed (Vignal *et al.*, 2000).

EP of stainless steel work pieces is useful for smoothing and brightening of the surface, where the regulation of the surface and very fine burrs can be eliminated (John Swain, 2010). The optimizing of an EP process requires the achievement of total control of the involved variables such as temperature, duration time and current density then, an adequately bright surface can be obtained (Andrade Leonardo *et al.*, 2005). The diffusion of dissolved metal ions away from the polished surface leads to the formation of diffusion layer and hence EP is improved by the formation of a viscous salt film in the recesses of the surface. The diffusion layer play an important role to retard the velocity of ions attack towards the polished surface then, the surface obtained has less defects or pitting with high smoothness and brightness (Wang Xue *et al.*, 2012). The thickness of diffusion layer is relevant to roughness scale of the surface. As the diffusion layer thickness less than the roughness scale (Macro-profile), the diffusion layer follows the surface contour and under the mass transport conditions, peaks and recesses are equally removed and the EP is failed. On the other hand, when the diffusion layer thickness higher than the roughness scale of the surface (Micro-profile), under mass transport conditions, the peaks are preferentially removed and EP is succeeded (Taylor *et al.*, 2011).

The present work is focused on the study of the effect of different conditions such as temperature, current density and time applied during EP process on the surface modifications such as roughness and brightness. These aspects were explored by investigate the variation of surface morphology and composition before and after



treatment with the aid of surface analysis tools e.g. SEM and EDX, respectively. The study was extended to follow spontaneous variation of final temperature and electric current and determination of weight loss after EP.

2. EXPERIMENTAL

2.1 Materials and apparatus

Stainless steel sheet of the type AISI 304 was cut into small samples with dimension 3×2 cm. Each sample was degreased before treatment by rinsing with acetone, tap water, and then distilled water. Two electrodes system was used, where sample was fixed as working electrode and lead plate as counter electrode into a glass cell 15 cm×7.5 cm×8 cm, as shown in Fig 1. GW Lab DC Power Supply GPR-3030 was used as the source of DC electric current.

Electrolytic solution was prepared using mixture of 55% H_3PO_4 , 14% H_2SO_4 and 31% H_2O by weight. Magnetic stirrer (PMC-BARNSTEAD/ THERMOLYNE-USA) was used for agitation of the mixture during preparation of the electrolytic solution, where the electrolytic solution was mixed well for 5 min to have a homogenous solution, and then agitation was stopped before starting EP in a stagnant solution.

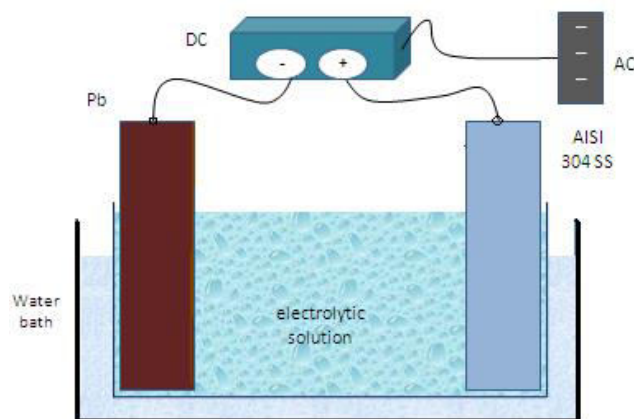


Figure-1. A schematic diagram of the electropolishing set up.

A water bath Julabo TWB 20 was used to control the temperature of the electrolytic solution, where the process was begun when the required temperature 25, 30, 40, 45 and 50 °C was certainly attained. After EP, each sample was taken out, and rinsed with tap water then

distilled water. All samples were dried at 105 °C for 15 min. All samples were stored well in a desiccator before weighing and surface analysis.

2.2. Surface characterization

Brightness of the surface was assessed by measuring gloss value (G) using ERICHSEN PICOGLOSS 503 Glossmeter, where the average gloss value (G) of different five points of the surface was measured.

