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PICKLING – PASSIVATION MECHANISM AND PROCESS OPTIMIZATION OF Q235 STEEL PIPELINE

MEHANIZEM PASIVACIJE ZARADI JEDKANJA IN PROCES OPTIMIZACIJE ČIŠČENJA CEVOVODA IZ JEKLA VRSTE Q235

Hongchao Ji[1*], Shuo Cai[1], Fengyun Zhu[2], Weichi Pei[1], Wenchao Xiao[3], Xuefeng Tang[4]

[1]North China University of Science and Technology, College of Mechanical Engineering, Tangshan, 063210 China
[2]CHINA 22 MCC GROUP CORPORATION LIMITED, Hebei Tangshan, 063035
[3]China University of Geosciences (Beijing), School of Engineering and Technology, Beijing, 100083, China
[4]Huazhong University of Science and Technology, State Key Laboratory of Materials Processing and Die & Mould Technology, 1037 Luoyu Road, Wuhan 430074, China

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Pipe cleaning is currently the most effective method to improve the cleanliness and corrosion resistance of pipes. In this paper, a new method of pipe cleaning is proposed, combining mechanical and chemical cleaning, offline tank cleaning and online cycle cleaning. Through experiments and characterization of the morphology changes, the mechanism of pickling and passivation of Q235 steel was explored, and the entire process of microstructure and morphology changes on the pipe wall's surface was analyzed to verify the feasibility of this technology. The cleaning process was optimised using response surface analysis to determine the optimum cleaning conditions. This study is of great relevance to the effective operation of continuous-casting equipment over a long period of time.

Keywords: pipeline cleaning, pickling-passivation, corrosion of pipeline, micromorphology

Najbolj učinkovita metoda izboljšanja čistosti in povečanje korozijske odpornosti cevi za pretakanje različnih vrst kapljevin (voda, para, olja itd.) je njihovo redno in učinkovito čiščenje. V tem članku avtorji predlagajo in opisujejo novo metodo čiščenja cevi s kombinacijo mehanskega in kemičnega čiščenja cevnih inštalacij, kakor tudi čiščenje vstopnega in izstopnega rezervoarja naprav za kontinuirno litje jekla. S preizkusi in karakterizacijo so avtorji analizirali morfološke spremembe, mehanizem jedkanja in pasivacije jekla vrste Q235 iz katerega so izdelane cevi. S pomočjo analize celotnega procesa razvoja oblog in mikrostrukture površine cevi ter morfoloških sprememb so verificirali predlagani postopek in ugotavljali njegovo praktično izvedljivost. Proces čiščenja so optimirali s površinsko analizo odgovora in določili optimalne pogoje čiščenja. Ta študija je zelo pomembna za učinkovito in dolgotrajno nemoteno obratovanje opreme za kontinuirno litje jekla.

Ključne besede: čiščenje cevnih inštalacij, pasivacija z jedkanjem, korozija oziroma rjavenje cevi, mikromorfologija

1 INTRODUCTION

The iron and steel industries are pillars of a country's activities, and the output and quality of steel are important indicators for measuring the level of a country's industrialization.[1] As a key part of the steel production process, continuous-casting equipment is equipped with a wide range of transport pipelines, mainly for the transport of hydraulic oil, nitrogen, oxygen, water vapour and more than ten kinds of industrial media, pipeline material Q235 steel, pipeline length of more than 1.1×10^4 m.[2] Among them, most of the media have tough requirements for the cleanliness and corrosion resistance of the pipeline. A little carelessness may cause accidents and endanger life and property.[3] Therefore, how to achieve high-quality and efficient cleaning of the medium pipeline of continuous-casting equipment has become an urgent technical problem that needs to be solved.

*Corresponding author's e-mail:
jihongchao@ncst.edu.cn

Pipeline corrosion includes uniform corrosion, pitting corrosion, erosion corrosion, stray-current corrosion, microbial corrosion and other types of corrosion, and the causes of corrosion vary for different types of pipelines.[4] Ilman et al. suggested that the corrosion failure of sub-sea petroleum media pipelines is a mechanical combination of electrochemical and flow-induced corrosion.[5] Saleem et al. found that elevated soil pH and sudden changes in the stress caused by dropping or submerging the pipeline were the main causes of corrosion in the X52 gas pipeline.[6] Currently, the most effective way to prevent pipeline corrosion is to have the pipes cleaned. Common pipeline-cleaning methods fall into two categories: mechanical cleaning and chemical cleaning.[7] Mechanical cleaning in the traditional sense includes water-jet cleaning, pipeline purging and rust removal, etc. For metal pipelines with a lot of pollutants and serious rust, the use of water and air sources cannot meet the requirements of the purging process, and the cleaning quality is not ideal.[8] The chemical cleaning methods are mainly of two types: offline tank cleaning and online cycle cleaning. Offline tank cleaning is mainly to decompose the installed pipes and put them into the cleaning tank for immersion. The advantage of this method is that the cleaning quality is easy to control and the cleaning effect is easy to check, but the cleaning speed is slow.[9] On-line circulating cleaning mainly uses short pipes, hoses, etc. to temporarily connect the entire system into a circulating flushing circuit, and uses a pump group to drive the cleaning fluid into the circuit for on-line pickling and passivation. The advantage of this method is that the cleaning speed is faster, but the cleaning quality cannot be fully checked, and the cost is high.[10]

