

Research Article

Pickling Behaviour of 2205 Duplex Stainless Steel Hot-Rolled Strips in Sulfuric Acid Electrolytes

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Pickling behaviour of the oxide layer on hot-rolled 2205 duplex stainless steel (DSS) was studied in H_2SO_4 solutions with electrolytic workstation. The pickling rate at 75°C increases slightly with the H_2SO_4 concentration from 100 to 250 g L⁻¹ but increases markedly as the concentration is higher than 250 g L⁻¹. The solution temperature greatly accelerates the pickling rate. Fe³⁺ ion is an effective oxidant, which shows stronger inhibitive effect. The rotating disk electrode was used to simulate the moving state of steel strips in practice. The movement with the speed from 0 to 20 m min⁻¹ results in weak acceleration effect on the pickling process. Under dynamic conditions, the pickling rate increases noticeably with changing the pulse current density from 0 to 0.2 A cm⁻². The industrial pickling efficiency of 2205 DSS hot-rolled strips increases from 5–8 m/min to 15–18 m/min. The surface quality is improved.

1. Introduction

Type 2205 duplex stainless steel (DSS) has been used widely in many industries for its excellent mechanical properties and corrosion resistance [1–6]. However, the oxide scales formed on the surface of 2205 DSS during rolling and heat treatment are stable and dense due to the high chromium, molybdenum, and nitrogen contents [7]. Moreover, oxide nodules may often grow on the surface of 2205 DSS for the different oxidation rates of ferritic and austenitic phases at high temperature [8, 9]. Therefore, the pickling of 2205 DSS hot-rolled strips is more difficult than that of standard austenitic grades [10].

The pickling process of stainless steels is used to remove the oxide scales and chromium depleted layer in order to restore the corrosion resistance and ensure the surface finish [11, 12]. The industrial pickling process includes mechanical descaling, prepickling, and mixed acid pickling. A mechanical descaling is achieved through breaking, grinding, roto blasting, and sand blasting processes. Prepickling is performed either anodically or cathodically in acid or neutral electrolytes with certain current densities [13–15]. The general electrolytes are Na_2SO_4 , H_2SO_4 , H_3PO_4 , HCl, HF, and H_2O_2 . Mixed acid pickling is the final step to dissolve the oxide scales in H_2SO_4 -HF [16], H_3PO_4 - H_2SO_4 [17], HCl-HF [18], or HNO₃-HF [19].

Prepickling plays a critical role in the industrial production process of 2205 DSS hot-rolled strips. Na₂SO₄ electrolytes can conduct electricity, but they cannot dissolve the oxides and matrix [20]. H₂SO₄ solutions can dissolve the oxide scales and matrix [16, 21]. H₃PO₄ solutions have good properties of dissolving oxide scales, but their costs are expensive [22]. HCl solutions can dissolve ferrous oxides, but chloride corrosion is the big problem [18, 23–25]. HF solutions can dissolve oxide scales and SiO₂, but there is a serious intergranular corrosion problem [16, 18]. The dissolving effect of H₂O₂ is remarkable on the Cr-depleted layer of stainless steel but unsatisfactory on the oxide scale [26, 27]. After comparison, H₂SO₄ solutions are extensively used in practice for their low cost and chemical stability.

Pickling process can be simulated with static and dynamic conditions at the laboratories. There are many studies on static pickling [16–26], but very few on dynamic pickling [22]. The stainless steel strips in the industrial pickling process are moving. Therefore, the studies focused on dynamic pickling are of practical significance.

The pickling technology and mechanism are well known for the austenitic and ferritic stainless steels in the literature, but there are few systematical reports focused on the pickling for duplex stainless steel, especially for DSS 2205 hot-rolled strips. With the rapid development of economy and living standard, the demand for 2205 DSS is increasing quickly. However, the production of 2205 DSS is restricted greatly by its pickling process. It is necessary to study the pickling laws and improve the pickling efficiency for 2205 DSS hot-rolled strips. In this work, the pickling behaviour of the 2205 DSS hot-rolled strip in H_2SO_4 electrolytes under both static and dynamic conditions was investigated. The pickling mechanism was discussed on the basis of mass loss and surface analysis.

