تأثير معالجة الأسطح ميكانيكياً على تآكل الصلب المقاوم للصدأ AISI 316-Ti في أوساط من الكلوريدات

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الخلاصة

تعتبر المكافحة والسيطرة على عملية التآكل من أهم الجوانب الأساسية للحفاظ على جودة المنتجات الهندسية. في هذا البحث تم التحقق ودراسة تأثير عدة أنواع من المعالجات السطحية الميكانيكية (التجليخ، التنظيف بحبيبات الجارنت، التنظيف بالكور المعدنية) على قابلية الصدأ للصلب المقاوم للصدأ AISI 316-Ti . تم قياس خشونة السطح والطاقة السطحية قبل التعرض للوسط المؤثر والمؤدي للتآكل. تم استخدام كل من كلوريد الصوديوم، ومحلول كلوريد الحديد $FeCl_3$ وخليط بينهما كوسائط مؤثرة ومؤدية للتآكل. وأثناء الدراسة قد تم إبقاء تركيز كلوريد المستمر ثابتا بينما تم اعتبار متنوع الأكسدة المحتملة كمتغير. وشملت إجراءات الاختبار استخدام طرق الغمر و مقاومة التحليل الطيفي الكهروكيميائية . تم فحص السطوح المتآكلة من العينات باستخدام المجهر الضوئي والماسح الضوئي، و جرى تقييم أحجام وكثافة النقرات المتكونة. أكدت النتائج أنه يوجد تأثير كبير للمعالجات السطحية الميكانيكية على سلوك تآكل الفولاذ المقاوم للصدأ تحت تأثير أنواع مختلفة من الكلوريد. وفقا لنتائج هذا البحث، أظهرت عينات مع الأسطح أن عملية المعالجة بالتجليخ تعطى أفضل مقاومة للتآكل بينما في حالة المعالجة بحبيبات الجارنت والكور المعدنية فان العينات تكون أكثر عرضة لمعدل عالي من التآكل.

Mechanical surface treatments effects on corrosion of AISI 316 Ti stainless steel in chloride environments

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ABSTRACT

The current paper investigates effects of various surface treatment techniques such as grinding, garnet blasting, and shot peening on the corrosion rate and behavior of austenite stainless steel of type AISI 316 Ti. The exposure to different corrosive solutions usually accompanying the coastal and industrial environments (sodium chloride and ferric chloride), as well as a combination of the two was considered. The corrosion behavior of AISI 316 Ti under these test conditions was investigated using immersion tests and electrochemical impedance spectroscopy together with optical scanning electron microscopy, in order to observe and to assess the changes in the surface configuration and topography such the shapes, distribution, and dimensions of the resulting pits. The presented results clearly show the relatively higher corrosive effect of ferric chloride, and the increased corrosion rate under greater surface roughness values, which imply greater real surface area and capillarity effects.

Keywords: Chloride environment; corrosion; mechanical surface treatments; pitting; stainless steel.

INTRODUCTION

Quality, durability and long-term stability of engineering products are increasingly in demand that requires continuous efforts to enhance their performance. Austenitic stainless steel, in addition to its distinct mechanical properties (high ductility and high toughness) and weldability, exhibits excellent resistance to many corrosive environments. It has shown high reliability and durability in demanding applications such as marine industries, chemical and petroleum refining, and medical implants (Sridhar *et al.*, 2003; Singh *et al.*, 2002). The excellent corrosion resistance of austenitic stainless steel is because it has a stable, thin and well-adhered passive layer (consisting of chromium oxide and maybe iron oxide).

Pitting is the main type of corrosion affecting stainless steel (Zatkalíkova *et al.*,

2010). Besides, causing degradation of product quality, corrosion propagation usually causes contamination and pollution. This is particularly problematic when stainless steel is used in food and medical implant applications, as the contaminants can jeopardize human health. Pitting may result from the interaction of chloride ions with the passive surface of stainless steel, or it may be the result of anodic surface defects such as inclusions and secondary phases (Liptakova, 2009).

Pitting resistance of stainless steel usually depends on many factors including the corrosive environment, chemical compositions and structure of the material, the surrounding temperature and the surface quality of the component (Liptakova, 2009). Resistance of austenitic stainless steel to pitting, in the presence of chloride or other halide ions, can be improved by increasing chromium (Cr), molybdenum (Mo), and/ or nitrogen (N) content (Pitting Resistance Equivalent: PRE = % Cr + 3.3×% Mo + 16×%N) (Department of Defense, 1993). This explains why the molybdenum-bearing austenitic stainless steels (such as AISI 316) have better pitting resistance than ordinary chromium-nickel austenitic stainless steel. In addition, adding about 0.5 % of titanium (to give AISI 316 Ti, for example) usually offers the advantage of prolonged endurance at elevated temperatures especially for manufacture of heavy, welded, sections (Liptakova, 2009).