Siringi et al. argue that traditional cleaning methods are no longer adequate for today's pipe-cleaning requirements and that the use of acid solutions supplemented by corrosion inhibitors in chemical cleaning can be more effective in cleaning pipe-wall corrosion.[11] Abidin et al. developed an in-pipe robotic cleaning device to perform functions including cleaning and de-rusting, checking for cracks, and repairing pipe damage.[12] However, the existing pipe-cleaning technology is not better for cleaning the medium pipe of the continuous-casting equipment, for Q235 steel pipe pickling passivation mechanism and in the cleaning process of the surface microscopic morphological changes are not yet clear. Therefore, this paper combines mechanical cleaning with chemical cleaning, offline tank cleaning and online circulation cleaning, and optimizes the design of the cleaning device. At the same time, a new method for cleaning the medium pipes of continuous-casting equipment is proposed: the pickling solution and passivation solution are passed sequentially into the cleaning tank containing the pipe to be cleaned, the surface rust layer is washed away and stripped, and a corrosion-resistant passivation film is formed on its surface, the liquid flow rate is used to take away corrosive deposits and finally an oil-free blowing dry process is carried out. In addition, through pickling and passivation tests and a response surface analysis, the mechanism of pickling and passivation of Q235 steel pipes was investigated and the cleaning parameters were optimized to effectively improve the quality of the pipe cleaning, which is of great practical significance for the long and efficient operation of continuous-casting equipment.

2 EXPERIMENTAL PART

2.1 Material and specimen preparation

A wide variety of medium pipes are laid in continuous-casting equipment, and their shapes and pipe diameters are different. In this experiment, ϕ40 pipes with a moderate diameter and uniform corrosion degree were selected for the preparation of samples, which were cut into test pieces of ϕ40 × 200 mm and 15 mm × 20 mm × 2 mm with a wire-cutting machine. The chemical composition of the Q235 pipe material steel is shown in **Table 1**.

Table 1: Mass fraction of main trace elements in Q235 steel

Steel model	Chemical element content (= %)					
	Fe	C	Si	Mn	P	S
Q235A	99.165	0.22	0.35	1.40	0.045	0.050
Q235B	99.19	0.20	0.35	1.40	0.045	0.045

2.2 Experimental equipment and procedures

The pipe-cleaning device was modeled in SOLIDWORKS software, as shown in **Figure 1a**. The device includes the cleaning tank, circulating pipeline, circulating water tank, circulating water pump, liquid level display and other necessary components for pipeline cleaning. Due to the large size of the device, the size of the cleaning tank alone exceeds 13 m, so it must be simplified during the experiment. The simplified experimental equipment is shown in **Figure 1b**.

