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A STUDY OF PASSIVATION BEHAVIORS OF SURGICAL AUSTENITIC AND MARTENSITIC STAINLESS STEEL ALLOYS (316 SERIES AND 410 SERIES) IN CITRIC ACID AND NITRIC ACID

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Abstract: Steels are said to be stainless when they resist corrosion; this is achieved by dissolving sufficient chromium in the iron to produce a coherent, adherent, insulating and regenerating chromium oxide protective film on the surface. Passivation is a protective layer formation technique. Stainless steels are passivated to avoid the corrosion. Mostly the surgical stainless steels are more sensitive and passivated to avoid the corrosion and increase the life time of the instruments. Passivation is carried out normally in nitric acid or citric acid. The key variables are time, temperature and process. The main objective of this research is to study the passivation of surgical steels. The effects of key variables such as temperature, time and process etc. were studied and compiled. A number of experiments were carried out using the chemical compositions such as Citric Acid (10 wt. %, 30 wt. % & 50 wt. %, respectively) and Nitric Acid (20 vol. %, 50 vol. % & 70 vol. %, respectively). All the chemicals and the key variables used for the passivation of surgical steels samples were found satisfactory. Most of the samples did not show any pink spots except a few one. Therefore, these steels are strong candidates for applications in biomedical applications as Biomaterials.
Keywords: Biomaterials, Surgical stainless steel, passivation, corrosion, Citric acid, nitric acid, Bioinstrumentation

1. INTRODUCTION

Fused salts are viewed as currently being a recognized choice in many professional functions. Within the last few many years a lot more interest is actually devoted to this biochemistry connected with fused salts [1]. They've been used since coolants within nuclear reactors, temperature exchange functions and as effect mass media within element in addition to electrochemical functions. That they match the specifications of countless electricity alteration functions, particularly within gasoline cell phone purposes [2-3]. Molten alkali carbonates have sketched a person's eye of countless analysis groups for their helpful purposes in the ecologically protected gasoline tissue as well as the latest developments which include gasoline healing such as hydrogen, methane as well as other hydrocarbons through healthy bio-compounds [4]. In such purposes, possibly corrosive chemical substances are formed in addition to conditions intended for elevated costs connected with deterioration are mixed together [5].
Stainless steel derives it is deterioration level of resistance coming from a slim, tough layer connected with chromium oxide of which varieties at the metal's surface area and provides steel it is feature `stainless excellent. The actual unaggressive picture on the steel surface area includes a mixture of in terms of iron oxide, chromium oxide in addition to, when molybdenum exists in the mass metal, molybdenum oxide [6-7]. Even so, for just a passivated picture to be effective, that must provide a protecting buffer of which will keep this deterioration latest within the metal-environment interface on reasonably low sufficient fee so your magnitude connected with deterioration injury is actually reduced [7]. A highly effective picture is actually one that resists this breakdown from the unaggressive picture of course, if abraded, repassivates right away; for this reason, this conditions of which showcase deterioration connected with lightweight aluminum and its metals, need to be the ones that regularly abrade this picture mechanically or even showcase side effects of which locally weaken this specific protecting oxide picture simply by reducing this accessibility to fresh air for you to repair that [7-8]. Metallic biomaterials are oppressed due to their corrosion resistance; they do not possess biofunctionalities like blood compatibility, bone conductivity and bioactivity. Hence, surface modifications are required and surface modification is carried out by passivation treatments. Now a days, large number of surgical stainless steels as biomaterials composed of nontoxic and allergy-free elements are being developed.

In the current study, Passivity of 316 and 410 surgical stainless steels were studied. A number of experiments were carried out using the chemical compositions such as Citric Acid (10 wt. %, 30 wt. % & 50 wt. %, respectively) and Nitric Acid (20 vol. %, 50 vol. % & 70 vol. %, respectively).

