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Improvement in the corrosion-erosion resistance of 304 stainless steel with alloyed yttrium

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It has been previously demonstrated that yttrium can improve the resistance of stainless steel to sliding wear in corrosive environment. However, the mechanism responsible for the beneficial effect of yttrium on corrosive wear is not well understood. In this work, the erosion behavior of Y-free and Y-containing 304 stainless steel in a dilute H_2SO_4 slurry containing silica sand was investigated, with the aim of exploring the role that yttrium plays. The failure and self-healing of passive films on Y-free and Y-containing 304 stainless steel samples under combined attack by corrosion and mechanical scratch were studied, using an electrochemical scratch technique. The failure of the passive films during dry scratch and identation was also investigated using a micro-mechanical probe with *in situ* monitoring changes in the electrical contact resistance. In addition, effect of yttrium on electron work function was investigated. All the results demonstrated that yttrium effectively improved properties of the passive film, including corrosion resistance, mechanical behavior and the film stability, resulting in enhanced resistance to corrosion-erosion.

1. Introduction

Stainless steel is widely used in oil production, chemical, and mining industries to make hydro-turbines, pumps and pipelines, because of its combination of good corrosion resistance and mechanical properties [1, 2]. The corrosion resistance of stainless steel is attributed to the formation of passive film, which protects the material from continuous corrosion attack. However, when subjected to the attack combining corrosion and erosion, the passive film could be damaged by solid particle impingement, resulting in exposure of bare metal surface to the corrosive medium and thus increasing the corrosion-erosion rate. Approaches for resisting the synergistic attack of erosion and corrosion include alloying elements and surface modification [3–10]. Usually, corrosion-erosion of stainless steel involves the failure and recovery of its passive film. Mechanical properties of the passive film and the substrate all affect the erosion damage. The interfacial bonding strength is also an important factor affecting the protective function of the passive film. If the bonding between the passive film and the substrate is weak, the impingement of solid particles may easily remove the passive film during corrosion-erosion, and the resultant fresh metal surface is vulnerable to continuous corrosion-wear attack. Therefore, both the mechanical properties and interfacial bonding strength of the passive film are of importance to the material against corrosion-erosion.

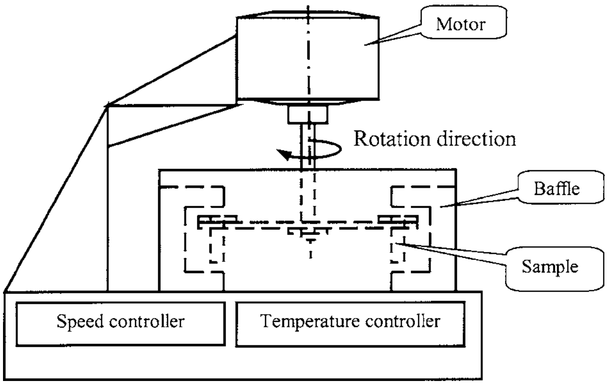
Yttrium is beneficial to the oxidation resistance of Cr- and Al-containing alloys, due to its high oxygen affinity that helps to form a protective oxide film [11, 12]. Yttrium can reduce the oxidation rate and suppress lateral growth of the film [13–15], which may diminish the internal stress and thus oxide buckling and interfacial failure. Yttrium also enhances the interfacial bonding by developing oxide pegs that penetrate into grain boundaries [12], by forming second phases of Y-Al garnets, and by reducing outward cation diffusion [16, 17]. The enhancement in oxide adherence would be beneficial in resistance to oxidative wear. Indeed, yttrium can enhance the resistance of 304 stainless steel to dry sliding wear [18], during which the flashing temperature could be high enough to induce local oxidation and thus oxidative wear. In the case of corrosive wear, the addition of yttrium also helps to diminish corrosive sliding wear of stainless steel in dilute acid [18] and corrosion-erosion of aluminide coatings in both acidic and salty solutions [10]. It has been shown that yttrium has little effect on mechanical properties of the materials but it significantly improves their corrosion resistance [18]. Therefore, modification of passive film could be the main role that yttrium plays. There are two possible mechanisms responsible for the beneficial effect of yttrium. One could be the improvement in the passive film adherence. If the bonding between the passive film and the substrate is enhanced by yttrium, rupture of the protective oxide film would be reduced and this may help to diminish further corrosive erosion. The other could be possible enhancement in the mechanical properties of the passive film.

