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Tribocorrosion behaviors of 304SS: Effect of solution pH

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ABSTRACT: The tribocorrosion behavior of 304SS in chloride-containing solutions with different pH values (pH 7.2 ~ 9.2) was investigated under rubbing conditions of 50N, 100 R min⁻¹ on a pin-on-disc tribometer with an Al₂O₃ pin mounted on a vertical rotating axis. When combined with rubbing, the corrosion of 304SS is accelerated due to the formation of galvanic couples between the mechanical depassivated areas and the surrounding passivated areas. Besides, the continuous sliding-over processes also make the pitting corrosion take place more easily due to the affinity of Cl⁻ to metal cations very near the active surface. However, OH⁻ owns better affinity than Cl⁻, so high pH solution mitigates the local corrosion with wear track. It is found that high pH solution exhibits good lubricity, and thus both friction coefficient and total material loss reduce obvious with increasing pH. From the calculation results and wear morphologies, we can confirm that pure mechanical and corrosion-accelerated wear, including abrasion and delamination, are among the chief reasons for material loss.

1. Introduction

As one of the familiar materials, austenitic stainless steels often perform excellent corrosion resistance due to its ability to spontaneously form a protective oxide film on the surface and become passive.¹⁻³ Such passive film, however, is often susceptible to localized breakdown when exposed to halide ions containing solution, notably the chloride ions, resulting in accelerated

dissolution of the underlying metal.⁴⁻⁶ The corrosion behavior of austenitic stainless steels are mainly determined by alloy composition⁷⁻¹¹ and solution chemistry characteristics which include temperature^{10,11}, chlorinity^{10,12-15} and aggressive solution classification and concentration^{12,16}, etc. Among them, the effect of pH on the corrosion susceptibility of austenitic stainless steels has been investigated in considerable detail by numerous researchers and is reviewed in a number of articles, reviews and books.^{12,14-20}

Malik et al. found that with increasing pH, the corrosion rate of 316L steel decreases in 1000 ppm Cl^- aqueous solution, being highest at pH 4 and lowest at pH 9.¹⁵ Recently, Liu and Wu found that in 3.5% NaCl solution, the acidity of the electrolyte (pH 0.8 ~ 5) influences the passivation performance of highly alloyed austenitic 254SMO stainless steel, in terms of the film's composition, structure, growth-rate and cations dissolution behavior, but has no effect on pitting resistance of the passive film.²¹ Similarly, earlier studies have also shown that the critical potential is not greatly affected in acid 0.1 mol L⁻¹ NaCl with pH ranging from 1 to 7; in alkaline solutions, pH 7 ~ 10, however, the critical potential is reported to shift in the noble direction on addition of NaOH to 0.1 M NaCl, which directly corresponds to increased resistance to pitting; above pH 10, the potential also moves in the active direction as pH increases, which is due to the formation of CrO_4^- instead of reflecting the pitting resistance.^{12,22}

Recently, special attention has been paid to investigate the role of pH on the corrosion susceptibility of metals and alloys during various friction and wear processes, including abrasion, fretting and erosion etc. in chloride containing corrosive solutions, because during these processes, the mass loss of materials is often not a simple sum of loss caused by mechanical wear and corrosion determined respectively but somewhat exceed this sum.²³⁻²⁸ So, the influence of pH on corrosion or wear resistance is bound to be reflected by tribocorrosion behavior of the materials. Licausi et al. found that during sliding friction and wear, the i_{corr} is three times higher in the acidic solution (pH 3) than in the neutral (pH 6) and basic (pH 9) ones for both cast and sintered alloys. However, at OCP, the total degradation of the cast alloy is independent on the pH, while for the sintered material it slightly increases with the pH.²⁹ Meantime, Mathew did not accept these results and proposed that the total degradation peaks at pH 6 because at this pH value, the reformed

passive film layer is not cohesive and the tribocorrosion products are easily sheared off in the presence of motion.³⁰ Unfortunately, little work has been carried out to investigate the influence of pH on corrosion, wear, and following tribocorrosion behavior of austenite stainless steels in chloride-containing solution.