2. Experimental

2.1. Materials. A hot-rolled 2205 duplex stainless steel strip with a thickness of 4.0 mm was used as the test materials. Its chemical composition is given in Table 1.

2.2. Sample Preparation. A plate of 300×300 mm was cut from the strip and annealed at 1140° C for 4 min. After annealing, the plate was taken out from the furnace and cooled down in air. The annealed plate was subjected to sand blasting to remove its main oxides on the surface and then was cut into the test specimen of 5×5 mm. The specimens were covered with epoxy resin leaving a working area of 5×5 mm and cleaned with alcohol before pickling.

2.3. Pickling Measurement. Pickling tests were carried out on the workstation as shown in Figure 1. A specimen fixed on the rotating disk with a diameter of 15 cm was used as the working electrode, a platinum sheet as the counterelectrode, and Hg/Hg₂SO₄ as the reference electrode. The analytically pure H₂SO₄, ferrous sulfate, iron sulfate, and deionized water were used to prepare the electrolytes. The pickling processes were performed with a Princeton potentiostat (PAR 273A).

2.4. Electrochemical Measurement. The corrosion potential and electrochemical impedance spectroscopy (EIS) of the test specimens were measured during the pickling process with a Princeton potentiostat (VMP3). Each specimen was fixed on the cell sidewall with a 1 cm² round hole. The test specimen was used as the working electrode; the platinum sheet was used as the counterelectrode; and the Hg/Hg₂SO₄ was used as the reference electrode (MSE).

All test samples were immersed in the electrolytes for 2 min. During electrolysis, the electrolytic time was 81 seconds with three periods. For each period, the specimen was polarized anodically for 18 seconds and cathodically for 9 seconds at various current densities. The pickling rate was

TABLE 1: Chemical composition of 2205 DSS specimens (wt. %).

С	Si	Mn	S	Р	Cr	Ni	Мо	Ν
0.024	0.62	1.42	0.001	0.024	21.13	5.46	3.11	0.15



FIGURE 1: Electrolytic workstation used for dynamic pickling tests. 1, thermostat water bath; 2, electrolyte; 3, rotating disk; 4, specimen.

calculated from the mass loss of the specimen with a precision of 0.01 mg. The specimens were characterized with scanning electron microscopy (FEI Quanta 600) and Raman spectroscopy (Renishaw, inVia).

3. Results

3.1. Characterization of Oxide Scales. In general, the oxide scale of the hot-rolled 2205 DSS strip after annealing can be divided into an inner layer and an outer layer [7]. Chromium is rich in the inner layer while iron is rich in the outer layer. The outer layer is consisted of Fe₂O₃, Fe₃O₄, and FeCr₂O₄, and the inner layer is composed mainly of FeCr₂O₄ [28]. The outer layer may be partially removed by mechanical descaling. The FeCr₂O₄ is the oxide with a spinel structure, which is insoluble in most industrial acids [7, 29].

As shown in Figure 2, a few oxide scales remain on the surface of the test specimen after shot blasting. Raman spectrum in Figure 3 indicates that the residual oxides on the surface of 2205 DSS hot-rolled strip are composed of Fe_2O_3 , Fe_3O_4 , and $FeCr_2O_4$. Among them, the content of $FeCr_2O_4$ is the highest as seen from its peak intensity. This implies that the pickling of the 2205 DSS hot-rolled strip is difficult.

3.2. Chemical Pickling Properties under Static Conditions. The static chemical pickling of stainless steel in H_2SO_4 solutions is mainly related with acid concentration, pickling temperature, and concentration of metal ions. For most steels, higher pickling temperature, higher concentration of H_2SO_4 solutions, and lower concentration of metal ions can improve its pickling efficiency in the initial pickling stage for its strong dissolving capacity. After pickling for a certain time, the strong dissolving capacity may deteriorate pickling result for the corrosion of matrix.



FIGURE 2: Surface image for the test specimen.



FIGURE 3: Raman spectrum for the test specimen.