2. EXPERIMENTAL WORK

The procedure for passivation and its testing contains three steps. The first Step was the Cleaning of Sample. A sample of 316 series of stainless steel was taken. The sample was cleaned with an emery paper. After that the sample was cleaned with a brush to remove dust. Then the sample was washed with plenty of distilled water. The second Step was the Preparation of Passivation Bath. The passivation bath was prepared as follows; 10 wt. % citric acid was taken in a beaker. Distilled water was added to make a 100 ml solution. After that the cleaned sample was dipped in this passivation bath. The beaker containing sample (dipped in the passivated bath) was heated in an electric oven at 150 °F (65 °C) for 30 minutes. After heating the sample was again cleaned as described in the step-I (excluding no.2 & 3.). The third Step was Testing. The sample was scrubbed with soap and water and then immersed in the ethyl alcohol for few seconds for the purpose of cleaning. A chemical solution of Copper Sulphate (4g), Sulphuric acid (10ml), and distilled water (90 ml) was made in a beaker. After cleaning the sample was dipped in the Copper Sulphate solution for 6 minutes at room temperature. It should then be immersed in the Copper Sulphate solution for 6 minutes at room temperature. The Copper Sulphate solution will react with any free iron on the surface of the jewellery or the passivated sample, and plate out copper on them, any reddish discoloration of the surface. In table 1 Passivation parameters for the stainless steel alloys in carbonate melt at different temperatures The images of samples used in this study has been captured which have been shown in figure 1.

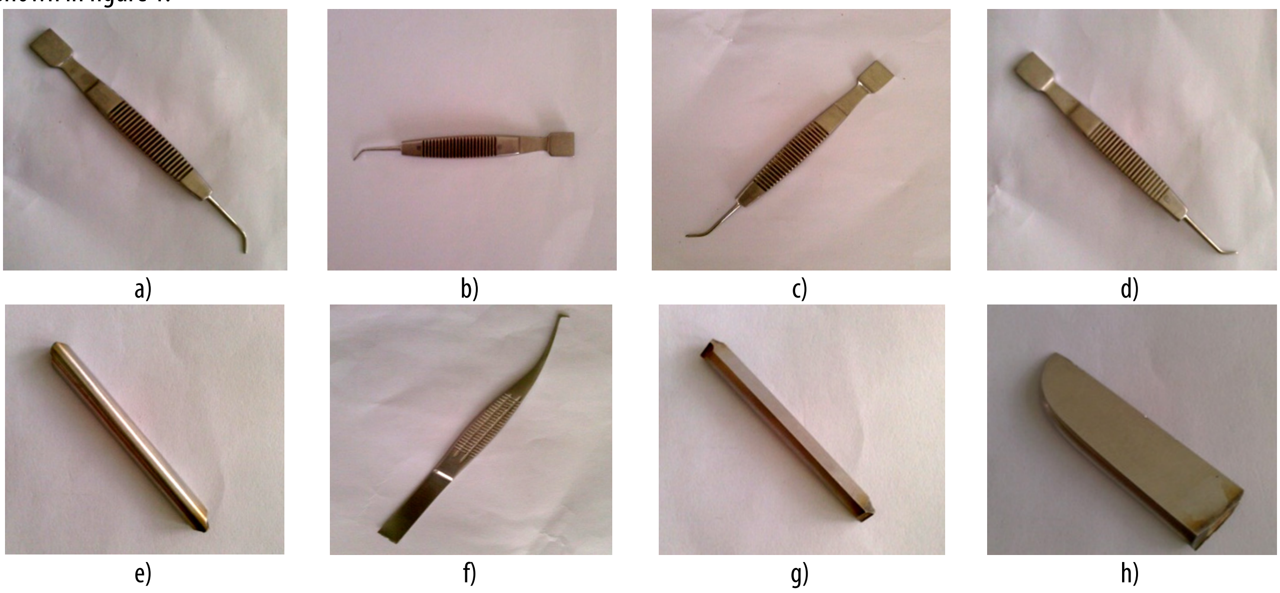

Figure 1 (a- h. Samples 1-8 before adopting the passivation behavior

Table 1: Passivation parameters for the stainless steel alloys in carbonate melt at different temperature