It is well known that austenite stainless steels (e.g. 304 and 316 steels) perform excellently as marine engineering materials for the distinguished general and even pitting corrosion resistances. However, tribocorrosion is an important and widespread phenomenon encountered in many practical engineering situations. When they undergo various contact motions, such as impacting, rubbing and rolling, the good anti-corrosion properties will degrade due to the destruction of protective passive film on metal surface.^{25,31–34} Besides, the performance of passive film is also constricted to its formation conditions, such as the chloride concentration, temperature, and pH. Indeed, the pH value is changing in different seawater environments and always ranges from 7.5 to 8.6. So, in this paper, a wider range of pH values (7.2 ~ 9.2) is intended to investigate its effect on tribocorrosion behavior of 304SS in chloride-containing electrolytes.

2. Experiment

2.1. Materials

The 304 austenitic stainless steel (304SS) in ring form (Φ_{out} 54 mm, Φ_{in} 37 mm) was used in the tests, and its composition is listed in **Table 1**. Prior to tribocorrosion tests, each sample was ground with SiC grinding papers (from grade 600 to 1500 grit), degreased ultrasonically in acetone, cleaned with distilled water, dried with N_2 . Before immersed into tribocorrosion cell, all surfaces except the upper-surface were sealed with insulating glue.

Table 1. Chemical composition of 304SS.

Elements	Cr	Si	Mo	Ni	Mn	C	P	S	Fe
Content / wt%	18.50	0.59	0.30	8.12	0.88	0.05	0.015	0.028	Bal.

2.2. Solution preparation

Analytical reagents and double-distilled water were used to prepare the artificial seawater referenced to ASTM D1141–98. Its chlorinity is 19.38, and pH was adjusted by adding 0.1 mol L⁻¹ NaOH when it would be used. In this work, the pH values of tested solutions were 7.2, 7.7, 8.2, 8.7 and 9.2, equivalent to OH⁻ ions concentrations ranging from 1.58×10⁻⁷ to 1.58×10⁻⁵ mol L⁻¹.

2.3. Tribocorrosion measurement

The tribocorrosion tests were carried out on a modified pin-on-disc tribometer and its schematic view of components was the same as already described in the previous publication (35). During the tests, a rotating alumina pin with a diameter of 4 mm slid against stationary 304SS specimen under the lubrication of 400 mL seawater with different pH values. It is worth noting that all tests, in the absence or presence of corrosion, were carried out under fixed frictional parameters of 50 N, 100 R min⁻¹. The friction coefficient was measured by an attached strain gauge and recorded by computer acquisition system. The mass loss was obtained by weighing the sample before and after each test. In addition, cathodic protection technology was performed by applying cathodic potential of 0.7 V to the free corrosion potential (E_{corr}), to eliminate the electrochemical corrosion during rubbing, and therefore it was possible to assess separately the role of corrosion and wear in the total degradation of material, and to evaluate the synergy between them. After tribocorrosion tests, the worn surface morphologies of 304SS samples were examined by scanning electron microscope (SEM, JEOL 5600, Japan).

The electrochemical characteristics of 304SS with and without rubbing in artificial seawater were studied using a conventional three-electrode cell and the assembly of electrodes was also described in Ref. 35. Potentiodynamic polarization tests were performed at a scanning rate of 2 mV s⁻¹ from -250 mV to +1000 mV with respect to E_{corr} of the steel. Upon the electrochemical results several electrochemical parameters, such as the free corrosion potential (E_{corr}), the pitting potential (E_{pit}), and the corrosion current density (i_{corr}), were calculated and listed in Table 1. The corrosion rate with (C , mm y⁻¹) and without (C_0 , mm y⁻¹) wear were also calculated according to ASTM G 102–89. It is worth noting that all tests were carried out at least three times at room temperature open to the

air, and the average values were reported in this paper.

2.4. Calculation

The mass loss rate and the synergistic effect between corrosion and wear in different solutions are calculated according to ASTM G119–09.