SEM and EDX analysis were measured by using JEOL, JXA-840 A: ELECTRON PROB MICROANALYZER Dimension 3100 Scanning Probe Microscope (SPM) to detect morphological and topographical variation on the surface of AISI- 304 stainless steel before and after EP technique.

2.3. Weight loss measurements

AISI 304 stainless steel samples were weighed before and after EP treatment to determine the loss of weight of the surface by using a digital balance JS – 110 Chyo YMC Co. Ltd. (Japan).

3. RESULTS AND DISCUSSIONS

Anodic dissolution of stainless steel makes the surface rougher, but if the electrolyte is suitably chosen and the right range of current densities and temperature employed, the irregularities are smoothed out and a bright surface results (Walton, 1959).

3.1 Effect of temperature

Brightness and smoothness of the surface were improved with the increase of temperature, as shown in Figure-2, where gloss value was increased from 450 to 4600 with the increase of temperature from 25 to 50 °C, respectively, as a result of the uniformly anodic dissolution of the surface. Furthermore, as the temperature increased, the rate of anodic dissolution determined by the weight loss of the surface was increased, where the weight loss was increased from 19.3 to 45.3 mg/cm² by raising temperature from 25 to 50 °C.

The rate of metal dissolution was inherently increased after thermal energetic activation and acceleration of ionic mobility through the electrolytic solution, where the uniform dissolution of the surface can be achieved at high temperature.

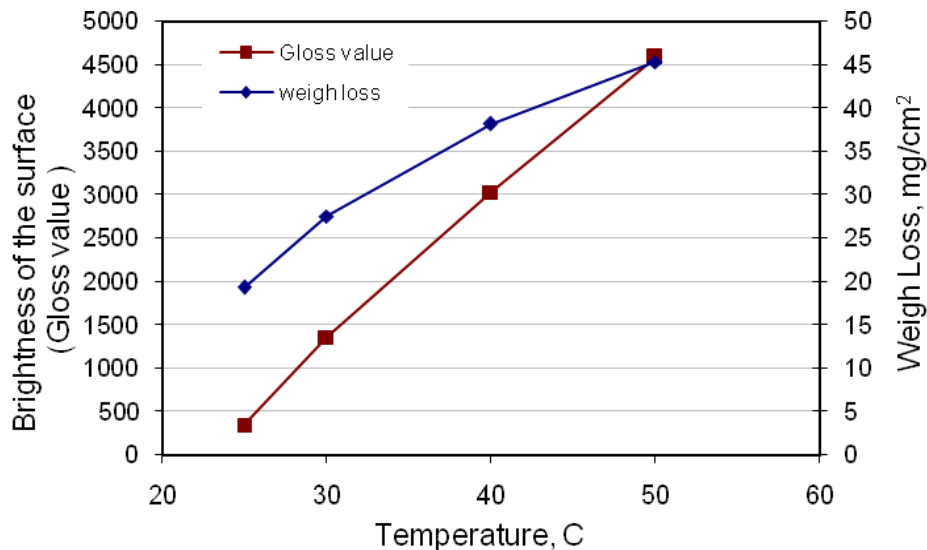


Figure-2. Effect of temperature on both measured gloss value and weigh loss of AISI 304 alloy after EP process.

EP process is exothermic and the anodic reactions are accompanied with heat. As illustrated in Table-1, it was found that the value of final temperature and final current are dependent on the initial temperature, where at the initial temperature 25 °C, the final temperature increased to 28 °C and the electric current was varied from 0.25 to 0.3 A/cm² after 10 min of EP. While at 40 °C, final temperature was increased to 48 °C and current density increased from 0.5 to 0.65 A/cm². The increase of

temperature and current can be attributed to the spontaneous heat release (exothermic), as a result of the anodic oxidation of the surface. The temperature of the electrolytic solution can be considered as the main factor of the EP process, where the highly viscous electrolytic solution (11 mPa at 25 °C) is highly resistive to the ionic mobility. Hence, the EP process requires an adequate temperature to overcome the ohmic resistance and increase the ionic mobility towards the working electrode.

