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INFLUENCES OF ELECTROLYTIC POLISHING PARAMETERS ON ROUGHNESS OF BORURED SURFACES, CASE OF X2CRNIMO17-12-2 STEEL (AISI 316L)[1]

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ABSTRACT

This study is about electrolytically polishing of 316L stainless steel boride surface, which is used in the field of implantology as biomaterials. The objective of this electrolytic polishing treatment is to improve the biocompatibility of this material, in particular its surface state. Tests have shown that electrolytically polished 316L has improved corrosion resistance over 316L boride. The formation of the Fe_2B boride layer does not allow us to use the same polishing technique as that used for the 316L substrate. The main objective is to develop the proper electrolytic polishing technique applied to the boride layer in order to compare electrolytically 316L and electrolytically polished 316L boride.

Keywords *- Stainless steel; boriding; biomaterial implant; electro-polishing.*

INTRODUCTION

Electro-polishing (EP) is a metal smoothing process where parts are processed in a generally acidic medium in the presence of current [1]. For my electro-polishing experiments, I used samples 5 mm thick and 16 mm in diameter; which are prepared by Mr BOUAZIZ. These samples are hypertreated at 1060°C for two hours and cooled with water. The initial surface hardness of X2CrNiMo17-12-2 grade steel (AISI 316L) is 175 ± 5.5 $HV_{0.1}$ [2]. These samples were subjected to free-air boriding treatment in a liquid environment by immersion in a salt bath electric furnace. The chemical composition of the boriding bath is 70% by weight of borax ($Na_2B_4O_7$) and 30% of silicon carbide (SiC). Three borating temperatures are used 850 °C, 950°C and 1000°C with treatment times of 2 h, 4 h and 6 h. After boriding, the hardness becomes on the surface is 1654 ± 110 $HV_{0.1}$ [2].

The chemical composition of the X2CrNiMo 17-12-2 stainless steel (AISI 316L) used was determined by laboratory spectroscopic analysis of Arzew GL2Z complex (Tab.1) [2].

[1] *How to cite the article:* Dabas N., Synthesis and Characterization of Calcium Carbonate Nanoparticles and their Effect on Vegetable Oil Based Polyurethane Adhesive; *International Journal of Research in Science and Technology*, Jan-Mar 2018, Vol 8, Issue 1, 18-30

Table 1.
Chemical composition of the steel used in mass%. [2]

C	Mn	Ni	Co	Cr	Mo	Ti	Si	V
0.03	1.3	12.2	0.35	17.4	2.28	0,44	0.45	0.07

Mechanical and electrolytic polishing treatment

By anodic dissolution of the surface of a sample, it is possible to obtain excellent polishing resulting in almost no deformation on its surface. The sample is polarized at the anode and connected to the cathode through an electrolyte bath concentrated in acid. The application of a voltage creates a current that travels through the bath and thus preferentially alter the surface to be polished. Before all this, it is preferable to pre-polish the surface of the sample with SiC abrasive paper type 240 grain / cm^2 [3]. This was done with the equipment available in the metallurgy laboratory of the Department of Mechanical Engineering of the National Polytechnic School of Oran.

A glass container was used as an electro-polishing cell. The boronized X2CrNiMo 17-12-2 (AISI 316L) steel sample is used as anode and a slightly larger sheet of 316 stainless steel is used as the cathode. The electrolyte and the parameters are changed according to the experiment. For each electro-polishing treatment, a new fresh electrolyte was used, as it is known that changes in the ionic metal concentration could have an influence on electro-polishing states [2].

After the electro-polishing operation the sample is cleaned by immersion in a mixture of hydrofluoric acid (2% v/v), nitric acid (10% v/v) and DI water (88% v/v), for 30 s at 50 °C in order to dissolve the salts without attacking the metal [2].
Before each electro-polishing operation, we carry out a mechanical polishing.

After each operation, the sample is ultrasonically cleaned in a solution of water and acetone for 10 minutes, and then dried with compressed air. The roughness is measured using a "Surftest 201" portable rugosimeter of the metrology laboratory of the mechanical department of the National Polytechnic School of Oran. Each measurement was repeated five times at different locations and the roughness values of the sample are recorded in a table after each experiment.