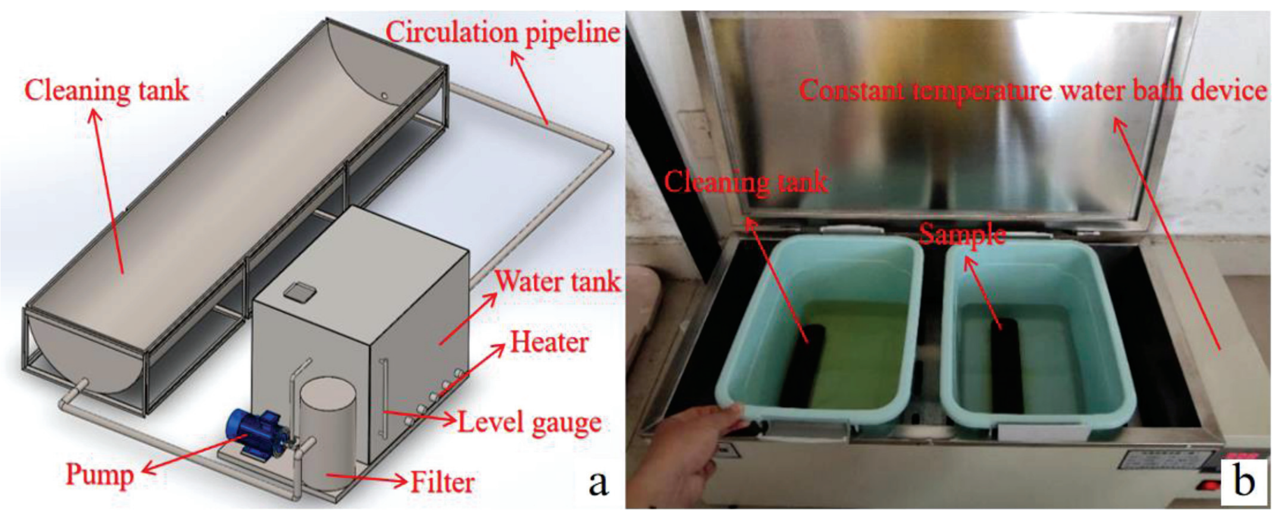

Figure 1: Offline trough circulating cleaning device and its experimental equipment simplification: (a) Cleaning equipment, (b) Simplified experimental equipment

In order to reduce the number of experiments and explore the optimal pickling and passivation conditions of the pipeline from the perspective of multiple factors, an orthogonal experiment with three factors and three levels was designed. The simulation test was carried out according to the orthogonal rotation test design, and the Box-Behnken Design model was selected. The selected significant-effect factors (concentration, temperature, time) were taken as independent variables, and the corrosion depth was used as the evaluation index, and a cross-experiment was carried out. The specific experimental steps are as follows. Prepare acid pickling solutions with concentrations of 10 %, 20 %, and 30 %. The acid pickling solution consists of hydrochloric acid, oxalic acid, and urotropine corrosion inhibitors. Use a pH detector to test the concentration of the acid pickling solution. The three concentrations of the pickling solution were placed in a HH-600 constant-temperature water bath box for water-bath heating, and were controlled at three temperatures of 40 °C, 45 °C and 50 °C. Next, two sample pieces that were prepared are hoisted into the pickling solution. The concentration of the pickling solution is checked every hour to observe changes in the surface morphology of the metal and to analyze the effect of the pickling on the pipe. During the test, the sample is subjected to acid corrosion in strict accordance with the specified test conditions and application sequence, and the three variables of the acid pickling solution concentration, acid pickling temperature, and acid pickling time are strictly controlled. The composition of the acid pickling solution is fixed. When the pickling time reaches 2 h, 4 h and 6 h, the experiment ends and the corrosion depth is measured. The number of samples in each group is 2, the number of parallel samples is 2, and the original sample is reserved for 2, a total of 3 × 3 × 3 × 4 + 2 = 110 pieces.

The passivation test is the same as the pickling test, only the pickling solution needs to be replaced with a passivation solution, and the adjacent sampling observation time is changed from an hour to half an hour. The main components of the passivation solution are 25 % sodium nitrite solution at room temperature and passivation promoters such as hydrofluoric acid and phosphoric acid.

2.3 Testing and characterisation methods

The microscopic morphological changes on the metal surface and at the fracture were doubly characterised using an optical microscope and an electron microscope, and the height difference between the original surface and the corroded surface was measured using a laser confocal scanning microscope, with the average of the three points taken as the depth of corrosion on the metal surface under this condition.

3 RESULTS AND DISCUSSION

3.1 Analysis of pickling and passivation effect under various factors

Metal pickling is to use a prepared acid solution to wash off the rust traces on the surface of the pipeline, which is essentially an electrochemical reaction of hydrogen evolution. As an example of a pickling test at a constant temperature of 45 °C and a concentration condition of 10 %, the effect of the pipe pickling is shown in **Figure 2**. Before pickling, the surface of the test piece is dark brown and the inner wall has almost no reflection, and the rust traces are obvious. During the pickling process, the rust traces of the specimen gradually decreased, revealing the original metal color, but there were still residual metal rust and protruding burrs on the inner and outer walls. After pickling, the inner and outer walls of the pipeline both exhibit a flat and smooth morphology, and the surface of the pipe wall is free of rust marks and residual metal burrs, showing the original metallic grey-white. The pickling effect is remarkable.

The pickling effect is mainly affected by the three factors of pickling-solution concentration, temperature and time. The test results are shown in **Table 2**. For example, after pickling for one hour, the rust product of the floating layer gradually dissolves, and black-brown solid