The objective of this study was to investigate the corrosion-erosion behavior of Y-containing and Y-free stainless steels in dilute H_2SO_4 slurry mixed with silica sand, with the intention to explore the mechanisms involved. The failure of passive films on Y-free and Y-containing 304 steel samples during corrosive scratch, mechanical scratch and indentation was investigated. Electron work functions of the samples were also measured for better understanding of yttrium effects on properties of the passive film.

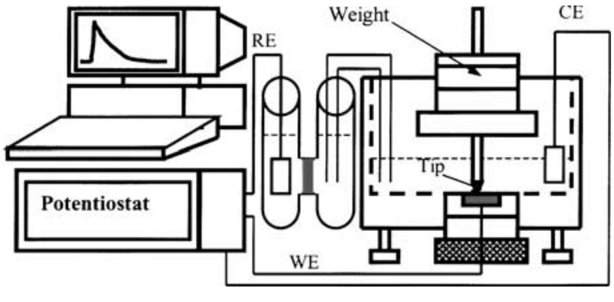
2. Experimental procedure

Steel specimens for erosion test were prepared from commercial 304 stainless steel powder using an induction-melting furnace. The steel specimens contained 19 wt% Cr, 9 wt% Ni, 2 wt% Mn, 1 wt% Si, 0.08 wt% C, 0.04 wt% P, and 0.03 wt% S. 1 wt% pure yttrium powder was alloyed into some of the specimens. The specimens having a size of $40 \times 5 \times 5$ mm^3 were mechanically ground using emery paper up to 600-grit.

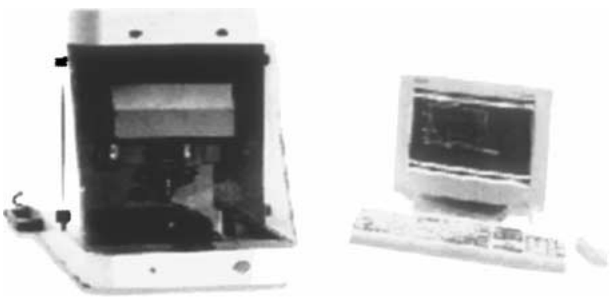
The corrosion-erosion test was performed at room temperature using a slurry-pot tester illustrated in Fig. 1a. The pot was made of stainless steel with volume capacity of 9 liters. Four vertical baffles were placed in the pot to break up the rotational flow. The slurry was prepared by mixing 30 wt% silica sand with 0.1 mol/l H_2SO_4 solution. A specimen was weighed before and after the erosion test to determine its weight loss. The specimen velocity relative to the slurry was varied from 0.7 m/s to 6.1 m/s. The erosion loss was then evaluated under different velocity and at a constant distance of 10 Km.



a. Slurry pot corrosive erosion tester



b. Electrochemical scratching tester



c. Universal micro-tribometer

Figure 1 Several tribological testers used in the present study.

To better understand the corrosion-erosion behavior of the Y-free and Y-containing stainless steel specimens, an electrochemical scratch tester, illustrated in Fig 1b, was employed to evaluate the resistance of the materials to corrosive scratch. During this test, a Vickers diamond tip scratched the surface of a sample that was immersed in a 0.1 mol/l H_2SO_4 solution. The current response under a constant applied electrochemical potential was measured using a Gamry framework commercial corrosion measurement system. Variations in the transient current provided the information about failure and self-healing of the passive film. A saturated calomel electrode (SCE) was used as the reference electrode, and a piece of platinum was used as an auxiliary electrode. Specimens having a size of $5 \times 5 \times 5$ mm^3 for the scratch test were mounted with epoxy resin and polished using emery paper up to 600-grit.

A CETR universal micro-tribometer (see Fig. 1c) was used to evaluate the resistance of a passive film to mechanical scratch and indentation. The tip was a pyramidal tungsten carbide. During the micro-scratch test or the micro-indentation test, the tip moved either horizontally or vertically under a force that was increased linearly from 0 to a designed level. The velocity of the tip was 0.02 mm/s and the duration of indentation and scratch processes was 30 s and 60 s, respectively. Failure of the passive film during micro-indentation and micro-scratch was determined by monitoring variations in the electrical contact resistance between the indenter tip and the target sample surface.

A scanning Kelvin probe was used to measure electron work functions of passive films respectively formed on Y-free and Y-containing 304 steel samples. The work function is related to the inertness of a material to electrochemical attack. The measurement was performed by applying a 5-volt DC potential to the sample and detecting the output signal via an amplifier connected to an Au electrode tip. The average peak-to-peak value (V_{ptp}) over 10 cycles with respect to the applied potential (5 volt) was determined by a data acquisition system. The fractional changes in the capacity and thus the electron work functions were determined from the obtained data. The test process was controlled by a computer.