Surface treatment of austenitic stainless steel has been a major interest of many industries and researchers. Surface conditions strongly affect the passive layers and consequently the corrosion rate (Zatkalíkova *et al*., 2010). Mechanical treatments of the surface can cause different arrangements of atoms in the surface plane, (Sedlacek, 1992) hence affecting surface properties of the material. Surfaces may be treated chemically (Li *et al*. 2005), mechanically (Ben Rhouma *et al*., 2001; Zhao *et al*., 2002) or by a combination of the two (Zatkalíkova & Liptakova, 2011; Fajnor *et al*., 2010). Common mechanical surface treatment techniques include grinding, polishing, brush cleaning, buffing and pressure abrasive cleaning. The aim is always to clean and remove the existing scale traces on the surface, to ensure effective adhesion of coatings, and to improve mechanical properties such as ability to withstand wear, stress-corrosion cracking and fatigue (Jiang *et al*., 2006). Other reported techniques to improve surface properties include solid solution hardening (Pelletier *et al*., 2002) and grain refinement (Fujiwara & Ameyama, 1999; Ucok *et al*., 1991). These techniques, which involve higher temperatures, allow a martensitic transformation to occur. However, the presence of a considerable amount of martensite may negatively affect the corrosion resistance of AISI 316 (Harvey, 1982). Another approach, surface mechanical attrition treatment (SMAT), has been used to refine austenitic grains while retaining the austenitic structure of AISI 316 (Chen *et al*., 2005).

In the present study, the aim is to investigate the effects of different mechanical surface treatments on the corrosion behavior of AISI 316 Ti under different chloride environments. Sodium chloride solution was used to simulate the coastal environment

and ferric chloride solution was used to simulate environmental conditions experienced by many industrial structures. A combination of the two solutions was also tested. Three mechanical surface treatments were used in this study: grinding, garnet blasting and shot peening. Surface energy, roughness and real area measurements were performed prior to corrosion testing. Corrosion behavior of AISI 316 Ti under the three different chloride solution environments was evaluated by: immersion tests and electrochemical impedance spectroscopy (EIS). Optical and scanning electron microscopy (SEM), were used to examine the exposed surfaces. The shapes, distribution and dimensions of pits were noted and characterized.

MATERIALS AND METHODS

Chemical composition (wt.%) of the employed austenitic stainless steel of type AISI 316 Ti is as following: 16.5 Cr; 10.6 Ni; 2.12 Mo; 1.69 Mn; 0.012 N; 0.41 Ti; 0.04 C; 0.43 Si; 0.026 P; 0.002 S and balance Fe. The material was supplied in 1500×1000 mm sheets with 1.5 mm thickness. The sheet was cold-cut into smaller samples, 30×80 mm in the same direction. As shown by Figure 1, during immersion tests, each sample was hung to a glass rod at one end and by using a pre-prepared 4 mm drilled hole at the other immersed end, where the center of the hole was 6 mm away from the upper edge of the rectangular sample. Edges and corners of all samples were wet ground with 320 grit silicon carbide paper to avoid concentrated pitting corrosion.

Three groups of samples are prepared according to the treatment performed. The first group of samples was prepared by dry grinding using 320 and 500 grit size silicon carbide paper in the transverse and longitudinal directions, respectively. Garnet blasting (garnet composition: 31% SiO_2, 21.6% Al_2O_3, 37% FeO, 7.4% MgO) was performed on the second group of samples using a pressure of 0.4 MPa. The garnet's maximum grit size was 0.2 mm; the blast was aimed perpendicular to the sample and the sample was positioned 220 mm from the nozzle. The third group of samples were shot peened at a pressure of 0.4 MPa using austenitic stainless steel balls with maximum diameter 0.2 mm. The peening was also directed perpendicular to the sample and the samples were held 220 mm away from the nozzle. For all categories, samples were then properly cleaned and immersed with distilled water and degreased with diethyl ether
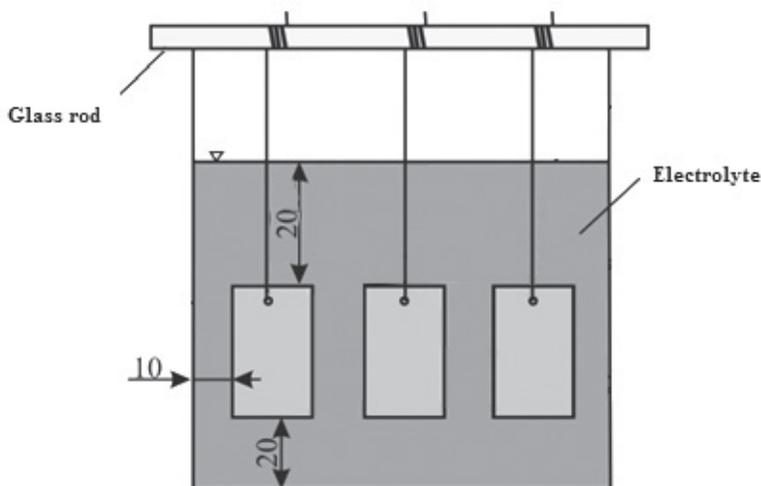
Fig. 1. Immersion test apparatus, showing samples and minimum
distance requirements (in mm.)