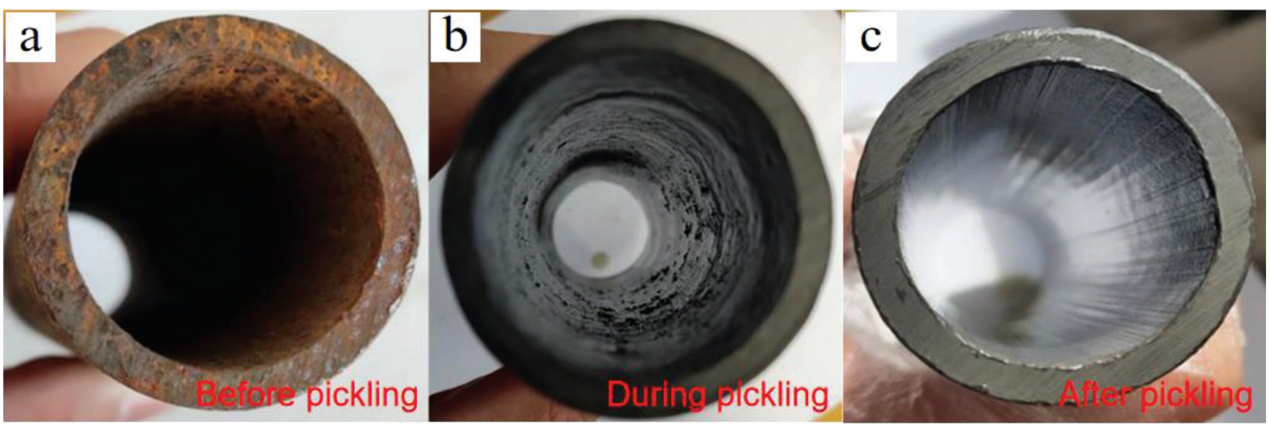

Figure 2: Comparison of the effects of the various stages of the acid-washing experiment: a) before pickling, b) during pickling, c) after pickling

precipitation appears. After pickling for two hours, the surface-rust layer fell off, and the severely rusted area began to dissolve. After pickling for three hours, except for the severely rusted area, there is no obvious rust trace on the inner and outer walls of the test pipe, but there may be metal burrs on the inner wall of the pipe. After pickling for four hours, no rust traces can be seen on the inner and outer walls of the pipe, the pipe wall is thinned by 10.6 μm. At present, the industrial pickling standard for carbon-steel pipes in continuous-casting equipment is that the pipe wall is thinned by 8–12 μm, so the expected pickling effect is achieved. But this does not mean that the higher the concentration of the pickling solution, the better the pickling effect. Under a constant temperature of 45 °C, in the pickling test with a concentration of 30 %, due to the inevitable inclusions, bubbles and other defects in the metal material, the corrosion rate of the defects is not the same as that of carbon steel, while an excessive pickling-solution concentration will exacerbate this gap and cause uneven corrosion. Therefore, in the case that the cleaning effect can be achieved, a relatively low concentration of pickling solution should be selected.

Table 2: Experimental data generated from pickling tests

Factors	Concentration /%	Temperature /°C	Time /h	Corrosion depth /μm
1	10	40	4	8.2
2	10	45	2	5.5
3	10	45	4	10.6
4	10	45	6	14.4
5	10	50	4	12.1
6	20	40	2	7.4
7	20	40	6	13.3
8	20	45	4	16.6
9	20	45	4	17.8
10	20	45	4	15.7
11	20	45	4	16.1
12	20	45	4	18.7
13	20	50	2	9.1
14	20	50	6	18.8
15	30	40	4	15
16	30	45	2	14.8
17	30	45	6	20.1
18	30	50	4	18.6

In addition, the pickling effect is greatly affected by temperature. Under the condition of a constant-temperature water bath below 40 °C, the pickling time of the test pipe is long because it is closer to room temperature. And after 4 h of pickling, there are still small traces remaining in the severely rusted area, and the test effect is not ideal. In contrast, when the temperature is too high (> 50 °C), the acid-corrosion reaction intensifies and releases heat. The solubility of the pickling agent is limited and the metal's thermal conductivity is greater than that of the pickling solution, which easily leads to the formation of pickling-agent crystals in the contact area between the pipe surface and the bottom of the cleaning tank. After removing the crystals, the crystallization area is severely corroded, and the corrosion is uneven, which seriously affects the test effect. Therefore, we can speculate that it is best to control the temperature condition of the pickling test at about 45 °C, as shown in **Figure 3**.

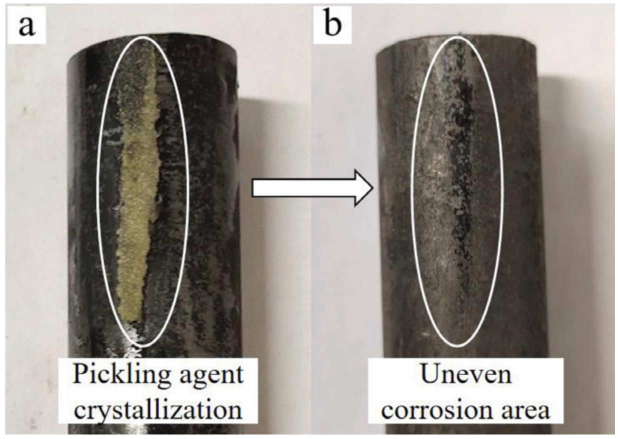

Figure 3: Appearance of pickling-agent crystallization will lead to uneven corrosion: a) pickling-agent crystallization, b) corrosion uneven areas

In fact the vast majority of metals will corrode spontaneously in the general environment. Passivation is essentially an interfacial phenomenon that aims to improve the life and corrosion resistance of pipes by forming a dense oxide film on carbon-steel pipes with a strong oxidising agent, reducing the surface activity of the metal without changing its own properties. In industry, there are multiple standards for the thickness of passivation films on carbon steel, but generally this should not exceed a maximum of 10 μm.