3. Results and discussion

3.1. Corrosion-erosion with respect to the erodent velocity

Weight losses of Y-free and Y-containing 304 stainless steel (304SS) specimens in a 0.1 mol/l H_2SO_4 slurry containing 30% silica sand were measured, with respect to the erodent velocity. Fig. 2 presents results of the corrosion-erosion test. One may see that at low velocities, weight losses of both Y-free and Y-containing specimens decreased as the velocity increased. However,

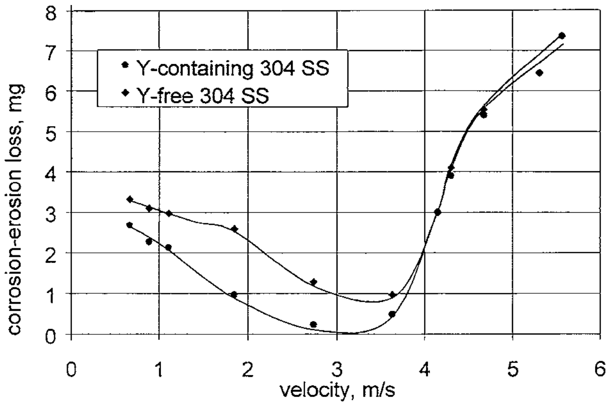


Figure 2 Variation in corrosive erosion loss versus the velocity for Y-free and Y-containing 304 stainless steels in 0.1 mol/l H_2SO_4 slurry containing 30% silica sand.

when the velocity exceeded a critical value around 3.3 m/s, substantial increases in the erosion loss occurred. As shown in Fig. 2, yttrium diminished the corrosion-erosion when the erodent velocity was below 4 m/s. However, when the erodent velocity exceeded 4 m/s, yttrium did not show any detectable influence on the corrosion-erosion performance of 304 steel.

As demonstrated previously [18], the Vickers hardness and strain-hardening capability of 304 stainless steel were almost unchanged when yttrium was alloyed into the steel, which implies that the enhancement in corrosion-erosion resistance of the stainless steel by yttrium is not attributable to possible changes in its mechanical properties. Therefore, the observed corrosion-erosion response of the Y-free and Y-containing specimens to the variation in velocity may result from effects of yttrium on properties of the passive film of 304 stainless steel.

3.2. Polarization behavior and electrochemical scratch resistance

Corrosive wear is a dynamic process involving rupture and reformation (repassivation) of the passive film. Many studies on stainless steel have shown the influence of passive film on wear or erosion of the material in dilute acid media [19–22]. The repassivation ability of an alloy and the properties of the passive film are important to the surface damage of the material when attacked by simultaneous solid particle impingement and electrochemical reaction. When yttrium is alloyed into 304 steel, it could help to form a stronger and more adherent protective passive film, thus leading to improve corrosion resistance.

The polarization test demonstrated that both Y-free and Y-containing 304 steels exhibited good capability of self-passivation. Various electrochemical parameters that characterize the polarization behavior of the materials were determined and are given in Table I. Details of the electrochemical polarization measurement have been given in Ref. [18]. As shown in Table I, 1 wt% yttrium strongly affected the passivation current, the passive potential, and the current in the passive state. Clearly, the addition of yttrium significantly improved the passivation behavior of 304 stainless steel. According to a phase diagram of Fe-Y binary system [23], Fe-Y alloy mainly consists of α-Fe and $Fe_{17}Y_2$ phases when the content of yttrium is less than 10 wt%. The solid solubility of yttrium in γ-Fe is less than 0.8 at% at the Fe-rich eutectic temperature [24]. In our case, however, the amount of yttrium was not sufficient to be detected by using available X-ray diffractometer and the energy dispersive X-ray spectroscopy (EDS). Further microstructural examination is therefore needed by using other techniques such as a transmission electron microscopy (TEM).

An electrochemical scratch technique was used to investigate the rupture and reformation of passive films, respectively, on the Y-free and Y-containing 304 stainless steel specimens during corrosive scratching. This test may simulate the damage process of a passive film on a target surface eroded by a single incident particle. The rupture and self-healing of the passive film could be monitored by recording variations in the transient current during scratching a stainless steel specimen under a certain corrosion potential. Results of the scratch test are illustrated in Figs 3 and 4. When the diamond tip scratched the sample surface, the transient current increased rapidly with time (line AB). This increase in the transient current reflects the electrochemical dissolution of 304 steel when the passive film is ruptured during scratch. Since the Y-containing and Y-free steel