The effect of H_2SO_4 concentration on static pickling rate and typical surface images of specimens at 75°C are shown in Figures 4 and 5. The pickling efficiency has a positive correlation with H_2SO_4 concentration. It can be seen that the pickling rate has a transition at about 250 g L^{-1} . When the concentration increases from 100 g L^{-1} to 250 g L^{-1} , the pickling rate is accelerated slightly and many oxide scales remain on the surface. When the concentration increases from 250 g L^{-1} to 450 g L^{-1} , the pickling rate is accelerated markedly to remove most of the oxide scales.

Figure 6 gives the static pickling rates of specimens in $300 \,\mathrm{g \, L^{-1} \, H_2 SO_4}$ solutions at different temperatures. The pickling efficiency has a positive correlation with the solution temperature. With the increasing temperature from 50°C to 90°C, the pickling rate increases gradually from about 1.74 to 12.8 g m⁻² min⁻¹ and the retained oxide scales reduced noticeably, as shown in Figure 7.

The metal ions in the H_2SO_4 solutions include iron, chromium, molybdenum, nickel, manganese, titanium, and niobium ions. The conventional metal elements in industrial H_2SO_4 electrolytes are shown in Table 2. Among them, iron is the main element. Therefore, the iron ions play the main impact on the pickling process.



FIGURE 4: Variation of the static pickling rate in H_2SO_4 at 75°C with different concentrations.

The effect of iron ions on the static pickling of the specimen was studied in $300 \text{ g L}^{-1} \text{ H}_2\text{SO}_4$ solutions at 75°C and the results are given in Figure 8. The pickling efficiency has a negative correlation with the concentration of iron ions. With increasing concentration of ferric ions from 0.5 g L^{-1} to 60 g L^{-1} , the pickling rate declines sharply at first to less than $1 \text{ g m}^{-2} \text{ min}^{-1}$ and then decreases insignificantly. With increasing concentration of ferrous ions, the pickling rate decreases slowly, but shows higher values in comparison with ferric ions. The ferric ions are an effective oxidant which can enhance the corrosion potential of test specimens [16] and then reduce their dissolution rate remarkably. In order to improve the pickling rate of 2205 DSS hot-rolled strips, new sulfuric acid solutions without iron ions are necessary.

3.3. Chemical Pickling Properties under Dynamic Conditions. In order to see the difference between dynamic and static pickling of hot-rolled 2205 DSS strips, the rotating disk was applied to create the movement between specimens and acid solutions. The variation of pickling rate of the specimen in $300 \text{ g L}^{-1} \text{ H}_2\text{SO}_4$ solutions at 75°C with rotary speed is given in Figure 9. The pickling efficiency has a positive correlation with moving speed of the specimen. The pickling rate is higher in the dynamic cases than in the static case. However, with the increase of rotary speed from 5 to 20 m min⁻¹, the pickling rate only enlarges slightly from about 7.5 to 7.8 g m⁻² min⁻¹. It is evident that the acceleration effect of dynamic speed is very weak.

3.4. Electrolytic Pickling Properties under Dynamic Conditions. The applied polarization plays a role in pickling process. Figure 10 gives the variation of the dynamic pickling rate at 10 m min^{-1} for specimens in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C with pulse current density. The pickling rate becomes higher with increasing the pulse current density from 0 to $0.2^\circ \text{A cm}^{-2}$. The rate is about 2.1 times larger at



FIGURE 5: SEM surface images after static pickling in H_2SO_4 solutions at 75°C with different concentrations: (a) 300 g L⁻¹ and (b) 450 g L⁻¹.



FIGURE 6: The static pickling rate of the specimen in $300 \, g \, L^{-1} \, H_2 SO_4$ solutions at different temperatures.



FIGURE 7: SEM surface images after static pickling in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at temperatures 60°C (a) and 90°C (b).

TABLE 2: Concentration of metal ions in industrial H₂SO₄ solutions.





FIGURE 8: The static pickling rate in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C with different iron ions.



FIGURE 9: Dynamic pickling rate in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C under different rotary speed conditions.