Electropolishing experiments:

a. Experience N ° 1:

- The electrolyte bath of the electropolishing operation consists of a mixture of:
  - ✓ glycerol 99% (47% v/v)
  - ✓ phosphoric acid 85% (42% v/v)
  - ✓ water D.I. (15% v/v)
- The distance between the two electrodes is fixed at 60 millimeters
- The current density used is 1 to 20 A / dm^2 (0.017A to 0.35A)

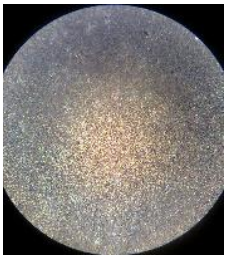
- The required duration of the electro polishing process: 10 mn
- The temperature of the electrolyte: 100 ° C

Mr Sarup Chopra [4] has proposed this composition of the electrolyte and these parameters. This electrolyte bath is used for stainless steels.
Note: the experiment is redone 3 times.

Table 2:
Ra results [µm]

Number / Test	1	2	3	4	5	Ra_{avg}
Mechanical polishing	0.07	0.1	0.06	0.06	0.07	0.08±**0.02**
Electro-polishing	0.26	0.27	0.31	0.35	0.27	0.3±**0.04**

Figure 1.
Stereomicroscope viewing of the sample after treatment



After observation with the stereo microscope, it was seen that the surface of the sample was attacked by the electrolyte. Asperities have been observed in the cavity form (Fig.1). So we decided to repeat the experiment starting with a mechanical polishing of another sample and by electrolyte change.

b. Experience N ° 2:

- The electrolyte bath of the electropolishing operation consists of a mixture of [5]:
  - ✓ Sulfuric acid H_2SO_4 (10% v/v)
  - ✓ phosphoric acid H_3PO_4 85% (90% v/v)
  - ✓ water D.I. (10% v/v)
- The distance between the two electrodes is set at 80 millimeters
- The current density used is 20 to 90 A / dm^2 (0.35A to 1.59A)

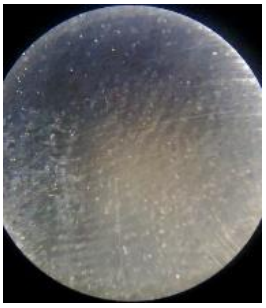
- The required duration of the electro polishing process: 10 mn
- The temperature of the electrolyte: 80°C

This composition of the electrolyte and these parameters were used for hard steels; because of the similarity of the characteristics of the hard steels with the boride stainless steels, this electrolyte composition was used to make the electropolishing treatment of our sample. This electrolyte bath is used for stainless steels. The roughness values of the sample are shown in Table.3:

Table 3
Ra results [µm]

Number / Test	1	2	3	4	5	Ra_{avg}
Mechanical polishing	0.06	0.05	0.06	0.09	0.07	0.07±**0.02**
Electro-polishing	0.29	0.23	0.36	0.21	0.3	0.2±**0.06**

Figure.2
Observation at the stereo microscope of the sample after treatment



The electropolishing bath becomes pale yellow after a treatment period. In addition, the surface of the sample becomes brown: it is covered with thin blackish brown films that detach over time. The observed color change is then likely related to the partial degradation of residual organic matter and initially present (use of a solvent during the electroerosion of the sample) on the surface of the sample [1]. Because of these reactions, we had inaccurate roughness values for our study; so we decided to repeat the treatment with another electrolyte.

c. Experience N°3:

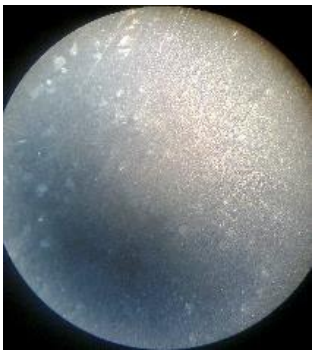
- The electrolyte bath of the electropolishing operation consists of a mixture of [5]:
  - ✓ Sulfuric acid H_2SO_4 (30% v/v)
  - ✓ phosphoric acid H_3PO_4 85% (70% v/v)
  - ✓ water D.I. (10% v/v)
- The distance between the two electrodes is set at 80 millimeters
- The current density used is 20 to 90 A / dm^2 (0.35A to 1.59A)
- The required duration of the electro polishing process: 10 mn
- The temperature of the electrolyte: 80°C

This composition of the electrolyte is similar to the previous one but with a change in composition and parameters. It is also used for hard steels; for the same reason, this electrolyte composition was used to make the electropolishing treatment of our sample. The roughness values of the sample are shown in Table 4:

Table 4.
Results Ra [µm]

umber / Test	1	2	3	4	5	Ra_{avg}
Mechanical polishing	0.06	0.05	0.06	0.09	0.07	0.07±**0.02**
Electro-polishing	0.21	0.25	0.13	0.21	0.08	0.16±**0.08**

Figure 3.
Stereomicroscope observation of the sample after treatment



During the operation, the same changes as the previous experiment were observed, but with an improvement of the roughness values; so we decided to redo the treatment with another electrolyte to see if we can minimize these roughness values.

d. Experience N°4:

- The electrolyte bath of the electropolishing operation consists of a mixture of [4]:
  - ✓ Chromium oxide Cr_2O_3 (25g)
  - ✓ Acetic acid (133 ml)
  - ✓ D.I. water (7 ml)
- The distance between the two electrodes is fixed at 60 millimeters
- The current density used is 0.09 to 0.22A/cm^2
- The required duration of the electropolishing process: 6 mn
- The temperature of the electrolyte: 17 to 19°C

Mr Sarup Chopra [4] has proposed this composition of the electrolyte and these parameters. This electrolyte bath is used for thermochemically treated stainless steels. To begin, Cr_2O_3 must be dissolved in the solution at 60-70 ° C

Result:

An attempt was made to dissolve the chromium oxide under the given conditions but this was not possible. After a little research on the internet, it was found that the process of dissolution of chromium oxide is impossible in water but it can be in the acid but with conditions: very high temperature and a long duration [6]. And we did not have a transition of electric current. To see if we can dissolve the chromium oxide under the conditions given by Mr Sarup Chopra we will redo the same experiment decreasing the amount of chromium oxide.

e. Experience N°5:

- The electrolyte bath of the electropolishing operation consists of a mixture of [4]:
  - ✓ Chromium oxide Cr_2O_3 (2.5g)
  - ✓ acetic acid (133 ml)
  - ✓ D.I. water (7 ml)
- The distance between the two electrodes is fixed at 60 millimeters
- The current density used is 0.09 to 0.22A/cm^2
- The required duration of the electro polishing process: 6 mn
- The temperature of the electrolyte: 17 to 19°C

To begin, Cr_2O_3 must be dissolved in the solution at 60-70 ° C

Result:

We arrived at the same result as the previous experiment: no dissolution of chromium oxide. And we did not have a transition of electric current.

f. Conclusion:

We make a comparison between our results and those obtained in a previous work [2]:

Table 5.
Roughness of the treated surfaces obtained

		Ra_{avg} **[µm]**		**Reference**
		Mechanical polishing	Electro-polishing	
Non-boronized sample		**0.07 ±0.02**	**0.05 ±0.02**	[2]
Borured sample		**0.06 ±0.03**	**1±0.8**	[2]
Borured sample	**Experience N°1**	0.08 ±0.02	0.3 ±0.04	
	Experience N°2	0.07 ±0.02	0.2 ±0.06	
	Experience N°3	0.07 ±0.02	**0.16 ±0.08**	