To examine the effects of surface properties on pitting corrosion, surfaces of samples were characterized after mechanical treatment prior to exposure to corrosive solutions. These measurements included determination of surface roughness and surface free energy. A MarSurf PS1 device was used to determine the surface average roughness (Ra) values for both the longitudinal and transverse directions of the samples. Surface free energies were measured using a surface energy evaluation (SEE) system (Advex, Czech Republic) and analyzed based on the Owen/Wendt method, using its operating software. From the state of the liquid on the tested material, the SEE determines a contact angle, which can be converted into surface energy. The recorded results were based on the average angle values for water and ethylene glycol drops on three samples for each surface finish.

Corrosion behavior was examined using immersion tests and electrochemical impedance spectroscopy. Immersion tests were conducted using 5% NaCl solution (according to ASTM G46), 4.6% $FeCl_3$ solution (according to ASTM G48) or a 1:1 mixture of 5.000% NaCl and 4.600% $FeCl_3$ solutions. The chloride concentrations of these solutions were kept constant at (3.032 g Cl /100 g of solution) with different redox potentials. Redox potential was measured using a Mettler Toledo InLab Redox electrode, which also measured pH. Samples were weighed on an analytical balance (Mettler Toledo XS 205 DU/M) with an accuracy of ± 0.00001 g. Three samples for each category of mechanical surface treatment were suspended by insulated wires onto a glass rod for 24 hours exposure at room temperature, 22 ± 1°C (Figure 1). All samples were placed in the same container having the same chloride solution. The minimum distances shown in Figure 1 were maintained to ensure that samples did

not interfere with the container or other samples. These procedures were repeated for the different chloride solutions. The sample surfaces were then properly cleaned and immersed in distilled water and dried thoroughly before the samples were reweighed using the same analytical balance. The masses before and after performing the immersion test were used, along with the surface area of the sample and the exposure time, to calculate the corrosion rate in (g. m^{-2}. h^{-1}).

Electrochemical impedance spectroscopy (EIS) was carried out on a VoltaLab device (Radiometer Analytical with measuring unit PGZ 100) to obtain the polarization resistance. Two samples were tested for each electrolyte-surface treatment group. The amplitude of the alternating voltage was 10 mV with response time potential between the sample and the electrolyte of 10 minutes. A saturated calomel electrode (SCE) was considered as the reference electrode. The exposed area was 1 cm^2 and the frequency range was 100 kHz to 10 mHz. Temperature was maintained at 22 ± 1 °C.

Additional measurements and evaluations were carried out to clarify and to explain the outcomes of the surface characterization and corrosion resistance measurements. The density and size of pits were evaluated according to ISO 11463 using both optical microscope and SEM equipped with energy dispersive x-ray (EDX) technology. For optical microscopy, samples were prepared by grinding (up to grit size 1000), polishing (up to 0.7 micron) and etching (10 mL HNO$_3$ + 30 mL HCl + 30 mL glycerin - dipped in for about 3 minutes).

RESULTS AND DISCUSSION

Surface characterization tests

Surface roughness has a very strong effect on corrosion resistance because it determines the real surface area (in other words the concentration of reactive metal) and also creates micro-crevices (Zatkalíkova *et al*., 2010; Smialowska, 2005). The most common way of characterizing roughness, the arithmetic average deviation of the profile, Ra, was measured for all treated surfaces. Table 1 lists values of the surface roughness measured in both longitudinal and transverse directions. Grinding produced the best surface quality, while the garnet-blasted surface was slightly better than the shot peened one. For each treated surface, an Olympus Lext OLS 3100 microscope was used to estimate the ratio between the real and the original assumed flat surface areas. Surface area increases were approximately 8.6, 9.0, and 35.6 fold for grinding, garnet blasting and shot peening, respectively.

According to Ben Rhouma *et al*., (2001), for given operating conditions, the surface with only grinding treatment exhibits better roughness than that produced by blasting as a second stage surface treatment. However, stress corrosion cracking usually existed, when grinding treatment was used independently while this was not observed when blasting treatment was carried out.