The test piece after pickling was placed in a passivation solution with a mass fraction of 25 % at a constant temperature of 25 °C for 30 min and the passivation effect is shown in **Figure 4**. It can be seen that the metallic silvery white colour of the carbon-steel

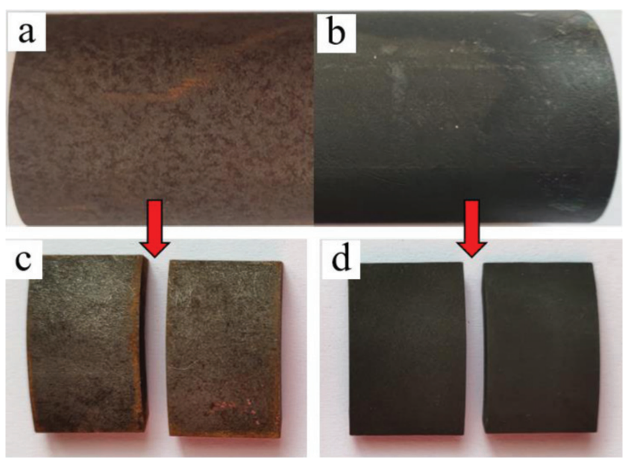

Figure 4: Passivated pipe and its micro morphology: a) not pickled and passivated, b) after pickling and passivation, c) original sample, d) passivation sample piece

surface disappears and is gradually covered by a black metal oxide, forming a dense oxide film. This oxide film can effectively resist the intrusion of corrosive media and has good corrosion resistance. This is not conducive to passivation-film formation because of the small defects left on the metal tube wall after pickling.

3.2 Variation analysis of metal micromorphology on pipe surface

From the perspective of microscopic morphology, the corrosion products on the test tube wall before pickling showed a variety of existing forms, the most important of which include crystal form, crystal cluster form and film form. The corrosion products are scattered and distributed due to the uneven degree of corrosion, as shown in **Figures 5a** to **5c**.

The reason is that the atmospheric corrosion of carbon steel is mainly divided into three stages: the first stage is surface hydroxylation that forms a thin layer of oxide or hydroxide on the surface in a very short time. In the second stage, the atmosphere acts as a thin liquid film attached to the surface of the pipe wall, and its constituent components dissolve in it, resulting in the transformation of a thin layer of oxide or oxyhydroxide into green rust. The third stage is that the number and size of the nuclei of the product gradually increase, and the green rust is transformed into a yellow-brown brittle layer of oxides and hydroxides, the corrosion products are mainly composed of α-FeOOH, γ-FeOOH and γ-Fe_2O_3.

During the pickling process, the rust marks gradually disappeared. Except for the burrs and dents on the inner wall of the pipe and other areas of severe rust, it showed that the surface of the pipe wall gradually presented a relatively flat corrosion layer and some areas began to display the original carbon-steel form. The residual metal burrs on the wall may be caused by impurities inside the metal. Although they are reduced during the pickling process, they still exist. Defects in carbon steel such as bubbles, heavy skins, and pull marks are very likely to cause uneven corrosion, which affects the pickling to a certain extent, as shown in **Figure 5d** and 5e. After pickling, except for small defects in a small part of the pipe-wall surface, most areas show a large area of flat and regular carbon-steel microstructure, indicating that an ideal pickling effect has been achieved. The causes of small defects can be attributed to the impurities soluble in acid contained in the metal pipe wall or serious corrosion due to internal defects, as shown in **Figure 5f** and **5g**.