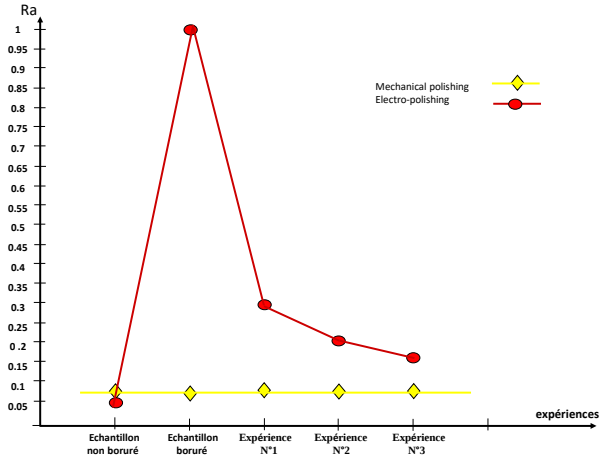


Figure 4. Evolution of roughness

From Table 5 we confirm that the electrolytic polishing of X2CrNiMo17-12-2 (AISI 316L) steel leads to a smooth surface compared to mechanical polishing except for the boride sample. The results are improved from one experiment to another by electrolyte solution change and/or parameter change (FIG. 4). The electrolyte of Experiment No.3 gave a better roughness compared to other electrolytes; to improve the roughness we will work on the parameters affecting the change of the surface condition.

- The electrolyte bath of the electropolishing operation consists of a mixture of [5]:
  - ✓ Sulfuric acid H_2SO_4 (30% v/v)
  - ✓ Phosphoric acid H_3PO_4 85% (70% v/v)
  - ✓ Water D.I. (10% v/v)
- The distance between the two electrodes is set at 80 millimeters
- The current density used is 20 to 90 A / dm^2 (0.35A to 1.59A)
- The required duration of the electro polishing process: 10 mn
- The temperature of the electrolyte: 80 ° C

g. Influencing parameters:

- **1st parameter:**

The duration of the electropolishing process

Table 6. Change in roughness by change in the duration of the electropolishing process

Parameters				Ra(µm)
distance (mm)	DC(A)	Duration (mn)	T (°C)	
80	**1**	**5**	**80**	**0.11±0.06**
80	1	7	80	0.14±**0.08**
80	1	10	80	0.16±**0.08**
80	1	12	80	0.68±**0.19**

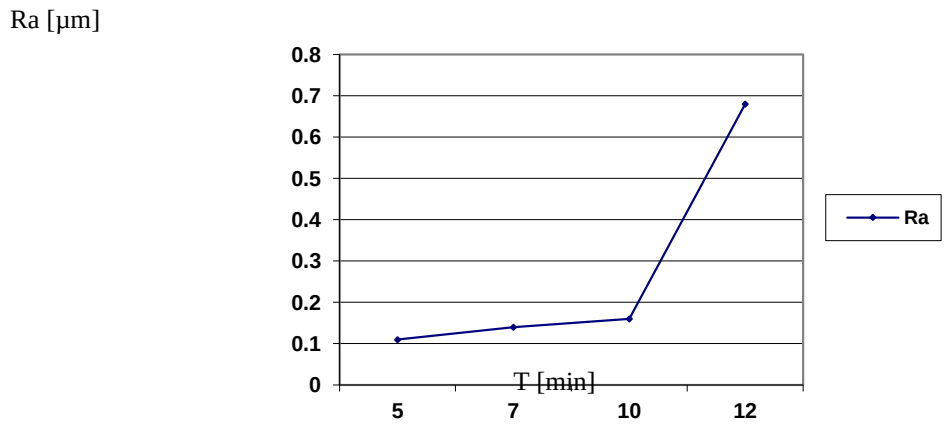


Figure 5.
Roughness change with respect to the duration of the electropolishing process.

Conclusion:

The duration of 5 minutes gave a better roughness compared to the other duration (Ra = 0.11 ± 0.06 µm).

- **2nd parameter:** The distance between the two electrodes.