Table 1. Roughness measurements (Ra) [in µm] in longitudinal and transverse directions for different mechanical surface treatments.

Roughness measurement Ra [µm.]	Grinding	Garnet blasting	Shot peening
In longitudinal direction	0.151	3.446	4.072
In transverse direction	0.185	3.199	3.931

The surface energy usually affects corrosion, because it influences the adsorption of reactants on the metal surface (Sadlacek, 1992). Surface free energy (SFE) was evaluated for each treated surface, and the results are shown in Table 2. Surfaces with larger values of free energy demonstrated better resistance to pitting in NaCl solution. The ground surface had the highest surface energy, while the garnet-blasted surface had the lowest value. Since adhesion largely depends on the surface property of the material, it is necessary to modify its near-surface properties, without affecting the properties of the bulk material. The properties and thickness of the passive layer are the important factors that control the integrity of such a layer (Kerber & Tverberg, 2000). The passive layer is formed by oxidation of both chromium and iron. Austenitic stainless steel which has Molybdenum may stabilize and enhance the passive layer.

Table 2. Measured surface free energies.

Surface treatment	Average contact angle-water	Average contact angle-ethylene glycol	Surface free energy [MJ/m²] Owen/Wendt method (Rudawska & Jacniacka 2009)
Grinding	52.95	32.69	46.92
Garnet blasting	95.30	64.91	31.57
Shot peening	58.52	33.91	42.43

Corrosion

Immersion tests

The redox potentials values for the three solutions employed in the study are listed in Table 3. Decreasing the concentration of $FeCl_3$ reduces the redox potential value and increases the pH value. Solutions with low pH and higher redox potential are expected to be more active in terms of pitting corrosion. This was verified by the corrosion rates from the immersion tests for untreated and treated sample surfaces. The sodium chloride solution used has a low redox potential and a neutral pH value; thus it is expected to exert a negligible corrosion attack on all the stainless steel surfaces within 24-hrs duration exposure, regardless of the treatment method employed. Moreover,

even when the exposure time was extended up to 21 days, no corrosion attack was visually detected.

Of the various treatment methods, the ground surfaces showed the smallest amount of corrosion, with corrosion losses for the ground surfaces being even lower than those of the untreated surfaces. As shown by Figure 2, in addition to data listed in Table 3, for all immersion solutions, the highest corrosion rate results when shot peening and garnet blasting surface treatment methods are employed, while a better corrosion rate results for the untreated surface. When ferric chloride solution is employed, about 21 to 50% corrosion increase is observed over that emerged on the untreated samples, while a corresponding range of about 110-160% increase is recorded when grinding treated surfaces are considered. However, when a combination of ferric chloride and sodium chloride solution is employed, only 40-50% corrosion rate reduction results for surfaces treated by grinding. Between surfaces which are prepared with either shot peening or garnet blasting methods, the former prevails lower corrosion resistance. Also, the ground surface also had the highest surface energy, which is consistent with a lower corrosion rate. While minimal corrosion rate was found in NaCl solution, much more corrosion was observed when $FeCl_3$ solution was used. For all solutions employed, the corrosion rates of shot-peened surfaces were greater than those for either ground or garnet-blasted surfaces.

Table 3. Corrosion rates (measured in immersion tests, g. $m^{-2}.h^{-1}$) of samples with different surface treatments including pH and redox potentials for different electrolytes.

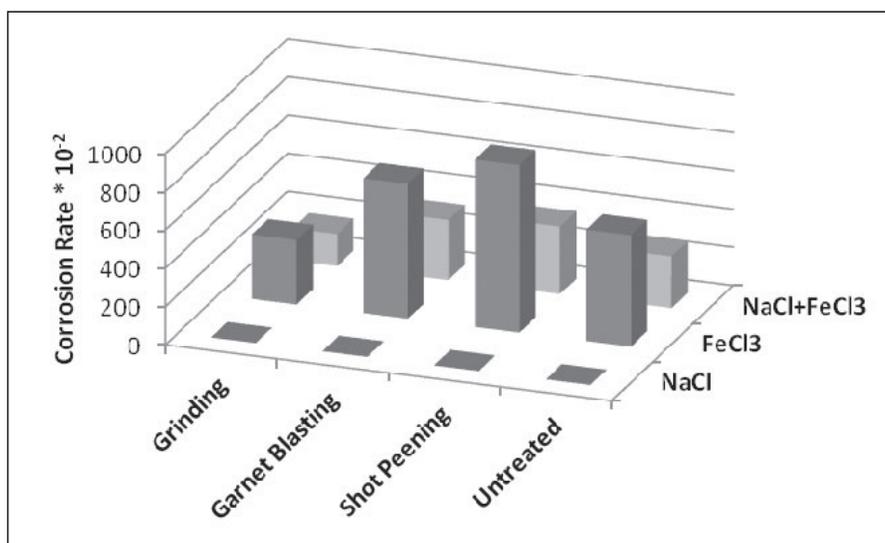|	Grinding	Garnet blasting	Shot peening	Untreated surface	Exposure time	pH	Redox potential [mV]
NaCl	0.0000	0.0247	0.0098	0.0000	21 days	7.27	300
$FeCl_3$	3.3786	7.0461	8.7501	5.8265	24 hours	1.20	674
Mix	1.6879	3.1409	3.5343	2.7082	24 hours	1.51	671

