

CHARACTERIZATION OF GRADE 316 STAINLESS STEEL AS A BIOMATERIAL BY ELECTROCHEMICAL ANALYSIS

By

M. T. Z. Butt¹, F. Riaz, F. Hussain and K. M. Deen

Abstract

The electrochemical corrosion behavior of Type 316 stainless steel has been studied in ringer 2.5% Goat serum solution. Chemical stability and biocompatibility in body fluids and tissues are the basic requirements for successful application of implant materials in bone fractures and replacements. Incidences of failure of implant devices reveal the occurrence of significant localized corrosion viz., pitting and crevice corrosion. The aim of this study was to replace the Type 316L stainless steel which is widely employed in medical implants such as orthopedic replacements and in surgical cutlery with the Type 316 stainless steel because of their relatively low cost and ease of fabrication. The effect of Surface morphology and treatment (passivation) on corrosion behavior of Type 316 Stainless Steel by holding it in 20-vol% HNO₃ at temperature of 60 °C for 30min was also studied. Cyclic polarization scans was the electrochemical technique selected for this work.

Introduction

Stainless steels (SS) are widely used as biomaterials and materials of construction. In biomedical applications they are used as coronary and pulmonary stents, hip prosthesis, screws, external fixations, etc. This is mainly due to their good resistance to general corrosion. Generally, the first requirement of any material serving in a biological system is that it should be inert and not cause any undesirable reaction with its surrounding. The literature has proven that any deviation from the natural concentration of the ions such as chromium, iron and nickel inside the body may severely prevent the body from functioning properly, and can ultimately lead to severe health-related consequences. Therefore, corrosion of the material is the first issue to be considered when it is designed to function in a human body [1]. Some improvement in pitting resistance has been achieved by these and some other methods. Nitric acid has been used as one of the most popular chemical passivation reagents for surface treatment of surgical SS implants.

Experimental

The corrosion analysis of 316 Austenitic Stainless Steel was carried out using DC Electrochemical techniques Via "Cyclic Polarization". The composition of AISI 316 Austenitic Stainless Steel is shown in table 1:

Table 1: Chemical composition of 316 Stainless Steel

Elements	C%	Cr%	Ni %	Mo %	Si %	Mn %	S %	P %
% Comp	0.08 max	16.0- 18.0	10.0- 14.0	2.0- 3.0	1.0 max	2.0	0.03	0.045

Metallograpy

The specimen were cut from a sheet and then cold mounted. The sample was cut into 1 by 1 cm² square shape. Each sample was first ground by using sand papers of grit sizes 120, 320, 400, 600, 1000, 1200, 1500 & 2000 respectively and then polished on billiard cloth with alumina oxide abrasive. Each specimen was electrolytically etched by using oxalic acid having 10 g/100 ml H₂O composition at 6V for 1-1.5 minutes and examined under the Optical Microscope.

1. Department of Metallurgy & Materials Engineering, CEET, University of the Punjab, 54590, Lahore, Pakistan.

Analysis

AISI 316 Austenitic Stainless Steel samples were studied by

1. Electrochemical Analysis
2. SEM

Surface Treatment

The Surface treatment (Passivation) of AISI 316 Stainless steel was done by the method as mentioned in table 2.

Table 2: Passivation experimental Conditions and Method

Passivation Bath (pH = 3)	
HNO ₃	20 % Vol
Bath Temperature	60-70°C
Immersion Time	30 min

Testing Environments

The corrosion tests were carried out in the Ringers'-Serum Solution. The composition of solution is given in table 3.

Table 3: Ringer-Serum solution composition

Ringers'-Serum Solution (1000 ml) pH = 6	
Ringers' Solution	975 ml
Goat Serum	25 ml

Typical composition of ringer solution is given in table 4.