 0.2 A cm^{-2} than at 0 A cm^{-2} (i.e., the chemical pickling). The electrolytic pickling efficiency has a positive correlation with the pulse current density. After dynamic electrolytic pickling, there is almost no oxide scales on the specimen surfaces compared with static electrolytic pickling. With the increase of current density from $0.04 \text{ A} \text{ cm}^{-2}$ to $0.2 \text{ A} \text{ cm}^{-2}$, the Crdepleted layer may dissolve obviously and the surfaces become smooth, as shown in Figure 11.



FIGURE 10: Variation of the dynamic pickling rate with current density in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C .

4. Discussion

The residual oxides on the surface of the test specimen are composed of Fe₂O₃, Fe₃O₄, and FeCr₂O₄, as shown in Figure 3. While pickling of 2205 DSS hot-rolled strips in H₂SO₄ electrolytes, it involves two processes, chemical pickling process and electrolytic pickling process.

When chemical pickling is carried on for 2205 DSS, FeO, Fe₂O₃, Fe₃O₄, and alloying elements can be dissolved in H₂SO₄ solutions. The chemical pickling process involves the following chemical reactions [30]:

$$FeO + H_2SO_4 \longrightarrow FeSO_4 + H_2O$$
 (1)

$$\operatorname{Fe_3O_4} + 4\operatorname{H_2SO_4} \longrightarrow \operatorname{FeSO_4} + \operatorname{Fe_2}(\operatorname{SO_4})_3 + 4\operatorname{H_2O}$$
(2)

$$Fe_2O_3 + 3H_2SO_4 \longrightarrow Fe_2(SO_4)_3 + 3H_2O$$
(3)

$$Fe + H_2SO_4 \longrightarrow FeSO_4 + H_2\uparrow$$
 (4)

$$Ni + H_2SO_4 \longrightarrow NiSO_4 + H_2\uparrow$$
(5)

$$\operatorname{Cr} + \operatorname{H}_2 \operatorname{SO}_4 \longrightarrow \operatorname{Cr} \operatorname{SO}_4 + \operatorname{H}_2 \uparrow$$
 (6)

The oxide scales on the test specimen are mainly comprised of FeCr₂O₄, as shown in Figure 3. The FeCr₂O₄ is insoluble in sulfuric acid solution [7]. The matrix of 2205 DSS is dissolved slowly in sulfuric acid solution for its excellent corrosion resistance. The oxide scales on the test specimen are dissolved very slowly in sulfuric acid solutions by means of peeling mechanically. Therefore, the chemical pickling of the 2205 DSS hot-rolled strip is very slow. For most stainless steels, higher pickling temperature and sulfuric acid concentration do not increase their pickling efficiency after a certain level [30]. But, for 2205 DSS, the oxide scales still exist after chemical pickling in 450 g L⁻¹ H₂SO₄ at 90°C.



FIGURE 11: SEM surface images after static (a-c)) and dynamic (d-f)) pickling in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C with current density 0.04 A cm⁻² (a and d), 0.12 A cm⁻² (b and e), and 0.2 A cm⁻² (c and f).

At the beginning of the static chemical pickling process, small gas bubbles nucleate and grow on the specimen surface. During the pickling process, more and more gas bubbles will form and adsorb on the specimen surface, which must reduce the contact areas of effective pickling. As for the dynamic pickling of specimens on the rotating disk, the movement may decrease the adsorption and growth of gas bubbles on the specimen surface to a certain extent [31]. Therefore, the chemical pickling rate of the annealed 2205 DSS specimens under dynamic condition is higher than under static condition. Nevertheless, the acceleration effect of dynamic speed is very weak, with about 5~10% increase in the pickling rates, as observed in Figure 8.