Fig. 2. Corrosion rate results from immersion test for different mechanical treatment-solution types.

Electrochemical impedance spectroscopy (EIS)

To monitor the behavior of the protective layer on the stainless steel when subjected to corrosive environments, the three groups of samples were examined using EIS to determine polarization resistances (R_p, values shown in Table 4). In NaCl solution, which has a neutral pH and a low redox potential, all treated surfaces showed their largest values of polarization resistance, revealing better resistance to local corrosion attack and the presence of an adherent passive layer. In contrast, resistance to local corrosion was reduced in electrolytes with lower pH and higher redox potentials; under these conditions the passive layer has poor protective properties. The ground surfaces showed greater resistance to local corrosion than the other mechanically treated surfaces. The behavior of the samples during EIS tests was somewhat different to that observed in the immersion tests. In all environments, the corrosion rate observed in the immersion test was affected by the real area, which, in turn, was related to surface free energy (SFE); the effect of the surface free energy itself on the polarization resistance was only slight.

Table 4. Polarization resistance R_p (measured in ohm. cm^2) of various samples with various surface treatments immersed in different electrolytes.

	Grinding	Garnet blasting	Shot peening
NaCl	118.2×10^3	17.8×10^3	11.3×10^3
FeCl$_3$	86.48	59.47	31.38
Mix	185.9	84.35	66.79

Pit dimensions and microscopy after immersion tests

To evaluate the density of corrosion and the pit size, the ISO 11463, 1995 standard was applied. The standard template was employed where notation A is for corrosion density while B is for pit size; the scale runs from 1 for the smallest to 5 for the largest densities and sizes. Three 1×1 cm^2 areas were marked on each sample and the average results of these areas are reported in Table 5. No pitting was observed for any of the treated surfaces in the sodium chloride electrolyte. In the other two electrolytes, the least pitting was seen on the ground surface. Very similar extents of pitting were seen for the garnet-blasted and shot-peened surfaces in solutions containing Fe^{3+} ions. Untreated surfaces in electrolytes containing Fe^{3+} ions showed the highest pit density but the smallest pit size.

Table 5. Pit densities and sizes for samples with different surface treatments in different electrolytes.

Solutions	Grinding	Garnet blasting	Shot peening	Untreated surface
NaCl	-	-	-	-
FeCl$_3$	1A 1B	3A 2B	3A 2B	4A 1B
Mix	1A 1B	2A 2B	2A 2B	3A 1B

Cross-sectional optical micrographs (Figure 3), indicated the depth of corrosion attack for each environment/surface treatment (or untreated) combination. Of all electrolytes examined in this study, the most severe attack was observed in FeCl$_3$, while the solution containing NaCl without Fe^{3+} ion produced almost no corrosion attack. Also, the ground samples showed less corrosion than samples with the other two surface treatments. The presence of Fe^{3+} ion in electrolyte, either separately or in combination with NaCl, accelerated corrosion attack of all samples. The pitting corrosion was strongly affected by the low pH of the solution and the oxidizing ability of Fe^{3+}. With decreasing pH and increasing redox potential of the corrosion environment, the influence of surface treatment was diminished.

Changes in surface and subsurface material were examined using a JEOL JSM 7600F scanning electron microscope (SEM) with EDX technology. Corrosion