Table-1. The effect of initial temperature on the final current and final temperature at constant potential 5 V and time 10 min.

Constant Time, min	Initial temperature T ₀ , °C	Initial current density A/cm ² (I ₀)	Final temperature T _f , °C	Final current density A/cm ² (I _f)
10	25	0.25	28	0.3
10	30	0.4	36	0.5
10	40	0.5	48	0.65
10	50	0.6	60	0.7

It can be observed that the final temperature and final current are spontaneously increased as the initial temperature increased, where the difference between initial and final temperature was increased from 3 to 10 °C as the initial temperature increased from 25 to 50 °C.

3.2 Effect of electric current

High electric current is necessary to achieve an adequate electric current distribution over the surface of working electrode and hence, a regular removal of asperities and fine burrs and homogeneous filling up of cavities. As shown in Figure-3, the brightness of the surface measured by the gloss value was increased by the

increase in electric current, where the gloss value of the substrate was improved from G=500 to G= 1800 by using 0.5 A/cm², and the surface reaction was accompanied by weight loss 36 mg/cm². While, the surface brightness was sufficiently improved by increasing current density to 0.65 A/cm², where gloss value was increased to G=4600 and the weight loss determined was increased also to 45 mg/cm². This indicates that a regular removal of higher weigh of surface asperities was regulated by using high electric current. So, the smoothness and brightness of the surface were highly improved and the surface seemed to be shinier and lustre.

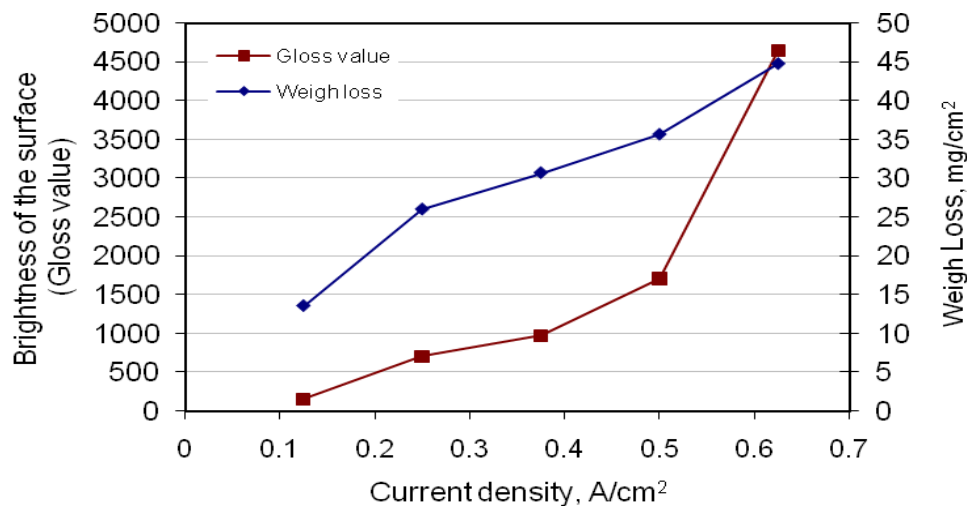


Figure-3. Effect of current density on both measured gloss value and weigh loss of AISI 304 alloy after EP process at constant initial temperature 30°C and time 10 min.

Low current densities 0.1, 0.25 and 0.4 A/cm² are not sufficient for ionic mobility through the viscous electrolytic solution and EP process suffered from the incomplete anodic oxidation for the attainment to the limiting current of polishing region, so the surface seemed to be matt with low gloss value ($G < 1000$). Due to low anodic reaction rate and the weak ionic mobility, the final temperature and current density were slightly increased, as

shown in Table-2. On the other hand, as the current density increased to 0.5 and 0.7 A/cm², the brightness of the surface was improved, where gloss value was increased to $G = 1700$ and $G = 4600$, respectively. This indicates the enhancement of driving force for the anodic reaction and surface regulation, so the final temperature and current density were spontaneously increased from 30 °C and 0.7 A/cm² to 63 °C and 0.8 A/cm², respectively.