Based on C , and total mass loss (T), the wear rate with corrosion (W , mm y^{-1}) can be calculated by the following equation:

$$W = T - C \quad (1)$$

Thereupon, combining with the pure wear rate (W_0 , mm y^{-1}) and the pure corrosion rate (C_0 , mm y^{-1}) obtained from the static Tafel curve, the wear increment (ΔW_c , mm y^{-1}) and the corrosion increment (ΔC_w , mm y^{-1}) can be deduced as follows:

$$\Delta W_c = W - W_0 \quad (2)$$

$$\Delta C_w = C - C_0 \quad (3)$$

Thence, the synergistic effect between mechanical and electrochemical material loss can be described by Eqs. (4) and (5):

$$S = T - W_0 - C_0 \quad (4)$$

$$S = \Delta W_c + \Delta C_w \quad (5)$$

Besides, three dimensionless factors, that is, the total synergism factor, corrosion augmentation factor, and wear augmentation factor, are used to depict the synergism degree of corrosion and wear.

The total synergism factor is:

$$\frac{T}{T - S} \quad (6)$$

The wear augmentation factor is:

$$\frac{W_0 + \Delta W_c}{W_0} \quad (7)$$

The corrosion augmentation factor is:

$$\frac{C_0 + \Delta C_w}{C_0} \quad (8)$$

3. Results and discussion

3.1. Effect of pH on electrochemical corrosion

The potentiodynamic polarization tests were carried out to investigate the influence of pH values on electrochemical and tribo-electrochemical corrosion of 304SS, and the related corrosion parameters are listed in **Table 2**.

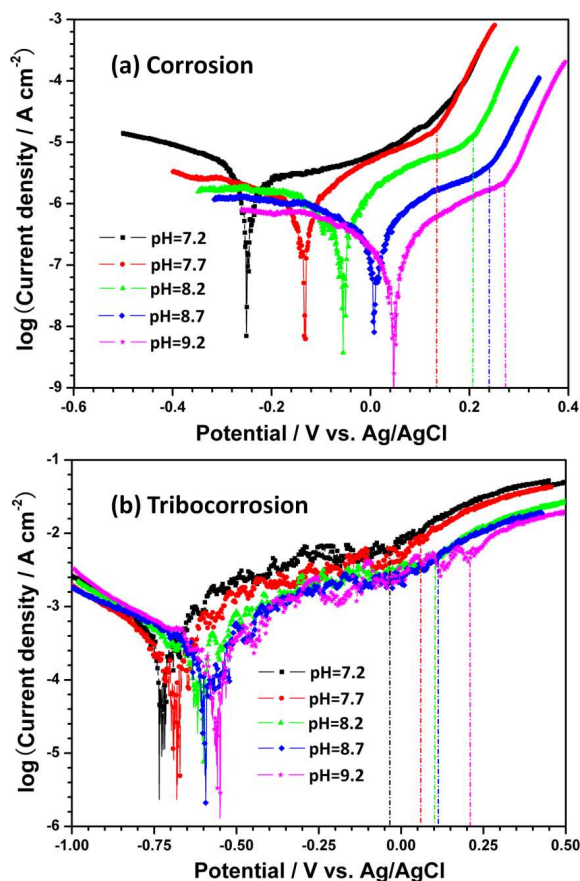


Fig. 1 Potentiodynamic polarization curves of 304SS obtained in different solutions (a) without and (b) with mechanical rubbing.

Fig. 1a shows the typical potentiodynamic polarization curves for 304SS in alkaline, chloride

solutions with different pH values. Consistent with the conclusions established previously,¹² both the free corrosion potential (E_{corr}) and the pitting potential (E_{pit}) shift obviously towards the noble direction with increasing pH values, indicating that high OH^- concentration inhibits the occurrence of corrosion for 304SS.

Table 2. Electrochemical parameters for 304SS in different pH seawater.