products such as oxides, corrosion initiators like chlorides, and foreign particles are particularly relevant to pitting corrosion. Figure 4 shows titanium carbides and oxides on ground samples, which were exposed to sodium chloride electrolyte, while Figure 5 shows oxides and carbides on ground samples after immersion in $FeCl_3$ electrolyte. Also, Figure 6 shows the presence of oxides and, clearly, titanium carbide for ground samples after exposure to the mixed electrolyte. All samples with other surface treatments show different formations of oxides and carbides, as well as foreign particles such as silicon, aluminum and magnesium. These particles are believed to be residual garnet and grinding grit on the surfaces (Figure 7 and Table 6). The chemical compositions of the surfaces of the tested samples confirm their high inhomogeneity (Sadlacek, 1992).
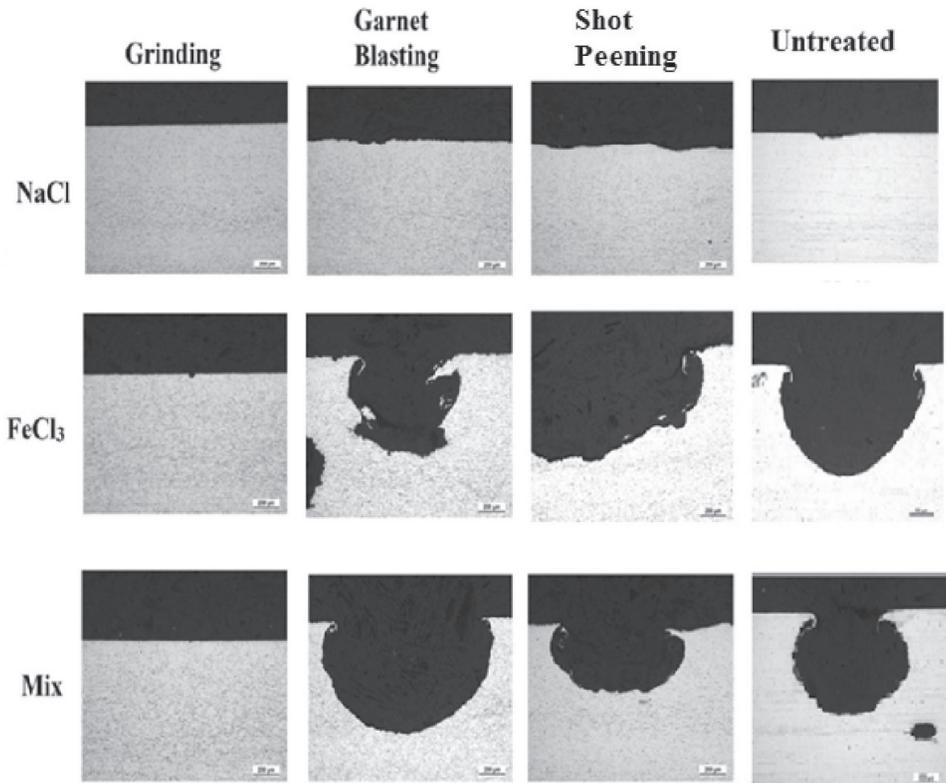
Fig. 3. Cross-sectional optical micrographs for each electrolyte/surface treatment and untreated combinations all scale bars represent 200 μm.
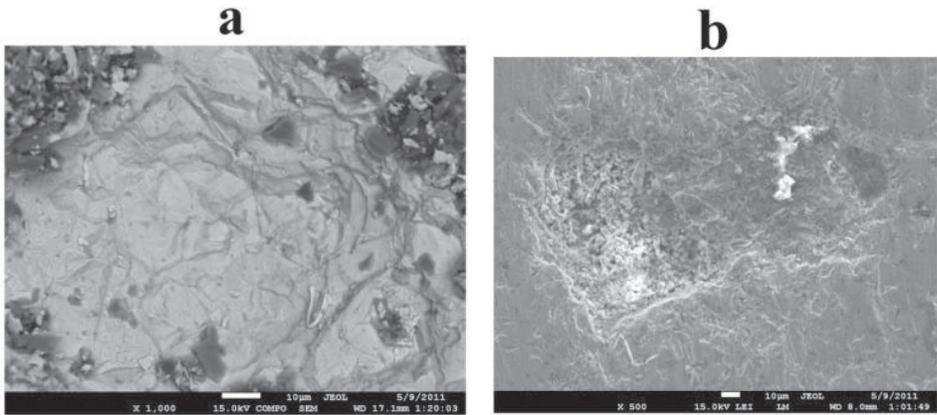
Fig. 4. SEM images of ground samples exposed to NaCl showing presence of (a) titanium carbide (scale bar represents 10 µm) and (b) oxides (scale bar represents 10 µm).
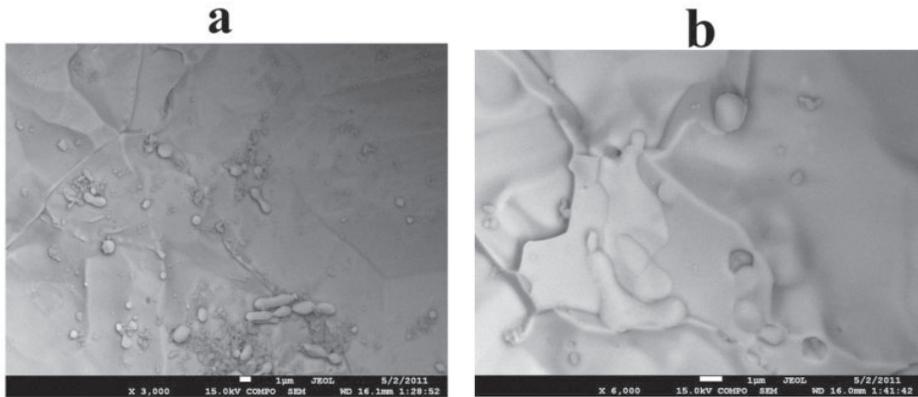
Fig. 5. SEM images of ground sample exposed to FeCl₃ showing presence of oxides and carbides. (a) scale bar represents 1 µm and (b) scale bar represents 1 µm.