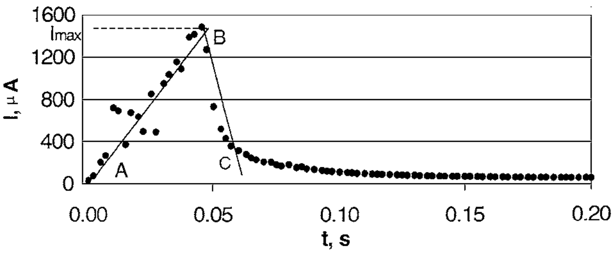


Figure 3 A typical transient current ~ time curve of Y-free 304 stainless steels recorded during scratch in 0.1 mol/l H_2SO_4 solution under a load of 6.9 N.

TABLE I Measured polarization parameters

Alloy	E_c	E_p	i_p	i_{cp}	E_b
Y-containing 304 SS	-80 mV$_{SCE}$	60 mV$_{SCE}$	2.2 μA/cm^2	1.7 μA/cm^2	852 mV$_{SCE}$
Y-free 304 SS	-100 mV$_{SCE}$	253 mV$_{SCE}$	32.3 μA/cm^2	58.4 μA/cm^2	932 mV$_{SCE}$

E_c—the corrosion potential.
E_p—the critical passivation potential.
i_p—the critical passivation current density.
i_{cp}—the passivation current density.
E_b—the film breakdown potential.

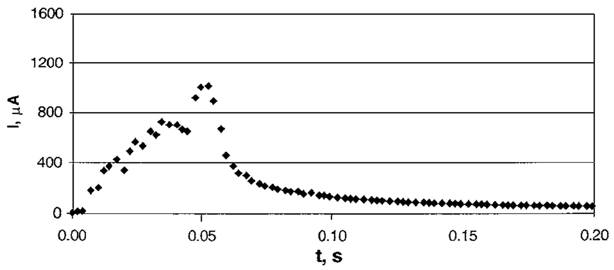


Figure 4 A typical transient current ∼ time curve of Y-containing 304 stainless steel recorded during scratch in 0.1 mol/l H_2SO_4 solution under a load of 6.9 N.

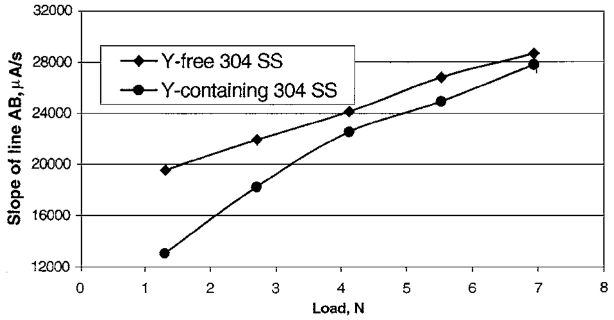


Figure 5 The slope of line AB (see Fig. 3) versus the applied load.

samples have similar mechanical properties [18], the slope of $I \sim t$ curve therefore mainly reflects the resistance of the passive film to scratch. A lower slope corresponds to greater resistance to scratch. Fig. 5 plots the slopes of the transient current ∼ time curve with respect to the normal load for both the Y-free and Y-containing specimens. Clearly, yttrium helped to diminish the failure of the passive film caused by scratch. The decrease in slope by yttrium addition was larger under low loads. The lower slope for Y-containing steel could be attributed to possible improvement in mechanical properties and the adherence of its passive film. The less damage of the passive film was beneficial to the prevention of corrosion-erosion. However, under higher loads, the difference in the slope of the transient current ∼ time curve between Y-free and Y-containing specimens decreased. This occured because the diamond tip could penetrate into the sample surface deeply and completely damage the passive film, thus decreasing the difference between the Y-containing and Y-free specimens. The small current fluctuations observed during the scratching process (see Figs 3 and 4) indicated that there was a dynamic competition between damage and recovery of the passive film. The peak current (I_{max}) corresponded to the maximum dissolution rate and increased with an increase in the scratching load. Under the same load, I_{max} of Y-free steel was larger than that of the Y-containing steel, implying that the Y-free steel was more vulnerable to corrosive wear. Microhardness test indicates that 1 wt% yttrium does not markedly influence the hardness of 304 steel [18]. Therefore, the enhanced resistance of the Y-containing 304 steel to electrochemical scratch could be mainly attributed to the improvement in properties of its passive film.