pH	Corrosion					Tribocorrosion				
	7.2	7.7	8.2	8.7	9.2	7.2	7.7	8.2	8.7	9.2
$E_{\text{corr}} / \text{V}_{\text{Ag}/\text{AgCl}}$	-0.234	-0.137	-0.070	-0.001	0.034	-0.730	-0.691	-0.604	-0.598	-0.567
$E_{\text{pit}} / \text{V}_{\text{Ag}/\text{AgCl}}$	—	0.136	0.199	0.233	0.265	0.009	0.058	0.096	0.111	0.208
$E_{\text{pit-corr}} / \text{V}_{\text{Ag}/\text{AgCl}}$	—	0.273	0.269	0.234	0.231	0.739	0.749	0.700	0.709	0.775
$i_{\text{corr}} / \mu\text{A cm}^{-2}$	2.30	0.99	0.83	0.76	0.18	347.0	337.31	300.07	227.50	209.35
$\beta_c / \text{mV dec}^{-1}$	-51.69	-54.15	-54.13	-54.06	-56.75	-63.32	-59.40	-61.26	-56.62	-60.64
$\beta_a / \text{mV dec}^{-1}$	26.99	48.37	50.78	49.35	48.55	65.04	66.16	63.73	57.99	63.31

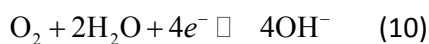
It is worth noting that, though it is difficult to distinguish the pitting potential for 304SS when polarized in pH 7.2 solution, pitting did happen as proven by the thickly dotted pits found on the surface. Besides, between pH 7.7 ~ 9.2, the E_{pit} is found to be a function of pH values. By linear regression, the following relation is established:

$$E_{\text{pit}} = 0.084\text{pH} - 0.503 \quad (9)$$

This equation can be derived on the view that competitive adsorption occurs between OH^- and Cl^- for sites on the passive surface. As discussed in detail by Kolotykin,^{4,36} the initiation of pitting is a process, where Cl^- ions constantly adsorb and accumulate on the passive surface of alloys by displacing adsorbed oxygen, until a sufficient concentration corresponding to the critical potential, Cl^- ions succeed at favored sites in destroying passivity. However, when OH^- ions are present, they

also tend to adsorb on the passive surface and displace Cl^- ions, due to the better affinity of OH^- ions for passive surface of 304SS than Cl^- ions.^{15,36,37} Hence, with the presence of OH^- ions, the initiation of pitting requires a shift of potential towards more positive values in order to enable Cl^- ions to reach a concentration adequate enough to break the protective passive film locally. And the higher the OH^- concentration, the more notable the shift of pitting potential for 304SS towards positive direction.

In addition, the i_{corr} also increases apparently with the decrease of pH values. When 304SS sample is immersed in pH 7.2 electrolyte, the i_{corr} increases over one order of magnitude compared with that in pH 9.2 solution. It is well known that in neutral or alkaline solution, the cathodic reaction of electrochemistry corrosion is dominated by the reduction of dissolved oxygen occurring on the passive surface as shown in reaction (10).



High OH^- concentration is adverse to shift reaction equilibrium to positive direction. In other words, high OH^- concentration may restrain this oxygen reduction reaction which provides the driving force for the anodic dissolution of 304SS. Therefore, a smaller and smaller i_{corr} for 304SS is displayed as the solution pH increases from 7.2 to 9.2.

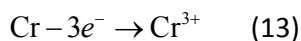
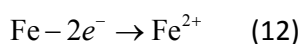
Meantime, in the case of tribocorrosion for 304SS as shown in Fig. 1b, E_{corr} , E_{pit} and i_{corr} present the same variation trend with increasing solution pH, that is, E_{corr} and E_{pit} become more positive, and i_{corr} decreases gradually with increasing pH. However, there is an essential difference between the course of the polarization of 304SS during static corrosion and tribocorrosion. The combination of corrosion and rubbing significantly shifts E_{corr} and E_{pit} towards more negative direction with respect to those in the absence of rubbing. Under our conditions, the change of pitting potential of 304SS with increasing pH follows the law below:

$$E_{\text{pit}} = 0.090\text{pH} - 0.643 \quad (11)$$

Besides, a larger difference between E_{corr} and E_{pit} (represented symbolically by $E_{\text{pit-corr}}$ and the values are listed in Table 2) is observed than that obtained from static corrosion experiment at the same pH, which should be attributed to the constant removal–recovery of passive film on 304SS

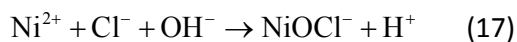
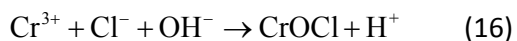
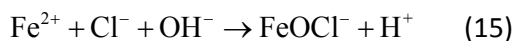
during the periodic sliding–over processes.