Table-2. The effect of initial current on the final current and final temperature at constant initial temperature 30°C and time 10 min.

Constant factors		Initial current density A/cm ² (I_0)	Final temperature T_f , °C	Final current density A/cm ² (I_f)
time, min	Initial temperature T_0 , °C			
10	30	0.10	35	0.12
10	30	0.25	41	0.28
10	30	0.40	45	0.44
10	30	0.50	54	0.6
10	30	0.60	63	0.8

3.3 Effect of time

As shown in Figure-4, the brightness of the surface was improved from $G = 150$ to $G = 3250$ by elongation of EP time from 5 to 20 min at constant initial current and initial temperature. Then, the brightness was dramatically decreased, where the gloss value was decreased from 3250 to 1800 by continuous elongation of time from 20 to 30 min, where a high dissolution of metal surface was severely occurred by using a long time, leading to the distortion of the polished surface.

On the other hand, the EP process was accompanied by metal dissolution and loss of weigh by anodic oxidation. The loss of weigh was increased by the elongation of time, where it was increased from 15 to 132 mg/cm² by elongation of time from 5 to 30 min. So, a paramount metal surface was irregularly lost to the

solution and the surface was distorted by the continuous electric potential over the surface. When the polishing time became longer, although the surface roughness remained pretty much the same low time, some round-offs at the edges were observed with lower gloss value. Final temperature was spontaneously increased with time and consequently, the current was increased in spite of the beginning with constant initial temperature 30 and initial current density 0.4 A/cm². Final temperature was increased from 30 to 79°C and current density from 0.4 to 0.75 A/cm², as shown in Table-3.

The long duration time of EP more than 20 min, leads to the EP potential region exceeds the polishing region at the limiting current to transpassive region with aggressive irregular dissolution of metal surface with high



pitting corrosion. So, low brightness and low gloss value and high amount of weight loss were expected and found.

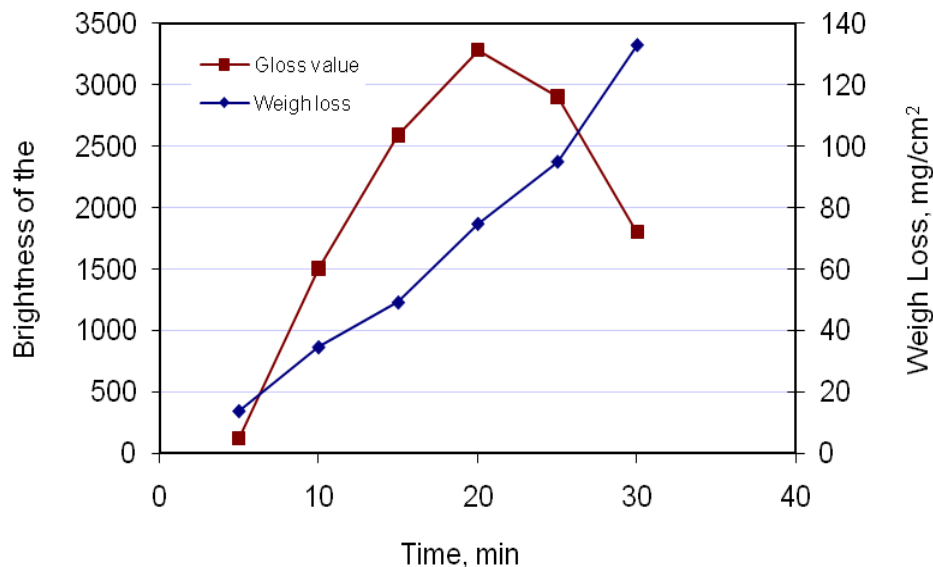


Figure-4. Effect of time on both measured gloss value and weigh loss of AISI 304 alloy after EP at constant initial temperature 30°C and initial current 1.5A.