Table 4: Composition of Ringer solution

Ringers' Solution (Per 1000 ml) pH = 6 (On pH paper)	
NaCl (Sodium Chloride)	8.36 g
KCl (Potassium Chloride)	0.3g
CaCl ₂ (Calcium Chloride)	0.15g

Nomenclature

The designation of sample or cyclic polarization tests was made as follows (Table 5)

Table 5: Nomenclature of testing setup

Sample Designation	Solution	Surface Conditions
CCRC-1	Ringer-Serum (Plain)	Grind (600 Grit size)
CCRC-2		Grind (600 Grit size) + Passivated

DC Electrochemical Test

The corrosion specimen must be prepared in a fashion indicative of the service material. Corrosion behavior is influenced by the slightest variation in metallurgical structure and surface preparation - thus care must be taken while preparing the surface of the testing specimens. Epoxy Embedded electrode was produced which acted as working electrode. Very less technical expertise and equipment was required for this. In this electrode electrical connection

was made by soldering. A glass tube was required to accommodate this conducting wire. The specimen was prepared in a manner as indicated in figure 1.

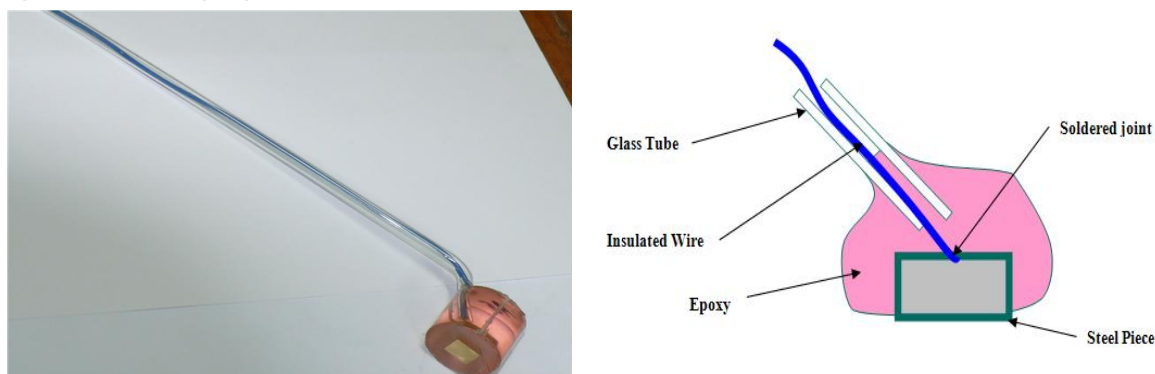


Figure 1: Prepared Sample for Electrochemical Testing

DC Electrochemical technique -- Cyclic Polarization (CP) was employed using M/S Gamry Potentiostat. The three electrode cell configuration use for electrochemical testing is shown in figure 2.

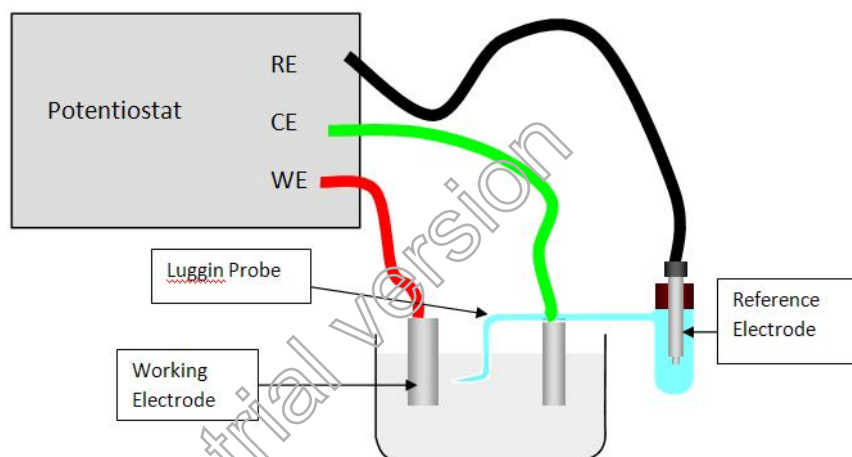


Figure 2: Three electrode cell design and experimental setup

The input values for cyclic polarization test were as follows (Table 6)

Table 6: Input parameters for cyclic polarization testing.