On the other hand, the passivated sample pieces were placed under the electron microscope and found to have

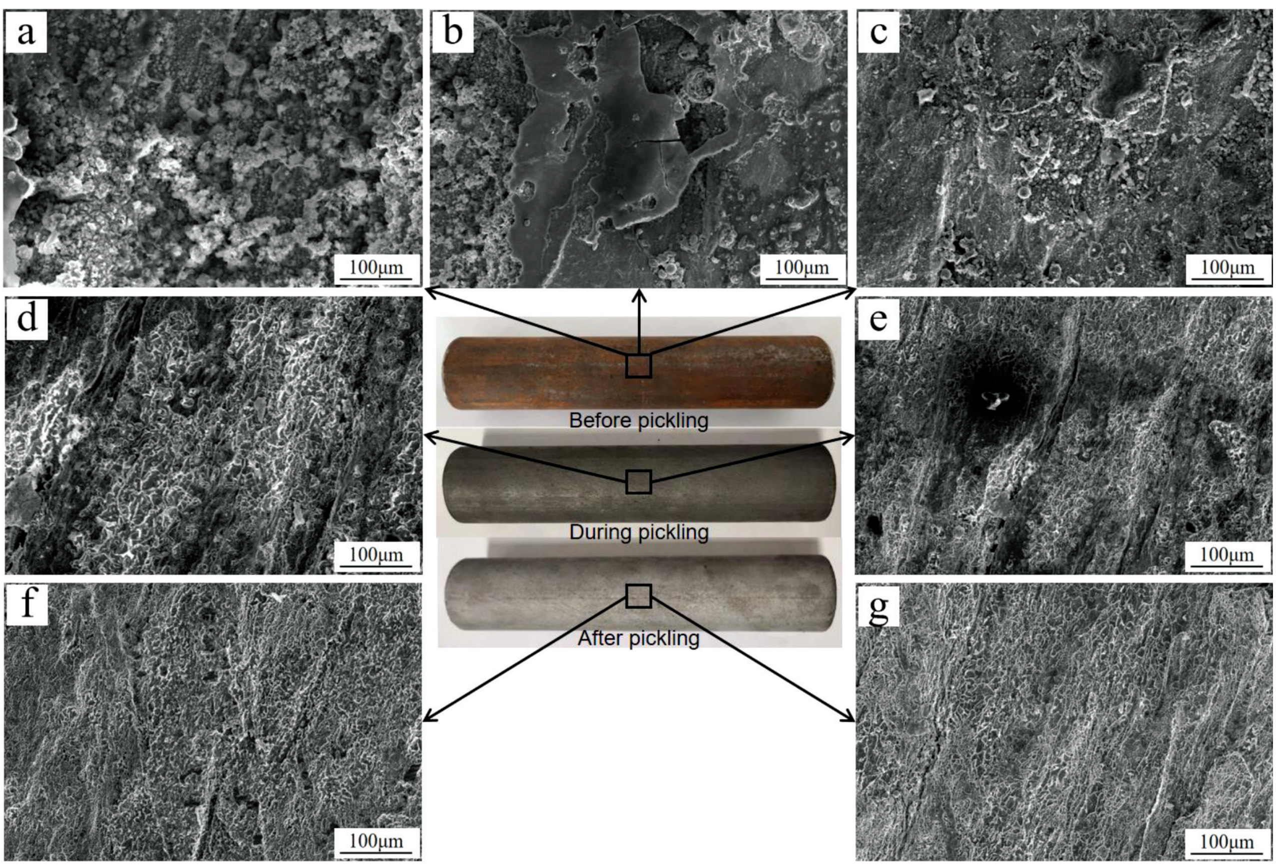

Figure 5: Analysis of metal micromorphology at various stages of pickling experiments: a) crystalline corrosion products, b) film-like corrosion products, c) cluster morphology, d) residual rust, e) metal burr, f) impurity defect, g) good corrosion layer

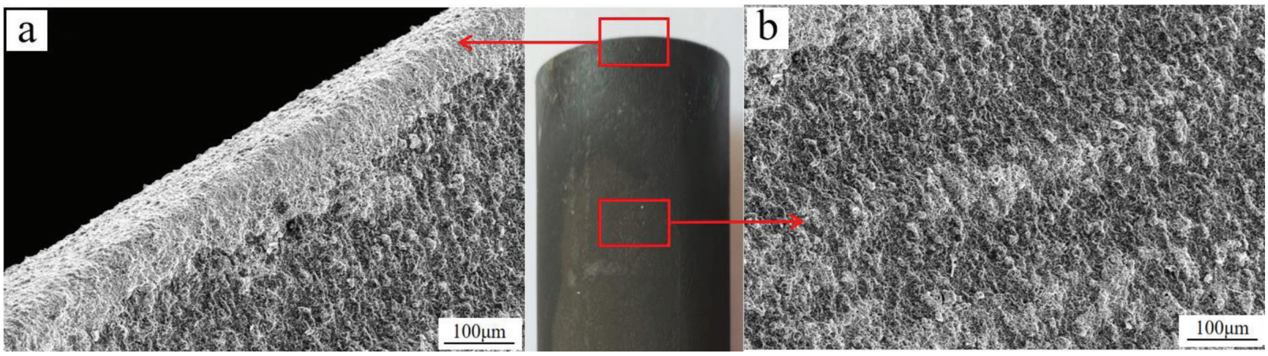

Figure 6: Micro morphology of passive film on pipe wall and fracture: a) fracture, b)pipe wall

a large area of flat and dense metal oxides, replacing the original carbon-steel form. The formation and growth of the passivation film was limited by the small area of surface concavity as air bubbles and inclusions had an effect on the surface flatness of the Q235 steel during the pickling test. At the same time, the fracture is also wrapped with enough passivation products. However, compared with the flat passivation layer, the accumulation of passivation products at the fracture is relatively loose, so that the passivation film at the fracture will break first in the corrosion process, as shown in **Figure 6**.