After reaching the peak current, the decay of the transient current corresponded to self-healing of the passive film or repassivation after the diamond tip left the scratched surface. In the rapid decay period, it appeared that the passive film on Y-containing steel grew slower than that on the Y-free stainless steel, which can be seen from the difference in the slope (line BC, see Fig. 3) between Y-free and Y-containing specimens (Fig. 6). The latter had a less steep slope than the former. This might be related to the decreases in the electrochemical reaction rate and the suppression of lateral growth of the film due to the presence of yttrium [13–15]. However, by comparing Fig. 3 with Fig. 4 under the same scratching load, one may not see significant difference in the total repassivation time between the Y-free and Y-containing specimens. These results imply that the reformed passive film on Y-free steel that completely covered the scratched surface area could be thicker than that on the Y-containing stainless steel. In general, a thicker passive film has higher interfacial stress at the passive film/substrate interface (on nano-meter scale), and thus it is relatively easier to be ruptured by mechanical scratch or particle impingement. Therefore, a thinner passive film of Y-containing steel could favor the resistance of the steel to corrosion-erosion or corrosive scratch.

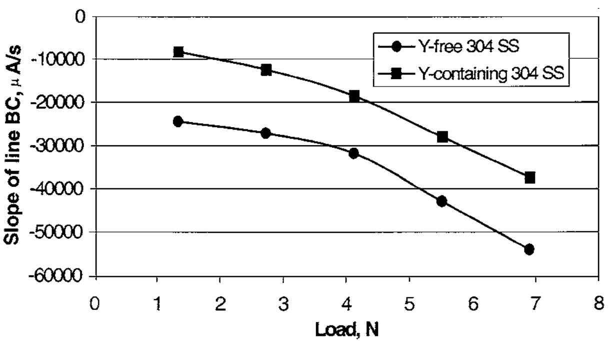


Figure 6 The slope of line BC (see Fig. 3) versus the applied load.

Another useful parameter is the area under the scratching curve, which may reflect the electrochemical dissolution of the metal by a single particle scratching action. It can be seen in Fig. 7 that the total consumed quantity of electricity for the Y-free steel from the beginning of scratching to the completion of repassivation was larger than that for the Y-containing steel under the same scratching condition. Obviously, yttrium diminished the electrochemical dissolution of 304 steel during scratching in the corrosive medium.

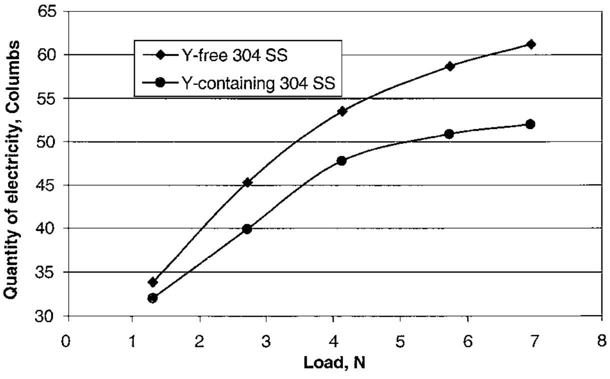
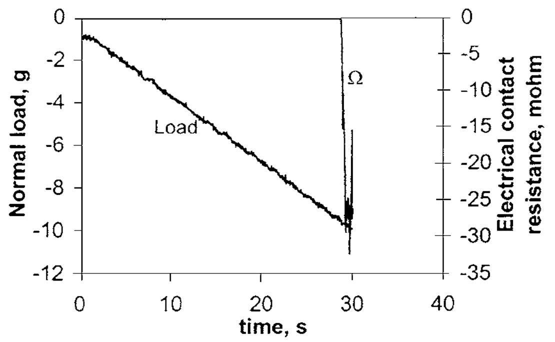


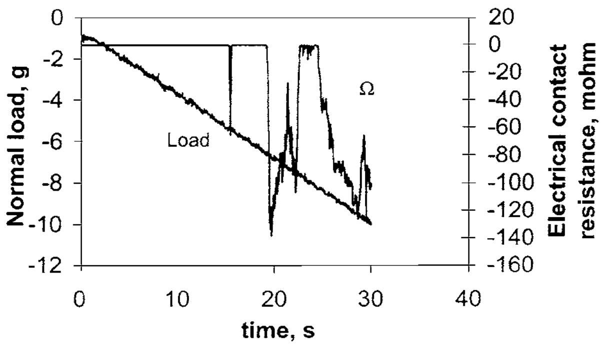
Figure 7 Quantities of electricity consumed during the electrochemical scratch process under different loads.