Parameters for Cyclic Polarization	
Initial Potential	-500mV vs. SCE
Apex potential	1500mV vs. SCE
Final Potential	-500 mV vs. SCE
Forward Scan Rate	5.0 mV/s
Reverse Scan Rate	2.5 mV/s
Apex current density	10mA/cm ²
Sample Period	1s
Sample Area	1 cm ²
Density	8 g/cm ³
Equivalent Weight	26.13

Results

Metallography

The resultant microstructure of 316 stainless steel sample is given in figure 3.



Figure 3: Microstructure of 316 Stainless steel (Electrolytically etched, 200X)

Cyclic Polarization Tests and Resulted Microstructures

Cyclic polarization was carried out in ringer's solution, by using the M/S Gamry Potentiostat (version 5.30). The cyclic polarization scans for samples CCRC-1 and CCRC-2 are given in figures 4 and 5 and optical micrographs and SEM images resulted after electrochemical testing are depicted in figures 3 and 6 respectively.

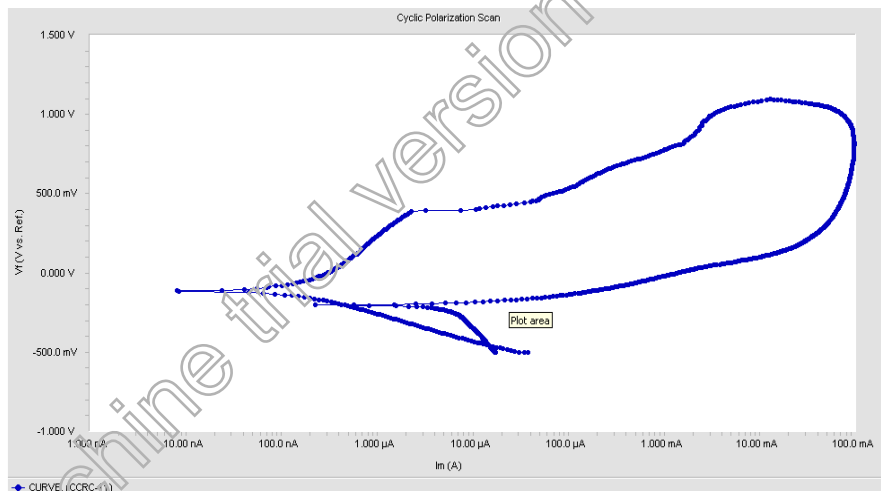
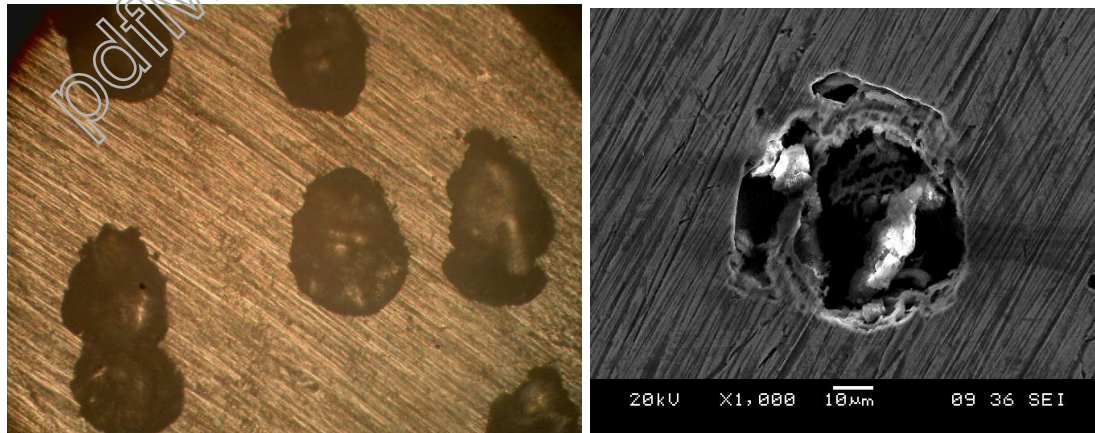


Figure 4: CCRC-1 (Non Passivated), Cyclic Polarization scan



(a) **(b)**
Figure 5: CCRC-1 surface after electrochemical (a) Optical micrograph, (b) SEM image