The microscopic morphological changes at the fracture can be observed more visually under an optical microscope. The surface of the Q235 steel base material before pickling was covered by a stack of thick, dark-brown rust layers, clear but uneven at the boundaries, with varying depths of pitting corrosion and uneven rusting, as shown in **Figure 7a**. After pickling, the surface of the sample was completely free of rust and corrosion, and the boundary of the substrate was flat, smooth, defect-free and clearly visible. This proves that the pickling process effectively removes the rust and corrosion products from the surface of the sample, and the pickling effect is excellent, as shown in **Figure 7b**. In addition, a black, metallic passivation film can be clearly seen on the surface of the passivated sample, which is of uniform thickness and has a high corrosion resistance, effectively protecting the substrate from external corrosive media, as shown in **Figure 7c**.

3.3 Pickling Condition Response Surface Optimization Model

In order to verify the accuracy of the test conclusions and explore the best pickling test conditions, the experimental results in **Table 2** were imported into the Design-Expert 12.0 software to establish a multiple regression equation model. The P values of the regression model of the variance analysis results of the regression model are all less than 0.001, indicating that the regression model is highly significant. The P value of the Lack of Fit item is greater than 0.05, indicating that the lack of fit of the model is not significant, and the regression model has a high degree of fit.

From the P values of concentration, temperature and time, it can be judged that the three test factors have extremely significant effects on the corrosion depth, the influence from large to small is concentration, time and temperature. $P < 0.05$ in the corrosion depth regression model, indicating that the three regression terms have significant interaction effects in the regression model. According to the analysis results of the regression model, a 3D response surface graph of the interaction effect of each factor is drawn, as shown in **Figure 8**.

It was found that the interactive responses of temperature and concentration, temperature and time are significant in the process of corrosion depth from 5 μm to 20 μm, especially the interactive response of temperature and time. Between 40 °C and 45 °C there is a relatively flat area around 3 h to 5 h among them, and it is specu-

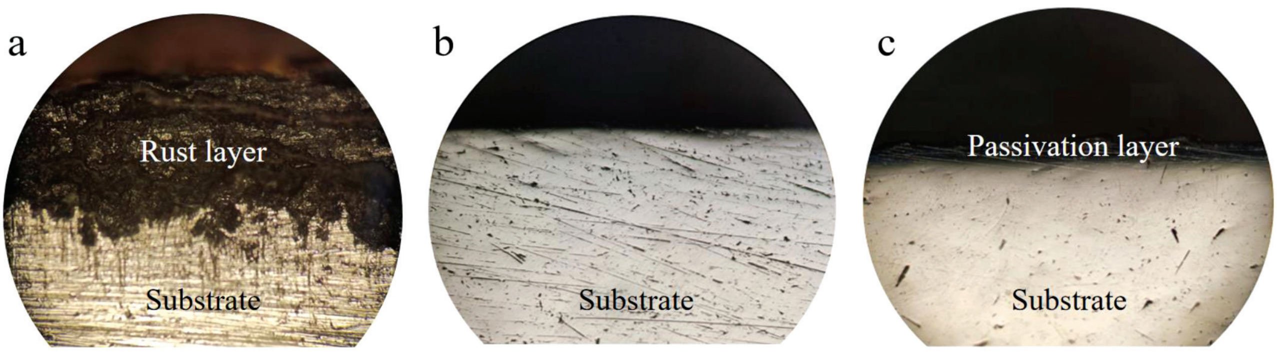

Figure 7: Analysis of microscopic morphological changes at the fracture: a) before pickling, b) after pickling, c) after passivation