3.3. Resistance to mechanical scratch and indentation

In order to clarify if yttrium benefits the mechanical properties of the passive film, micro-indentation and micro-scratch tests were performed on surfaces of Y-free and Y-containing 304 stainless steel samples, respectively. All the samples experienced passivation treatment in a dilute sulfuric acid solution in order to obtain thicker passive films for study. During the tests, the electrical contact resistance was *in situ* measured. Since the passive film on stainless steel is a semiconductor, its electrical resistance is significantly larger than that of metals. When the passive film failed during indentation or scratch by a tungsten carbide tip under an applied load, the electrical contact resistance dropped steeply. The critical normal load corresponding to the drop in the electrical contact resistance is a measure of the laod-carrying capability of the passive film. Results of the indentation and scratch tests are presented in Figs 8 and 9, respectively. Two curves in each figure represent the applied normal load and the contact electric resistance, both of which are plotted versus time. One may see that as the applied load was increased and reached a critical value, the contact resistance suddenly dropped as Figs 8 and 9 illustrate. During the indentation test, the passive film of the Y-containing steel failed when the applied load reached about 10-gram (Fig. 8a); while the passive film on the Y-free 304 steel failed when the load was about 5.5 g (Fig. 8b). In the case of micro-scratch test, the situation was similar. The passive film on the Y-containing steel started to fail when the load exceeded 5 g and severe damage occurred when the load reached about 7.5 g (Fig. 9a), while the critical load for the Y-free specimen was about 4 g (Fig. 9b). Both the micro-scratch and micro-indentation tests indicated that the passive film on Y-containing steel surface had higher load-carrying capability than that on the Y-free steel surface.

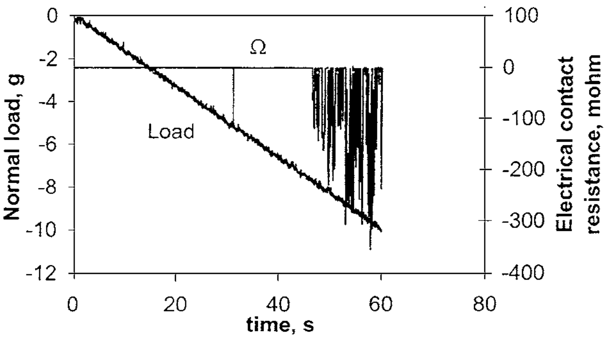


(a) Y-containing 304 stainless steel

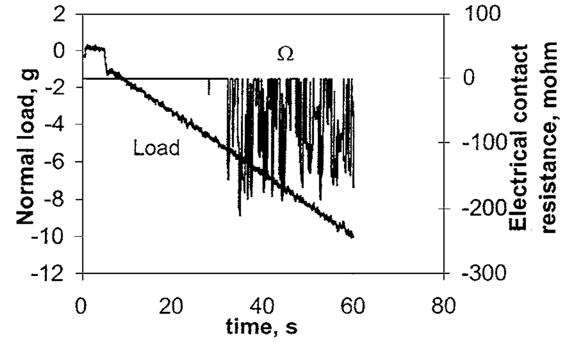


(b) Y-free 304 stainless steel

Figure 8 Variation in the electrical contact resitance with respect to the indentation load.



(a) yttrium-containing 304 stainless steel



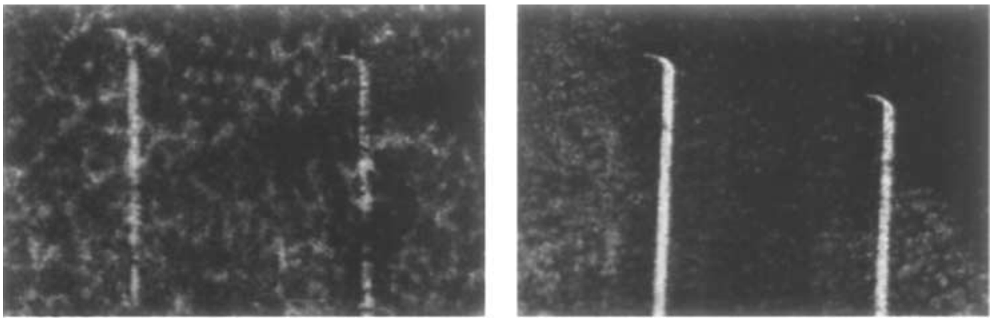
(b) yttrium-free 304 stainless steel

Figure 9 Variation in the electrial contact resistance with respect to the scratch load.