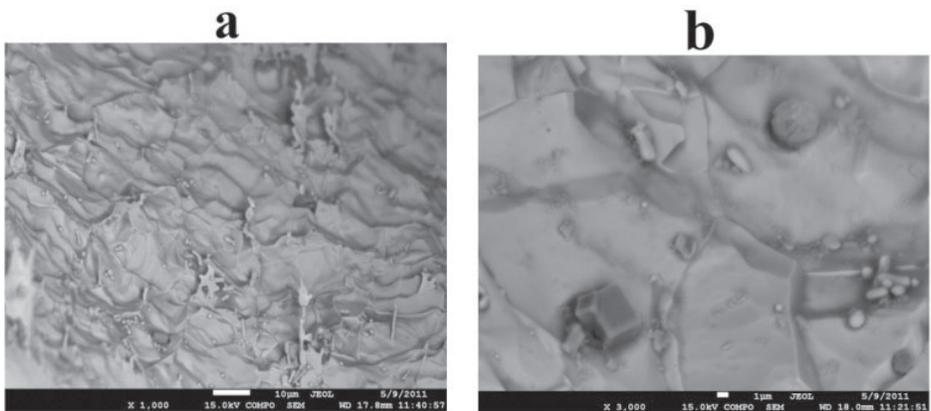**Fig. 6.** SEM images of ground sample exposed to mixed electrolyte, showing presence of (a) oxides (scale bar represents 10 µm) and (b) titanium carbide (scale bar represents 1 µm).
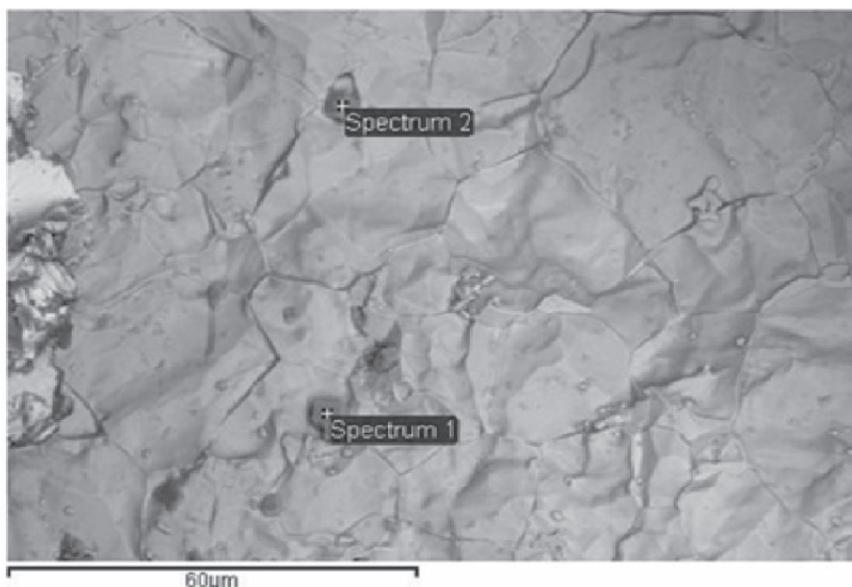
Fig. 7. SEM of garnet-blasted sample exposed to FeCl$_3$ electrolyte (scale bar represents 60 μm).