It has been well established that, the sliding contact between 304SS and Al₂O₃ must destroy the completeness of passive film and make the electrochemically active surface with a lower equilibrium potential exposed to corrosive solution. Firstly, this partial destruction of protective film initiates the occurrence of galvanic corrosion as previously described by Lucas,³⁴ and shifts the E_{corr} to the negative direction. Additionally, the results that i_{corr} increases by even four orders of magnitude compared with that obtained under static corrosion should also be attributed to the establishment of the galvanic couples between the mechanical depassivated areas (anode) and the surrounding passivated areas (cathode). Most importantly, the continuous sliding–over processes also make the pitting corrosion take place more easily. As discussed above, the dissolution of active surface is actuated by rubbing processes in seawater as described by reactions (12–14).



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As a result of these reactions, the electrolyte very near the active surface gains positive electrical charge relative to the surrounding electrolyte. Then, the anions (Cl⁻, OH⁻, SO₄⁻, etc) are attached and migrate to active surface so as to get charge balance, and some even react with metal cations to form Fe²⁺, Cr³⁺ and Ni²⁺ chlorides at the anode areas or pits, destroying passivity and preventing its recurrence by dissolved oxygen as follows:^{38–41}



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pH of the electrolyte very near the active surface decreases due to the formation of H⁺, which causes further acceleration of corrosion of 304SS and even stimulates pitting corrosion of 304SS by nucleation of more metastable pits as demonstrated by Burstein and Sasaki.⁴² However, the survival of these metastable pit below the pitting potential is determined by the perforated cover over the

pit mouth by providing an additional diffusion barrier which enables the concentrated pit solution to be maintained.⁴³ But the continuous rubbing processes must rupture or even remove the perforated cover, and make the process of pit growth displays a wide range of amplitude in a certain scale of potentials under tribocorrosion, evidenced by the larger $E_{\text{pit-corr}}$ values.

On the basis of the experimental data reported above, it is clear that rubbing may significantly increase the susceptibilities of general corrosion and pitting corrosion, as well as corrosion rate. We can therefore draw the conclusion safely that the electrochemical signals are governed by the continuous depassivation caused by rubbing. Besides, the pH values of chloride-containing electrolytes also have distinguished influence on the electrochemical signals.

3.2. Effect of pH on friction and wear

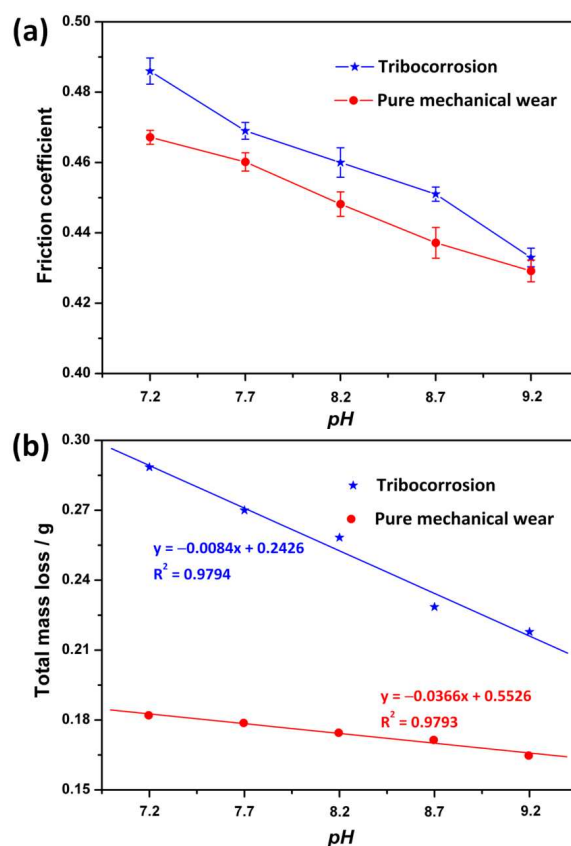


Fig. 2 Friction coefficient (a) and total mass loss (b) curves of 304SS in different solutions.