The scratch tracks were examined under an optical microscope. Fig. 10 illustrates scratch tracks of the Y-free and Y-containing 304 steel specimens. For the Y-free steel, its scratch track showed brittle-spalling characteristics with micro-cracks (Fig. 10a). While the scratch track of the Y-containing steel was continuous and the damage was made by plowing with little brittle-spalling characteristic (Fig. 10b). In addition, no micro-cracks were observed on the passive film of the Y-containing 304 steel. It was thus demonstrated that the passive film of the Y-free steel was brittle, compared to that of the Y-containing steel. Clearly, yttrium improved the mechanical properties of the passive film by reducing its brittleness with enhanced resistance to scratch and indentation.

3.4. Electron work function

The electron work function reflects the ability of an electron to escape from the Fermi surface of a material and become a free electron. Because the formation and dissolution of a passive film involve electron transfer, the electron work function could therefore be a measure of the stability or inertness of the passive film. Fig. 11 presents electron work functions of Y-containing and Y-free samples after passivation treatement in a dilute sulfuric acid solution. The work function of the passive film of Y-containing sample is remarkably higher than that on Y-free sample. This implies that the passive film on Y-containing steel is more stable or inert



(a) Y-free 304 stainless steel (b) Y-containing 304 stainless steel

Figure 10 Optical micrographs of scratch tracks.

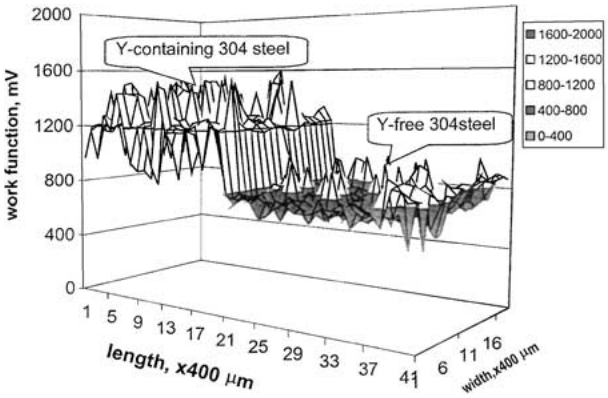
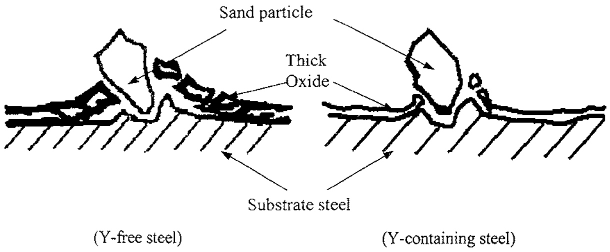
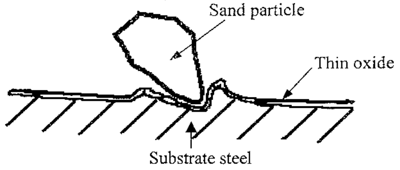


Figure 11 Electron work functions of the passive films on Y-containing and Y-free steel samples.

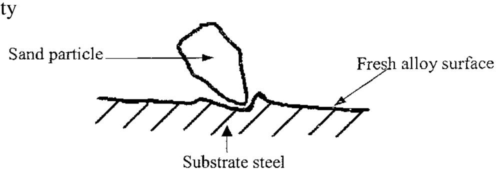
against electrochemical attack than the passive film on Y-free steel. The result of the work function measurement is consistent with the electrochemical scratching test and the polarization experiment [18]. Because the passive film could contain Y_2O_3 phase when yttrium was implanted into the surface layer of the stainless steel [25], this phase might be responsible for the difference in electron work functions between the passive film of Y-containing steel and that of Y-free steel.



(a) Low corrosion-erosion resistance due to the rupture of thick passive film at low sand erosion velocity



(b) Improved corrosion-erosion resistance due to the reduction in thickness of passive film with lower interfacial stress at moderate sand erosion velocity



(c) Corrosive erosion of fresh steel surface at high sand erosion velocity

Figure 12 Possible mechanism responsible for the variation in corrosion-erosion of 304 stainless steel at different erodent velocities.

3.5. Possible corrosive erosion mechanism

When yttrium was added, corrosion-erosion was reduced when the erodent velocity was below a critical value around 3.3 m/s (see Fig. 2). The electrochemical scratch test showed that the resistance of the passive film on the Y-containing 304 steel to corrosive scratching was higher than that of the passive film on the Y-free 304 steel, evidenced by the difference in slope of the transient current curve (line AB) between Y-free and Y-containing specimens. The resistance to corrosive scratching enhanced by yttrium should be largely attributed to the improvement in the mechanical behavior of passive film as Figs 8–10 illustrate. In addition, the increase in electron work function of the passive film could also be beneficial, which may improve the stablity of the film against electrochemical attack. Another possible effect of yttrium is the enhancement of the interfacial bonding between the passive film and the substrate. Such an expectation is based on the beneficial effect of yttrium on the oxide adherence that was demonstrated by previous studies on oxidation, although direct evidence is needed before drawing a firm conclusion. Besides, another possible factor favoring the adherence of the passive film on Y-containing 304 steel is its smaller thickness. It should be relatively easy for the interfacial misfit stress to be relaxed when the film is thin (on nano-meter scale).