Table 6. EDX result of garnet blasting sample exposed to FeCl$_3$ electrolyte (wt.%)

Spectrum	C	N	O	Al	Ti	Cr	Fe	Ni	Total
1	3.83	12.88	7.76	0.64	66.85	2.33	5.53	0.19	100
2	11.84	11.60	5.61	0.39	65.24	1.49	3.44	0.40	100

Indentations due to abrasive grits from garnet blasting and shot peening may cause the initiation of pitting in large scale cavitations. This can be seen in Figure 8(a) for garnet blasting and in Figure 8(b) for shot peening; both surfaces were exposed to FeCl$_3$ electrolyte. Pitting was propagated by the dissolution of metal and maintenance of a high degree of acidity in the bottom of the pit.
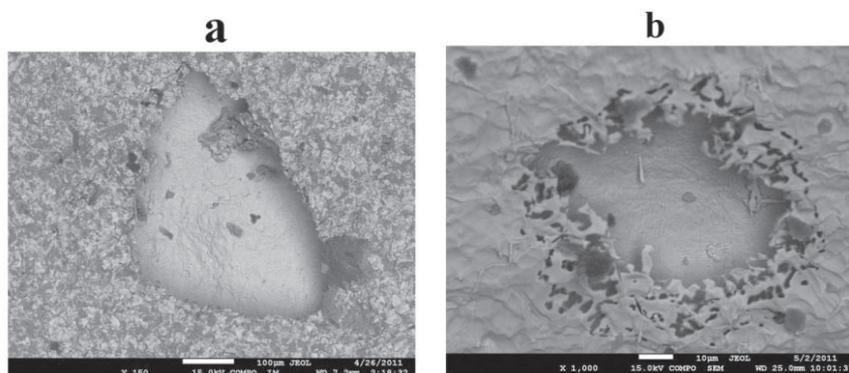
Fig. 8. Corrosion pits after exposure in FeCl$_3$ electrolyte of (a) garnet-blasted sample (scale bar represents 100 μm) and (b) shot-peened sample (scale bar represents 10 μm).

CONCLUSIONS

The surface treatment of AISI 316 Ti significantly influences corrosion behavior in various aggressive environments. Among the employed treatment methods, samples with ground surfaces showed the best corrosion resistance over those treated by garnet-blasting and shot peening, considering all immersion solutions used. Due to the enhanced surface quality obtained by grinding, samples exhibit better corrosion resistance than that of untreated samples.

The severity of the corrosion depended on the exposed environment, where all tested surfaces corroded less in NaCl solution than in other solutions. The trend in extents of corrosion in NaCl solution for the various treated surfaces was consistent with their values of surface free energy, with the greatest corrosion occurring in the sample with the lowest surface free energy. The ground surface had the highest surface energy while garnet blasting gave the lowest value. However, for all treated surfaces, corrosion rates in solutions containing Fe^{3+} ions are of greater values, as expected from the oxidizing nature of these solutions (redox potential). Moreover, in the existence of such aggressive Fe^{3+} ions, a surface with higher rough surface values (and therefore greater real surface area and capillarity effects) shows greater corrosion rates. This indicates that the concentration of Fe^{3+} ions solution is the major controlling (influential) parameter to determine the corrosion level and rate. This confirms that the corrosion mechanisms are different with different Fe^{3+} ions concentration. The surface area influences the reduction rate of Fe^{3+} ions which accelerates oxidation of metal and thus corrosion.

Samples treated with garnet blasting and shot peening show different formations of oxides and carbides in addition to the presence of extraneous particles containing elements such as silicon, aluminum and magnesium. These particles are believed to be deposited on the sample surfaces due to the garnet grits. In addition, indentations from the abrasive grits of garnet blasting and shot peening may cause initiation of pitting in large scale cavitations. Pitting was propagated by the dissolution of metal and maintenance of a high degree of acidity at the bottom of the pit.

However, the offered results are obtained according to standard testing procedures and, they may be adopted to consider the practical conditions and environment. This may be the basis for a future investigation in which the effect of each of temperature as well as the different surface quality grades offered by grinding are considered. This may include the concentration of the ferric chloride solution.

ACKNOWLEDGEMENT

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* تهدف إلى معالجة المشكلات المعاصرة والقضايا المستجدة من وجهة نظر الشريعة الإسلامية.

* تشمل موضوعاتها معظم علوم الشريعة الإسلامية: من تفسير، وحديث، وفقه، واقتصاد وتربية إسلامية، إلى غير ذلك من تقارير عن المؤتمرات، ومراجعة كتب شرعية معاصرة، وفتاوي شرعية، وتعليقات على قضايا علمية.

* تنوع الباحثون فيها، فكانوا من أعضاء هيئة التدريس في مختلف الجامعات والكليات الإسلامية على رقعة العالمين: العربي والإسلامي.

* تخضع البحوث المقدمة للمجلة إلى عملية فحص وتحكيم حسب الضوابط التي التزمت بها المجلة، ويقوم بها كبار العلماء والمختصين في الشريعة الإسلامية، بهدف الارتقاء بالبحث العلمي الإسلامي الذي يخدم الأمة، ويعمل على رفعة شأنها، نسأل المولى عز وجل مزيداً من التقدم والازدهار.