When sulfuric acid is used as an electrolyte, it can conduct electricity. The test specimen serves as the anode and cathode with the alternative change. When the test specimen serves as the relative anode, the insoluble $FeCr_2O_4$ is oxidized into soluble $HCrO_4^-$ and $Cr_2O_7^{2-}$. The anodic electrolytic reactions are as follows [10, 30, 32]:

$$2H_2O \longrightarrow O_2\uparrow + 4H^+ + 4e \tag{7}$$

$$\operatorname{FeCr}_2O_4 + 4H_2O \longrightarrow \operatorname{FeO} + \operatorname{HCrO}_4^- + 7H^+ + 6e$$
 (8)

$$\operatorname{FeCr}_{2}\operatorname{O}_{4} + 4\operatorname{H}_{2}\operatorname{O} \longrightarrow \operatorname{FeO} + \operatorname{Cr}_{2}\operatorname{O}_{7}^{2-} + 8\operatorname{H}^{+} + 6e \qquad (9)$$

$$2\text{FeO}_3 \longrightarrow 4\text{Fe}^{3+} + 3\text{O}_2\uparrow + 12e \tag{10}$$

When the test specimen serves as the relative cathode, $Cr(OH)_3$ and $Fe(OH)_3$ are deposited on the surface of the test specimen. H_2 is precipitated between the oxide layers, which is beneficial to the separation of the deposited sediment from the matrix. The cathodic electrolytic reactions are as follows [10, 30, 32]:

$$2H_2O + 2e \longrightarrow 2OH^- + H_2\uparrow$$
 (11)

$$\operatorname{Fe}^{3+} + 3\operatorname{OH}^{-} \longrightarrow \operatorname{Fe}(\operatorname{OH})_{3} \downarrow$$
 (12)

$$\operatorname{CrO}_4^{2-} + 4\operatorname{H}_2\operatorname{O} + 6e \longrightarrow \operatorname{Cr}(\operatorname{OH})_3 \downarrow$$
 (13)

$$\operatorname{Cr}_{2}\operatorname{O}_{7}^{2-} + 4\operatorname{H}_{2}\operatorname{O} + 6e \longrightarrow 2\operatorname{Cr}(\operatorname{OH})_{3} \downarrow + 8\operatorname{OH}^{-}$$
(14)

Electrolytic pickling is faster than normal chemical pickling for most stainless steels [20]. The electrolytic pickling process in H_2SO_4 electrolytes is the combined effect of electrolysis and chemical pickling. When the electrolytic pickling is performed under static conditions, a maximum of about 20% of the current goes to dissolution reactions, whereas about 80% of the current is consumed in oxygen gas production [10]. For the dense and compact oxide scales of the test specimens, spallation or peeling of the oxide scales induced by gas evolution does not play a decisive role. As shown in Figure 11, the oxide scales are remained on the test specimens after static electrolytic pickling. When the



FIGURE 12: Corrosion potential in $300 \,\text{g}\,\text{L}^{-1}\,\text{H}_2\text{SO}_4$ solutions at 75°C containing different Fe ions.



FIGURE 13: Nyquist plots in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C containing no ions (a), $10 \text{ g L}^{-1} \text{ Fe}^{2+}$ (b), and $10 \text{ g L}^{-1} \text{ Fe}^{3+}$ (c), and the corresponding circuit of the impedance spectrum (d).

TABLE 3: Fitted results for EIS spectra in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C containing different ions.

Ions	R_s , Ω cm ²	$Y_{0-f} S^{\alpha}, \ \Omega^{-1} \mathrm{cm}^{-2}$	$lpha_{ m f}$	$R_{\rm f}, \Omega {\rm cm}^2$	$Y_{0-\rm dl} S^{\alpha}, \Omega^{-1} {\rm cm}^{-2}$	$\alpha_{\rm dl}$	$R_{\rm t}, \Omega{\rm cm}^2$
No ions	0.82	0.00218	0.87	2.80	0.0658	0.92	1.6
$10 \text{ g L}^{-1} \text{ Fe}^{2+}$	0.94	0.00185	0.96	3.14	0.093	0.84	10
$10 \text{ g L}^{-1} \text{ Fe}^{3+}$	1.04	0.000133	0.95	54.99	0.000281	0.68	70.6

electrolytic pickling is performed under dynamic conditions, the rotary disk develops a rational way for producing active edges efficiently, where catalytic activity of the test specimens is improved and its pickling rate is accelerated [33, 34]. The dynamic electrolytic pickling rate at 0.2 A cm⁻² is about 2.1 times higher than the chemical pickling, which may give rise to a smooth specimen surface without oxide scales.