Regarding the variation in material loss with respect to the erodent velocity, when the erodent velocity exceeded 3.3 m/s, there was almost no difference in weight loss between Y-containing and Y-free 304 steel specimens. This phenomenon could be explained by considering the following possible process of surface damage, as shown in Fig. 12. At low velocities of the sand flow, the impact force by solid particles is relatively small, and the time interval between two successive particle impacts is relatively larger so that a thicker passive film could form. Because of the lattice mismatch and the constraint from outer-growth atomic layers, a thick passive film may introduce relatively large interfacial stress and this increases the probability of interfacial debonding. Rupture of the thicker passive film could thus be easier during erosion, compared to that of a thinner passive film. As a result, the corrosion could be promoted because the exposure of bare metal surface to the corrosive medium was increased and the electrochemical dissolution is also accelerated due to the undamaged passive region as cathode. Fig. 12a schematically illustrates the rupture and peeling of a relatively thick film under the impact of a solid particle. As demonstrated by the erosion test, at moderate velocities, the corrosion-erosion loss decreased. This phenomenon could be attributed to the formation of a thinner passive film as the erodent velocity was increased. A thinner passive film should be associated with lower interfacial stress, which is beneficial to the reduction of interfacial debonding or failure under the influence of internal stress and external impact force by solid particles (see Fig. 12b). As a result, the resistance to corrosion-erosion could be improved. However, this situation is valid only when the film involves limited atomic layers (on nanometer scales, as the case for passive films). Such a difference in the interfacial stress between thin and thick films should not be seen if the films are thick enough. At high erodent velocities, no passive film could form timely and the bare metal surface is always under the successively combined attack of electrochemical dissolution and direct solid particle impingement (Fig. 12c). The steep increase in material loss with respect to the velocity could thus be resulted. When yttrium is added, it improves the mechanical and electrochemical properties of the passive film and may also reduce the thickness of the film as suggested by the electrochemical scratch test. The resistance to corrosion-erosion is thus enhanced. However, when the velocity exceeds a critical value, yttrium may not show any effect on the resistance to corrosion-erosion, since no passive film could form. Thus, no difference in corrosion-erosion was observed between the Y-containing and Y-free specimens when the erodent velocity was high as Fig. 2 illustrates. One may notice that the critical velocity for Y-containing 304 steel at which the erosion loss increased was slightly lower than that for the Y-free 304 steel; this might be attributed to thinner passive film on the former.

In summary, the corrosion-erosion, electrochemical scratch and polarization tests have clearly demonstrated that yttrium is beneficial to 304 stainless steel for improved resistance to corrosion-erosion and corrosive wear. A small amount of yttrium does not markedly influence mechanical properties of the stainless steel but plays a role in improving properties of the passive film. As demonstrated by the micro-scratch and micro-indentation tests as well as the electron work function measurement, the failure resistance and the stability of passive film were enhanced due to the presence of the yttrium addition. Yttrium also reduced the thickness of passive film, which could help to relax the interfacial misfit stress and thus benefitted the interfacial bonding. However, whether or not yttrium can markedly enhance the interfacial bonding as it does in the case of oxidation needs further investigation.

4. Conclusions

Effects of yttrium on the corrosion-erosion behavior of 304 stainless steel were investigated using different experimental techniques, including corrosion-erosion test, electrochemical scratch, mechanical scratch and indentation, polarization and electron work function measurement. The following conclusions were drawn from the study:

1. The corrosion-erosion of 304 stainless steel is related to the failure and self-healing of its passive film; in the velocity range for the present corrosion-erosion test, the weight loss of 304 stainless steel initially decreased to a minimum and then rapidly increased as the erodent velocity increased;
2. Yttrium addition improved the resistance of 304 stainless steel to corrosion-erosion. However, yttrium did not show any influence when the erodent velocity exceeded a certain level;
3. Yttrium enhanced the resistance of the passive film to corrosive scratch in dilute H_2SO_4 solution;
4. Yttrium improved the mechanical properties of the passive film, resulting in enhanced resistance to mechanical scrtatch and indentation. Yttrium also stabilized the passive film and made it more inert to electrochemical attack.

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