Serial no.	Time	Temperature	Chemicals
Sample no.1	30 min	150 F	10 wt. % Citric Acid, Distilled Water, 95% Ethyl Alcohol, Copper Sulphate ($CuSO_4$-$5H_2O$) 4g, Sulfuric Acid ($H_2 SO_4$) 10g.
Sample no.2	30 min	120-140 °F (48-60 °C)	20 vol. % Nitric Acid (HNO_3), Distilled Water, 95 % Ethyl Alcohol, Copper Sulphate $CuSO_4$-$5H_2O$) 4g, Sulfuric Acid ($H_2 SO_4$) 10g.
Sample no.3	30 min	150° F	50 wt. % Citric Acid, Distilled Water. 95% Ethyl Alcohol, Copper Sulphate ($CuSO_4$-$5H_2O$) 6g, Sulfuric Acid ($H_2 SO_4$) 10g.
Sample no.4	30 min	200 °F (93°C)	10 wt. % Citric Acid, 95% Ethyl Alcohol, Copper Sulphate ($CuSO_4$-$5H_2O$) 4g, Sulfuric Acid ($H_2 SO_4$) 10g, Distilled Water.
Sample no.5	30 min	120-140°F	10 wt. % Citric Acid, 5% NaOH, Distilled Water, 95% Ethyl Alcohol, Copper Sulphate ($CuSO_4$-$5H_2O$) 4g, Sulfuric Acid ($H_2 SO_4$) 10g.
Sample no.6	30 min	120-140 °F	20 vol. % Nitric Acid (HNO_3), Sodium Dichromate ($Na_2Cr_2O_7$), Distilled Water, 95% Ethyl Alcohol, Copper Sulphate (CuSO4-5H20) 4g, Sulfuric Acid (H2SO4) 10g.
Sample no.7		120-140 °F	30 wt. % Citric Acid, 5% NaOH, Distilled Water, 95% Ethyl Alcohol, Copper Sulphate ($CuSO_4$-$5H_2O$) 4g, Sulfuric Acid ($H_2 SO_4$) 10g.
Sample no.8	30 min	120-140°F	50 vol. % Nitric Acid (HNO_3), Sodium Dichromate ($Na_2Cr_2O_7$), Distilled Water, 95% Ethyl Alcohol, Copper Sulphate ($CuSO_4$-$5H_2O$) 4g, Sulfuric Acid ($H_2 SO_4$) 10g.

3. RESULTS AND DISCUSSION

Water Immersion Test verifies the passivation process on all grades of stainless steel, with exception of 440C. After testing, specimens do not show no signs of rust or corrosion. Salt Spray Test is performed in accordance with ASTM B117 for 2 hours, when specified by engineering drawings. After testing, specimens also do not show no signs of rust, stain or corrosion. Surface of each sample has also been represented in figure 2 (a-h).