Table-3. The effect of time of EP on the final current and temperature at constant initial temperature 30°C and initial current 1.5A (Potential = 6 V).

Constant factors		Time, min	Final temperature T_b , °C	Final current density $A/cm^2(I_f)$
Initial temperature T_0 , °C	Initial current density $A/cm^2(I_0)$			
30	0.40	5	39	0.43
30	0.40	10	50	0.52
30	0.40	15	55	0.60
30	0.40	20	62	0.64
30	0.40	30	79	0.75

3.4 Scanning Electron Microscope (SEM)

Levelling of the surface morphology was efficiently performed after EP treatment and the surface was rendered to the virginal surface after elimination of all micro asperities and the sharp roughness of the surface was regulated to wavy smooth. SEM analysis of AISI 304 stainless steel revealed that the surface morphology was

obviously smoothed out after EP process, where the roughness of substrate including scratches, asperities, lines and cavities are totally ceased after EP treatment, as shown in Figure-5. All imperfections found over the surface of substrate before EP were totally disappeared after EP process.

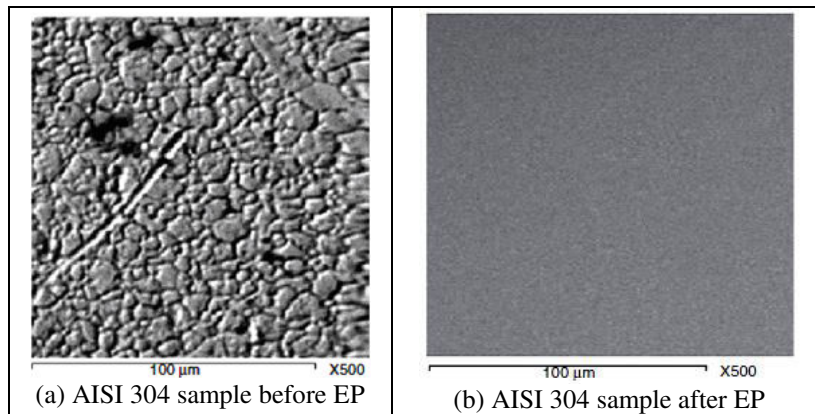


Figure-5. SEM analysis for AISI 304 sample before and after EP

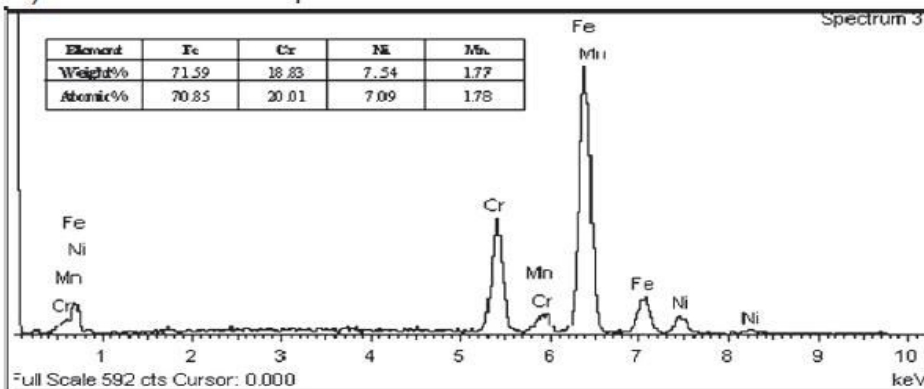
3.5 Energy dispersive X-Ray analysis (EDX)

Low conductivity electrolytes may be used to focus the electric current on surface asperities, whereas the electrolyte resistance is increased, the potential gradient between the asperities and the recesses becomes greater and the asperities are preferentially removed. Before EP, the $\text{Cr}_2\text{O}_3 / \text{Fe}_2\text{O}_3$ ratio is 0.97 and after EP, $\text{Cr}_2\text{O}_3 / \text{Fe}_2\text{O}_3$ ratio is increased to 2.58 and the major component of the passive film is Cr_2O_3 with passive layer thickness 25°A (Shuo-Jen and Jian-Jang, 2003).