The Fe³⁺ ions are an effective oxidant which enhanced the corrosion potential, as shown in Figure 12. When no Fe ions were added in the H₂SO₄ solutions, the corrosion potential quickly declined at the beginning of the immersion time; after about 50 s, it remained constant (approximately $-0.75 V_{MSE}$) subsequently as the immersion time increased. When $10 \text{ g L}^{-1} \text{ Fe}^{2+}$ was added in the H₂SO₄ solutions, the corrosion potential quickly declined at the beginning of the immersion time; after about 100 s, it remained unchanged. When $10 \text{ g L}^{-1} \text{ Fe}^{3+}$ was added in the H₂SO₄ solutions, the corrosion potential increased slowly from 0.062 V_{MSE} to $0.137 V_{MSE}$ as the immersion time increased. The EIS Nyquist plots recorded on the test specimens immersed in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions containing no ions, $10 \text{ g L}^{-1} \text{ Fe}^{2+}$, and $10\,g\,L^{-1}$ Fe³⁺ under the E_{corr} conditions (i.e., the free corrosion states) is presented in Figure 13. The semicircle size enlarged slightly with the addition of $10 \,\mathrm{g \, L^{-1}}$ Fe $^{2+}$ but increased noticeably with the addition of the $10 \,\mathrm{g \, L^{-1}}$ Fe $^{3+}$ for its high impedance.

In order to show the dissolution process for the specimens in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ solutions at 75°C containing different Fe ions, the equivalent circuit model is proposed in Figure 13(d) according to the EIS features in Figure 13. R_s is the electrolyte resistance. R_f is the resistance of oxide layer remained on the specimens. R_t is the charge transfer resistance. C_f and C_{dl} can be replaced with constant phase element (CPE) [35]. The impedance of CPE is written in equation (15), where Y_0 is the admittance magnitude of CPE and α is the exponential term. Table 3 gives the fitted results of EIS spectra. The calculated spectra are shown as a solid curve in Figure 13, which fit the experimental data very well. It can be concluded that the model provided a reliable description for the corrosion systems.

$$Z_{\rm CPE} = \frac{1}{Y_0 (j\omega)^{\alpha}}.$$
 (15)

5. Application and Performance

According to the abovementioned results, the pickling process of hot-rolled 2205 DSS is optimized through the high H_2SO_4 concentration, high solution temperature, and proper electrolysis current density in the industrial prepickling of 2205 DSS hot-rolled strips. The pickling efficiency is improved remarkably, which increases the



FIGURE 14: Optical surface of the 2205 DSS hot-rolled strip after industrial pickling with optimized pickling process.

production rate from $5-8 \,\mathrm{m\,min^{-1}}$ to $15-18 \,\mathrm{m\,min^{-1}}$. Besides, the surface finish after pickling is notably enhanced, as shown in Figure 14.

6. Conclusion

The pickling behaviour of hot-rolled 2205 DSS with annealing and mechanical descaling treatments in H_2SO_4 solutions under both static and dynamic conditions has led to the following conclusions:

- In the static chemical pickling process, the pickling rate may be accelerated noticeably by increasing the solution temperature and H₂SO₄ concentration, but be decelerated greatly by Fe³⁺ ions.
- (2) The chemical pickling process can be enhanced weakly by the moving speed of the specimen from 0 to 20 m min⁻¹ because the movement decreases the adsorption and growth of gas bubbles on the specimen surface to a certain extent.
- (3) Under dynamic conditions, the electrolytic pickling rate increases markedly with changing the pulse current density from 0.04 to 0.2 A cm^{-2} . The electrolytic pickling rate at 0.2 A cm^{-2} is about 2.1 times larger than the chemical pickling rate in $300 \text{ g L}^{-1} \text{ H}_2 \text{SO}_4$ at 75° C, resulting in the smooth and clean specimen surfaces.

Data Availability

The tables and figures data used to support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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