To study the effects of solution pH on the friction and wear behavior of 304SS, the friction

coefficient was determined. As illustrated in **Fig. 2a**, corrosion significantly increases friction coefficient, because its occurrence may deteriorate the characteristics of contact surfaces between 304SS sample and Al_2O_3 pin,³⁵ resulting in an increased friction coefficient and accelerated total material loss (Fig. 1b) compared with those under pure mechanical wear. Besides, through linear regression analysis, we obtain the relevant equations of the solution pH and the total materials loss in the absence and presence of corrosion as shown in Fig. 2b. It is clear that, corrosion reactions interact with mechanical friction under our conditions to show a positive synergistic effect, and result in an accelerated total mass loss. More than that, high pH solution performs good lubricity as well as good corrosion inhibition, so the resulting surface deterioration of 304SS mitigates with increasing pH, and the corresponding friction coefficient and total material loss show obvious reduction.

3.3. Morphologies of worn surfaces

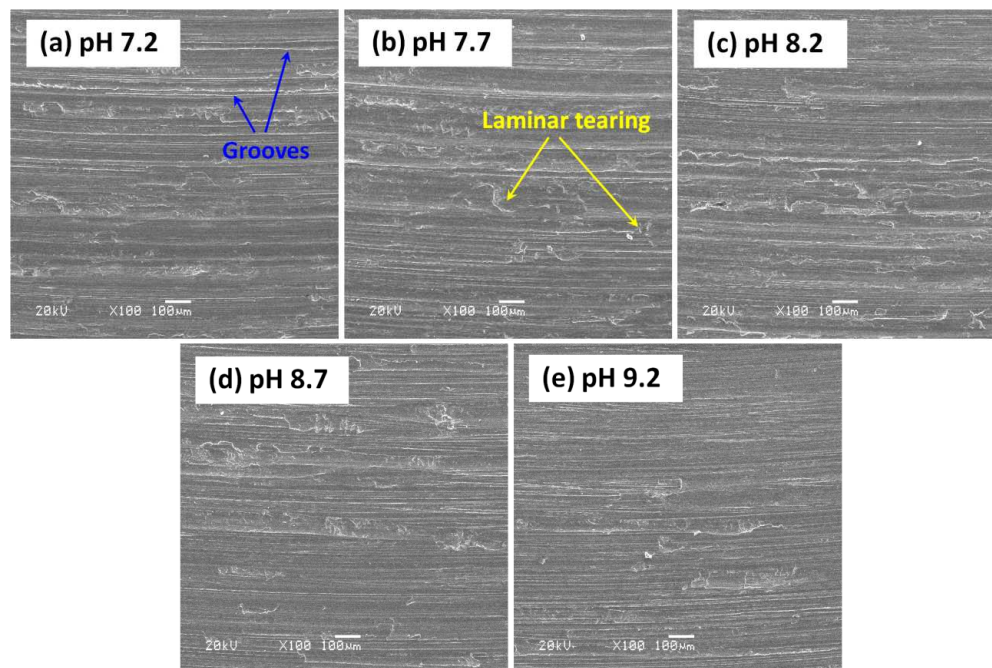


Fig. 3 pH dependence of worn surface morphologies of 304SS after tribocorrosion in different solutions.

In order to determine the wear mechanism between 304SS and Al_2O_3 tribocouples lubricated with

different solutions, the morphologies of worn surfaces were examined using a scanning electron microscope. **Fig. 3** reveals that the roughness of worn surface decreases obviously with solution pH increasing, which coincides with the change of friction coefficient mentioned above. This decrease of roughness proves that different pH will make great influence on 304SS surface characteristics, especially roughness. Besides, many grooves, corresponding to typical abrasion morphology, are observed on the tested surface over the whole range of solution pH. And, the more alkaline the solution, the more shallow the grooves due to better lubrication of high pH solution. Meanwhile, laminar tearing also appears, manifesting that delamination wear also prevails. Consequently, the combined action of abrasion and delamination leads to the very high and continuously increased material loss of 304SS in the process of tribocorrosion.

3.4. Synergy between corrosion and wear

In order to ascertain the synergistic effect between corrosion and wear in different pH solutions, a large amount of computation was performed, and the results are shown in **Fig. 4**.