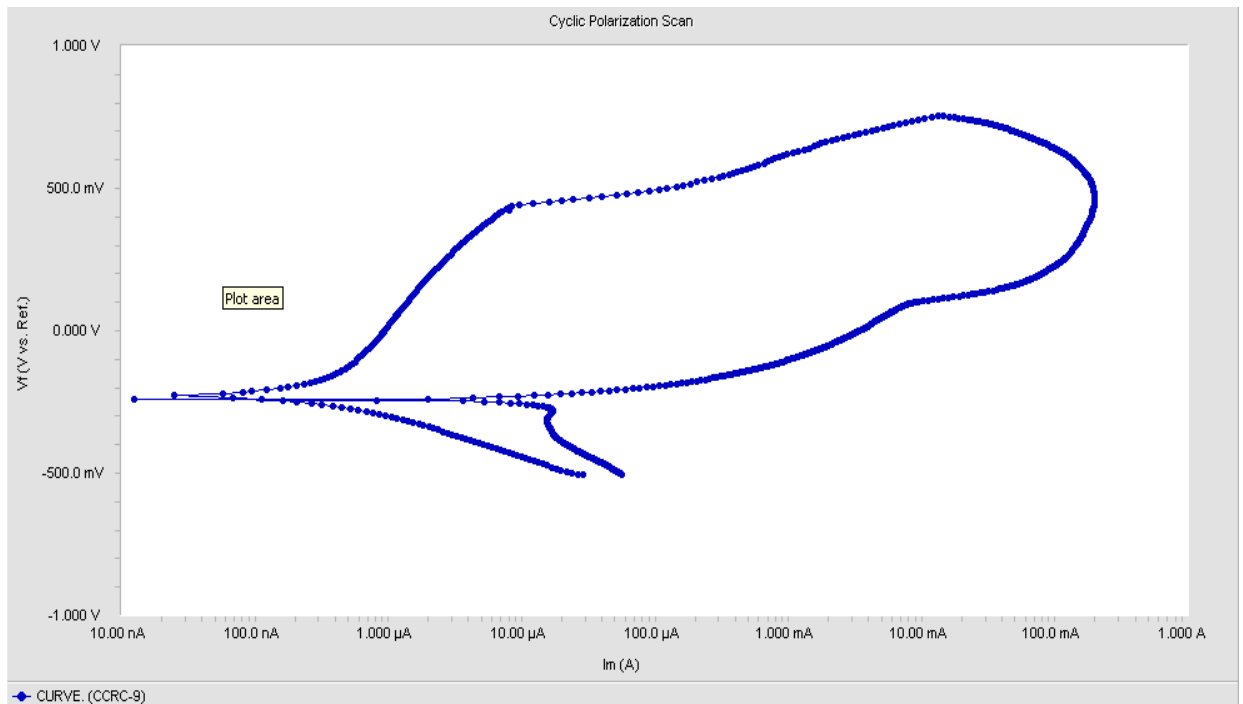


Figure 6: CCRC 2 (Passivated), Cyclic Polarization scan

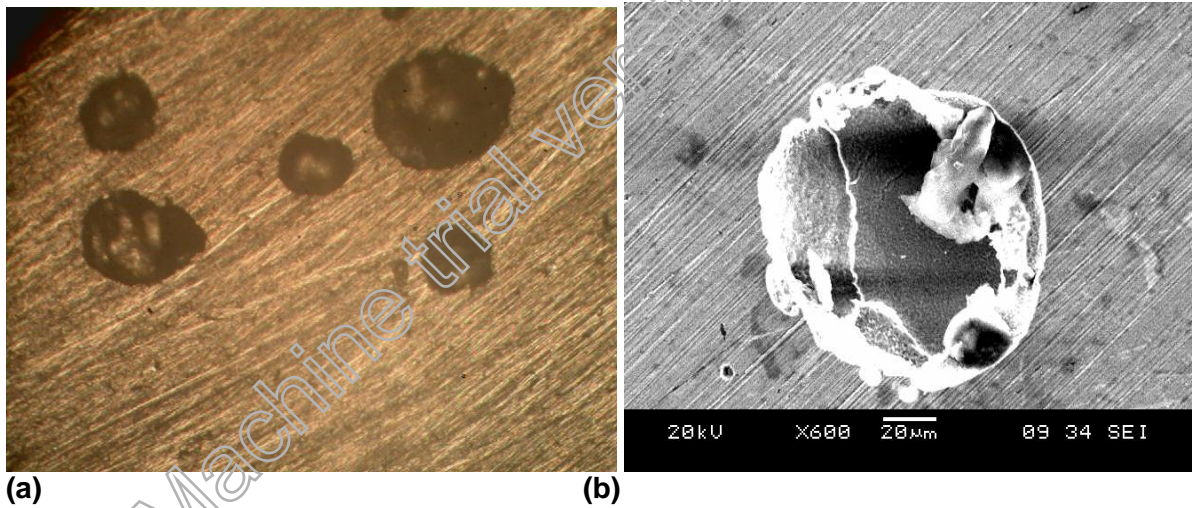


Figure 7: CCRC-2 surface after electrochemical (a) Optical micrograph, (b) SEM image

Cyclic Polarization Data

The values obtained after cyclic polarization scans are given in table 7 below.

Table 7: Output values obtained after cyclic polarization scans

Sample ID	OCP (mV)	E_{Corr} (mV)	E_{pit} (mV)	E_{rp} (mV)	$E_{rp}-E_{Corr}$ (mV)
CCRC-1	-268.02	-228.2	-520.25	-260.6	-40.1
CCRC-2	-198.24	-256.51	-486.70	-145.3	100.21

Discussion

The microstructure of prepared and etched sample showed equiaxed austenitic grains with twin lines within the grain. At higher magnification it was observed that there was also some evidences of sigma intermetallic phase which may initiate pitting corrosion at localized points. [3] The formation and performance of surface passivation film on 316SS is also dependent on surface morphology. In this study, rough surface without passivation provided no protection as depicted by negative value of ($E_{rp} - E_{corr} = -40.1\text{mV}$) than passivated one which provided higher pitting resistance ($E_{rp} - E_{corr} = 100.21\text{ mV}$) as analyzed in ringer's-serum solution by cyclic polarization scans. It was examined that E_{rp} of passivated sample in ringer's-serum was less negative (noble) than non-passivated sample (active). The more negative E_{rp} value (-260.6mV) than E_{corr} suggest lower potential range for pitting protection of non passivated rough sample. It was also observed that