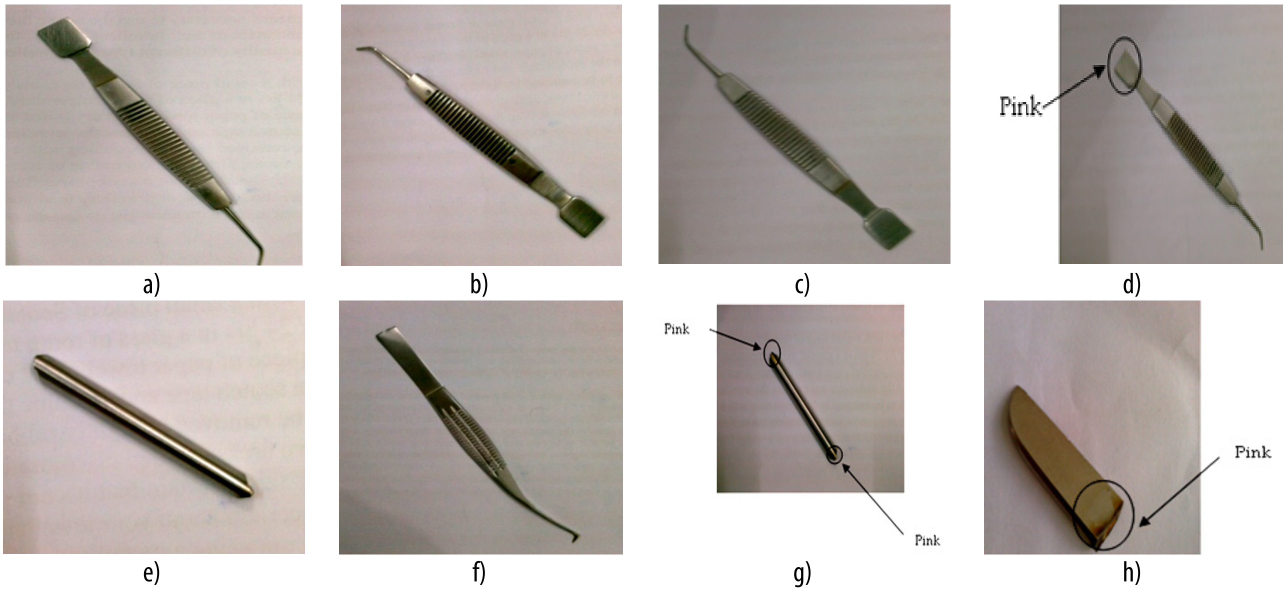

Figure 2 (a- h). Samples (1-8) after passivation behavior

Passivation and surface behavior of each sample has been described. Sample 1 shows the surface of the sample was quite bright. No red discoloration or any pink spot on the sample was observed. This shows that the passivation on the specimen was done successfully. Thus, the samples passivated in the standard solution of 10 wt. % citric acid work satisfactorily. Sample 2 shows the surface of the sample was quite bright. No red discoloration or any pink spot on the sample was observed. This shows that the passivation on the specimen was done successfully. Thus, the sample was passivated in the standard solution of 20 vol. % nitric acid. Sample 3 shows the surface of the sample was quite bright. No red discoloration or any pink spot on the sample was observed. This shows that the passivation on the specimen was done successfully. Thus, the sample was passivated in the standard solution of 50 wt. % citric acid. Sample 4 shows a red discoloration on the welded portion of the sample was observed. The possible reason for pink spots may be that the cleaning of the welded portion of the samples was not done properly. Sample 5 shows the surface of the sample was quite bright. No red discoloration or any pink spot on the sample was observed. This shows that the passivation on the specimen was done successfully. Thus, the samples passivated in the standard solution of 10 wt. % citric acid work satisfactorily. Sample 6 shows the surface of the sample was quite bright. No red discoloration or any pink spot on the sample was observed. This sample is passivated in the standard solution of 20 % by volume nitric acid .The sample was cleaned and passivated properly. So No pink spots appear on the surface of sample. This shows that the passivation on the specimen was done successfully. Sample 7 shows the Sample has pink spots at the edges. This sample was passivated in a concentrated solution of citric acid. 30 wt. % citric acid was taken for passivation. Increased amount of acid should support the passivity but unfortunately due to improper cleaning pink discoloration appeared. This is because the sample was not cleaned properly at the edges to welding because the edges are welded portions of the sample. Sample 8 shows the sample has pink spots at the edge. This sample was passivated in a concentrated solution of nitric acid. 50 vol. % nitric acid was taken for passivation. Increased amount of acid should support the passivity but unfortunately due to improper cleaning pink discoloration appeared. This is because the sample was not cleaned properly at the edges to welding because the edges are welded portions of the sample.

CONCLUSIONS

All the chemicals and the key variables used for the passivation of surgical steels samples were found satisfactory. The testing of the passivated samples was carried out using Copper Sulphate testing. Most of the samples did not show any pink spots except a few one. Thus, it may be concluded the passivation processes used during the present work are satisfactory. Pink spot observed on a few samples may be due to improper cleaning. It is suggested that samples should be cleaned properly prior to the passivation treatment. These surgical steels are strong biomaterials for bioinstrumentation and other promising applications like as dentistry and orthopedics.

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