Table 7. Roughness change by change of the distance between the two electrodes

Parameters				**Ra(µm)**
distance (mm)	**DC(A)**	**Duration (mn)**	**T (C°)**	
80	**1**	**5**	**80**	**0.11±0.06**
90	1	5	80	0.12±**0.08**
100	**1**	**5**	**80**	**0.1±0.03**
110	1	5	80	0.23±**0.08**

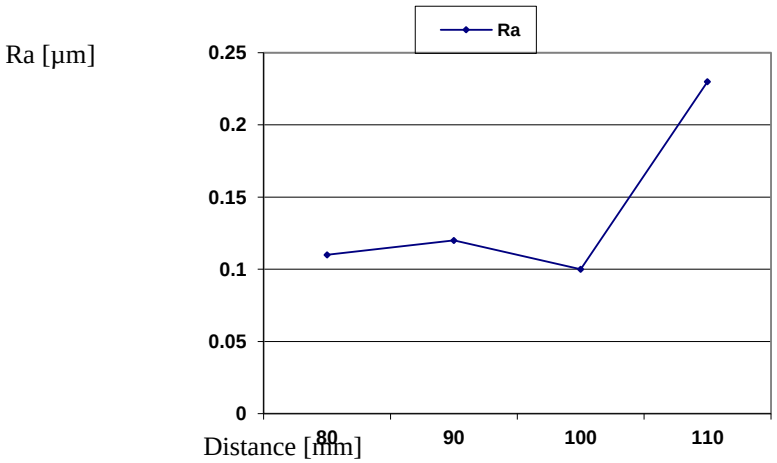


Figure 6.
Roughness change with respect to the distance between the electrodes

Conclusion:

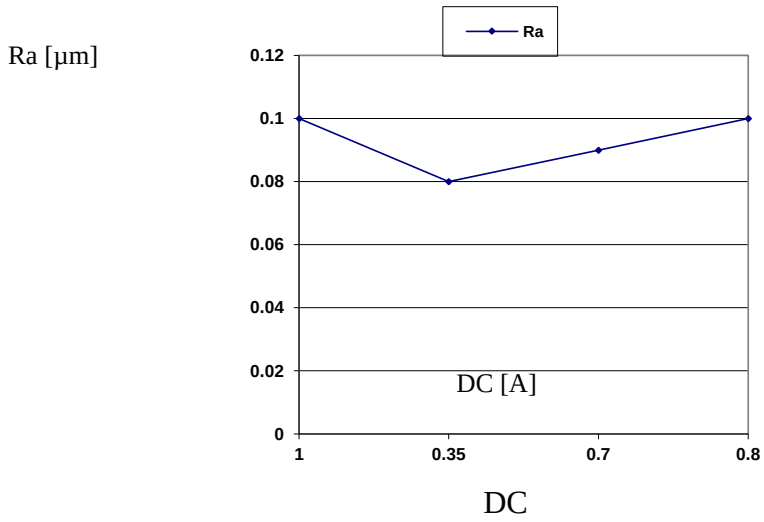
The distance of 100mm gave a better roughness compared to the other distances (Ra = 0.1 ± 0.03 µm).

- **3rd parameter:** The current density

Parameters				Ra (µm)
distance (mm)	**DC(A)**	**Duration (mn)**	**T (C°)**	
100	**0.35**	**5**	**80**	**0.08±0.06**
100	0.7	5	80	0.09±**0.03**
100	0.8	5	80	0.1±**0.05**
100	1	5	80	0.1±**0.03**

Table 8. Change of Roughness by Change of Current Density

Figure 7.
Roughness change with respect to current density.



Conclusion:

The current intensity 0.35A gave a better roughness compared to the other intensities (Ra = 0.08 ± 0.06 µm).