the value of E_{pit} for passivated surface depicted low corrosion tendency in ringer's-serum solution than non-passivated as provided by noble corrosion potential, E_{Corr} . [4-6] Higher pitting resistance of passivated sample concluded that the passivation process may enhance pitting corrosion resistance but this process does not completely qualify as a protective methods because of larger pits formed at the passivated surface as represented in micrographs obtained by optical microscope and SEM images.

Conclusion

Cyclic polarization scan showed that the tendency of the pits to nucleate at the surface was relatively higher in ringer's-serum solution for both passivated and non-passivated surfaces. It was also concluded that the re-passivation tendency of 316 stainless steel samples was less in non passivated surface than passivated one in ringer's-serum solution this behavior suggest that uncontrolled severe pit growth. It was also observed that rough surface promote more pitting corrosion which determines that surface morphology greatly effect the pitting corrosion tendency.

References

1. A. Shahryari et al. / Materials Science and Engineering C 28 (2008) 94–106
2. ASTM F86-91, Standard practice for surface preparation and marking of metallic surgical implants.
3. J. S. Noh, N.J. Laycock, W. Gao, D.B. Wells, Corr. Sci. 42 (2000) 2069.
4. T. Hong, T. Ogushi, M. Nagumo, Corr. Sci. 38 (1996) 881
5. F. Mansfield, C.B. Breslin, A. Pardo, F.J. Perez, Surf. Coat. Technol. 90 (1997) 224.
6. S. Fujimoto, T. Yamada, T. Shibata, J. Electrochem. Soc. 145 (1998) L79.