Anodic dissolution of the outer surface of AISI 304 stainless steel surface was occurred by EP process. So,

according to the applied electric current and alloy composition, the dissolution threshold and dissolution rate of AISI stainless steel metal surface content were varied after EP process. Firstly, iron atoms are dissolved and escaped to the electrolytic solution at low electric potential. So, it can be observed that Fe weight % was decreased from 71.59 to 71.29 %, as shown in Figure-6. A bright and shiny surface was produced as a result of the increase of chromium density over the surface from 18.83 to 19.33 weigh %, imparting the luster and bright aspect of the polished surface.

a) EDX of blank1 sample



b) EDX of sample after EP

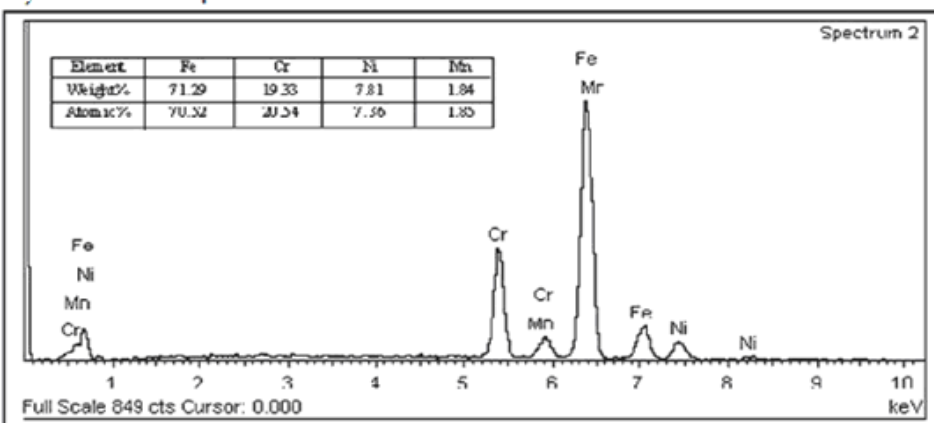


Figure-6. EDX analysis of AISI 304 sample before and after EP.



The surface content of Fe, Cr, Ni and Mn was varied after EP process as shown in Table-4. The increase of nickel content over the surface from 7.54 to 7.81 weight % improve the corrosion resistance of the surface, while the increase of manganese content from 1.77 to 1.84 weight % improve the hardness of the surface.

Generally, the surface of AISI 304 stainless steel acquired new advantage characteristics after EP process such as brightness, smoothness, corrosion resistance and high hardness, where Fe, Cr, Ni and Mn weight % were advantageously varied after EP treatment, accordingly, this technique can be utilized in different fields of metallurgical industries.

Table-4. Variation of the surface metal content of AISI 304 stainless steel after EP process

Metal	Before EP weight %	After EP weight %	difference weight %
Fe	71.59	71.29	- 0.3
Cr	18.83	19.33	0.5
Ni	7.54	7.81	0.27
Mn	1.77	1.84	0.07

CONCLUSIONS

Roughness and irregularities of the surface of AISI 304 stainless steel was eliminated by EP treatment. A high brightness can be obtained ($G=4500$) by the control of the conditioning factors such as temperature, current density and duration time, where the optimum conditions are vital to overcome the high viscosity (11 mPa at 25°C) of the prepared electrolyte. The highest gloss value ($G=4500$) was achieved by the increase of temperature to 50 °C and current density to 0.6 A/dm². While, the highest gloss value ($G=3250$) was obtained after 20 min, and decreased to ($G=1800$) with the elongation of time to 30 min. due to the aggressive dissolution of metal surface. SEM revealed the improvement of the surface morphology after EP treatment and EDX analysis showed the new advantageous characteristics of brightness, corrosion resistance and hardness. The new characteristics were found as a result of the surface content variation of Fe, Cr, Ni and Mn.

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