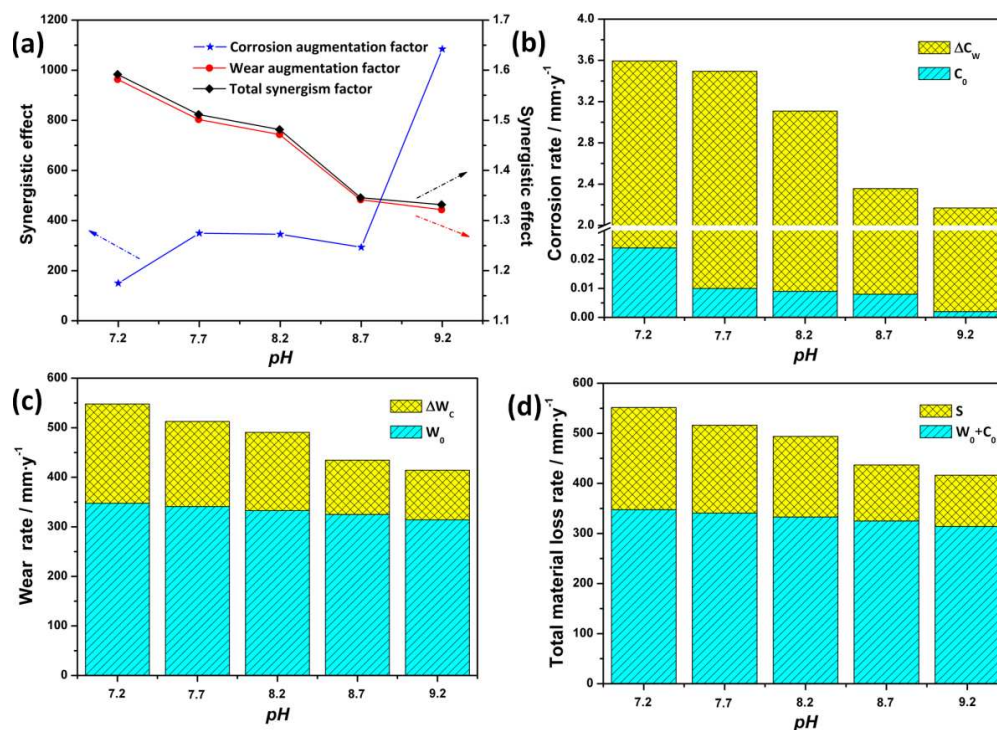


Fig. 4 Synergistic contributions of mechanical wear and corrosion to each other and to total mass

loss of 304SS in different solutions.

Fig. 4a displays the synergistic effects between corrosion and wear, which were calculated according to equations (6)–(8). It is evident that corrosion and wear are claimed to interact and accelerate each other, evidenced by all three dimensionless factors bigger than 1, and the augmentation extent of wear to corrosion is much greater. With increasing pH, the acceleration of corrosion due to wear increases; rather, the acceleration of wear by corrosion decreases gradually as shown in Fig. 4a. The opposite acceleration tendencies can be explained in the following two respects. On the one hand, corrosion can accelerate wear in that its occurrence may deteriorate the characteristics of contact surface between 304SS sample and Al₂O₃ pin. In our situation, with increasing pH, the absolute corrosion rate decreases apparently with the presence of rubbing in chloride solutions (Fig. 4b), so the corresponding surface deterioration mitigates, and presents a declined acceleration of wear. On the other hand, the absolute wear rate decreases gradually with increasing pH (Fig. 4c), for higher alkaline solution owns better lubricity and makes the rubbing process more smoothly as was pointed out above. Consequently, under the same mechanical condition, after each rubbing–over process the fresh wear track areas which correspond to the damage areas of regenerated passive film within wear track are small in high pH solution. In other words, during the periodic removal–regeneration processes, the completeness of the passive film is in a state of dynamic balance, and the area ratio of passive surface to mechanically depassivated surface, corresponding to cathode–to–anode area ratio (A_c/A_a), is a quasi– constant when 304SS experiences tribocorrosion in the same pH solution.