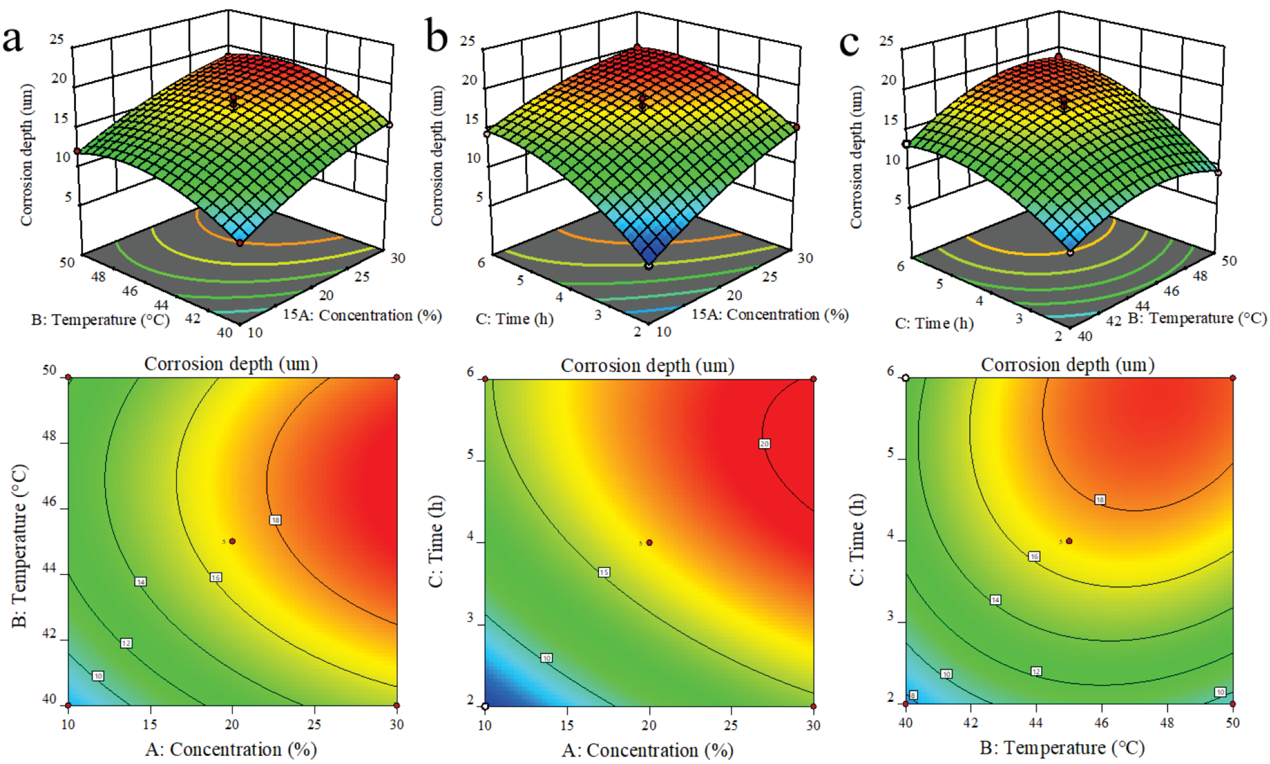

Figure 8: Response graph of the interaction of concentration, temperature and time: a) temperature and concentration, b) time and concentration, c) temperature and time

lated that there is an optimal solution in this area. In contrast, the corrosion depth increases at a certain rate with the increase of time and concentration during the whole process, and the rise rate tends to be linear. The interaction curve between time and concentration tends to be mild, and the interaction is not obvious when the factor level is low.

The response-model formula is:

$$R=-219.78+0.992f_c+9.117f_w+3.093f_t- \\ -0.002f_cf_w-0.045f_cf_t+0.095f_wf_t- \\ -0.011f_c^2-0.101f_w^2-0.576f_t^2 \quad (1)$$

Where R is the corrosion depth, µm, f_c is the concentration, %; f_w is the temperature, °C, and f_t is the time, h_0.

The experimental results are optimized using the numerical optimization module. According to the quality standard for the pickling of industrial medium carbon-steel pipes specified in GB/T 25146-2010, the value of the target expected corrosion depth is 10 µm. Among the 72 kinds of results calculated by the optimization solver, the best experimental condition is to place the pipeline at a constant temperature of 42 °C and pickle it in a pickling solution with a concentration of 10 % for 3.8 h. This optimization scheme is consistent with the previous experimental conclusions.

4 CONCLUSIONS

1) This paper proposes a new method and new device for cleaning the media pipes of continuous-casting equipment and combines orthogonal experiments with response surface analysis for process optimisation. The optimum cleaning conditions for Q235 steel pipes are injecting a pickling solution with a constant temperature of 42 °C and a mass fraction of 10 % for pickling for 3.8 h, and then inject a passivation solution with a constant temperature of 25 °C and a mass fraction of 25 % for passivation for 0.5 h.

2) The chemical activity of the pickling solution drives the dissolution rate of the rust defect area on the Q235 steel surface higher than other areas, and the entire surface tends to be flat and uniform after pickling. The strong oxidation properties of the passivation solution enable the Q235 steel's surface to rapidly generate a very thin oxide film, which makes the surface stability of the metal abruptly increase, which is essentially an interfacial phenomenon and does not change the nature of the metal itself.

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