- **4th parameter:** the temperature of the electrolyte

Parameters				Ra (µm)
distance (mm)	DC (A)	Duration (mn)	T (C°)	
100	**0.35**	5	60	0.14±**0.06**
100	**0.35**	**5**	**80**	**0.08±0.04**
100	**0.35**	5	90	0.13±**0.08**

Table 9. Roughness change by changing the temperature of the electrolyte

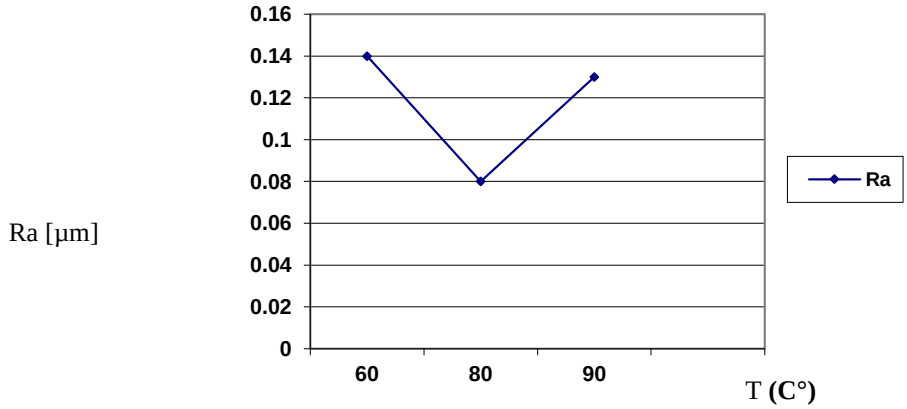


Figure 8.
Roughness change with respect to temperature.

Conclusion:

The temperature 80 ° C gave a better roughness compared to the other temperatures (Ra = 0.08 ± 0.04 µm).

- **Optimum result:**

Table 10. Optimum Roughness

Ra [µm]

		Ra_{avg} [µm]		Reference
		Mechanical polishing	Electro-polishing	
Unborured sample		**0.07 ±0.02**	**0.05 ±0.02**	[2]
Borured sample		**0.06 ±0.03**	**1±0.8**	[2]
Borured sample	**Experience N°1**	0.08 ±0.02	0.3 ±0.04	
	Experience N°2	0.07 ±0.02	0.2 ±0.06	
	Experience N°3	0.07 ±0.02	**0.16 ±0.08**	
	Ra optimum	0.07 ±0.02	**0.08±0.04**	

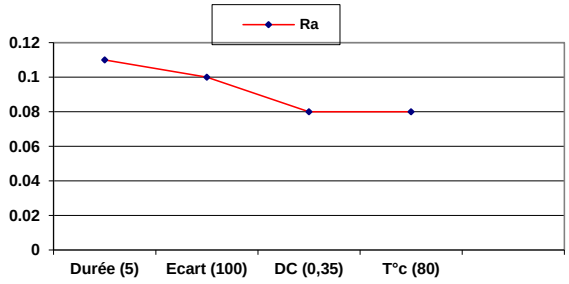


Figure 9. Evolution of roughness.

h. Conclusion

The objective of the present study was to see the effect of electrolytic polishing on the surface of a stainless steel X2CrNiMo 17-12-2 (AISI 316L) undergoing a boriding treatment. The set of

treatments had a goal to improve its biocompatibility in view of its use as an implant because the combined action of the chemical attacks of the blood medium and the mechanical constraints imposed during the installation of the prosthesis can induce important phenomena corrosion.

The experimental methodology is developed in two stages. First, try several electrolytes to make the choice of the best between them that give a minimum roughness. The second step in the experimental procedure was to optimize the roughness by changing electrolytic polishing parameters affecting the surface condition of the boronized X2CrNiMo 17-12-2 (AISI 316L) steel. From one experiment to another, we have seen the evolution of roughness, where we observe the influence of each parameter. The limit value we were able to obtain in the case of electrolytic polishing of the boride layers of X2CrNiMo17-12-2 (AISI 316L) stainless steel is Ra = 0.08 ± 0.04 µm, it remains slightly higher than that obtained after electrolytic polishing substrate (Ra = 0.05 ± 0.02 µm) (Table 11.)

The results obtained were interpreted by graphs. These curves make it possible to see the evolution of the roughness from one experiment to another.

Table 11. Roughness of the treated surfaces obtained and optimum roughness

Parameters				**Ra (µm)**
distance (mm)	**DC (A)**	**Duration (mn)**	**T (C°)**	
100	**0.35**	**5**	**80**	**0.08±0.04**

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