It is well established that in a galvanic cell, the apparent external current is nil at OCP.²⁹ Considering the cathodic i_c and anodic i_a current densities, one equation can write for the case of tribocorrosion experiments:

$$i_a A_a = -i_c A_c \Leftrightarrow -i_a/i_c = A_c/A_a \quad (12)$$

where A_a and A_c correspond to the mechanically depassivated areas and the passive areas which include the surrounding passive surface and the regenerated passive surface within wear track, respectively. Hence, the absolute value of anodic–to–cathodic current density ratio ($-i_a/i_c$) in the

galvanic cell is determined by the cathode-to-anode area ratio (A_c/A_a). As described above, the more alkaline the solution, the smaller the anode area, and therefore a bigger A_c/A_a value. However, as mentioned above, high OH^- concentration will inhibit the occurrence of corrosion for 304SS. So, affected jointly by corrosion inhibition and lubricity, the absolute value of i_a/i_c and subsequent corrosion rate presents a downward trend as pH increases (Fig. 4b), but the relative increment of corrosion rate peaks at pH 9.2 (Fig. 4a). More importantly, for the selection of severe mechanical parameters, the changing trends of total synergism factor and the total material loss rate are remarkably consistent with those of wear (Fig. 4a, c and d), demonstrating that the problem of material loss mainly comes from pure mechanical wear and corrosion-accelerated wear in our conditions.

4. Conclusions

In this paper, the pH dependence of tribocorrosion behavior for 304SS/ Al_2O_3 tribocouples in seawater was studied on a pin-on-disc tribometer. Within the whole studied ranges of pH (pH 7.2 ~ 9.2), corrosion and wear are claimed to accelerate each other. However, high pH solution performs both good lubricity, and excellent anti-corrosion and anti-pitting properties. So with solution pH increasing, corrosion rate and wear rate decrease. Besides, delamination and abrasion wear mechanism coexist when the 304SS is matched with Al_2O_3 under 50 N, 100 R min^{-1} condition and the abrasion becomes mild with the increase of solution pH. Of particular note is that the occurrence of rubbing may shift the pitting potential to more negative direction, and it also makes 304SS more easily suffer from corrosion.

Acknowledgements

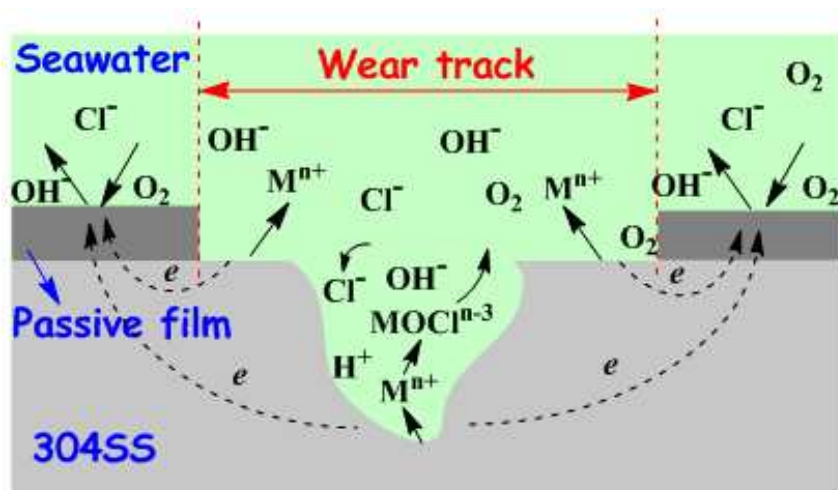
This work was financially supported by the National Natural Science Foundation of China (Grant No. 51405478), and National Basic Research Program of China (973 Program, Grant No. 2014CB643302). The authors also gratefully thank the reviewers for their detailed, rigorous and helpful comments.

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Graphic Abstract

Tribocorrosion behaviors of 304SS: Effect of solution pHYue Zhang,^{a,b} Xiangyu Yin,^{a,b} Yunfeng Yan,^a Jianzhang Wang^a and Fengyuan Yan^{*a}

The tribocorrosion behaviors of 304SS in chloride-containing solutions are pH dependent, which can affect both the corroding and the wearing behaviors, and eventually determines the total material degradation.