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Optimization and corrosion behavior of copper plating process on steel surface

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Abstract
Copper plating on steel surface is considered an effective means to improve its corrosion resistance. However, the recent copper plating process using cyanide electrolytes involves CN^- with high toxicity, and there is a great safety risk to process control and maintenance, so it is a trend to develop the non-cyanide copper plating process. In this study, four non-cyanide copper plating methods were selected and the corrosion resistance characteristics of the plated samples are compared to obtain the best copper plating method and parameters.

Keywords: Copper plating, corrosion resistance, HEDP, electroless copper plating, EIS analysis

Introduction
Steel has become the most widely used material in the construction and transportation fields because of its low cost, simple production process, good plasticity, toughness and processability. However, steel is greatly limited in its application due to its easy corrosion. Hence, the research to improve the corrosion resistance of steel has been a hot topic in the iron and steel field in recent years [1-3]. The addition of copper to steel can significantly reduce the corrosion rate of steel in the atmosphere. At present, the production method of steel containing copper is still to add a certain amount of copper or copper alloy in the smelting process, and use the corresponding heat treatment process to prevent the corrosion of steel. However, this method has a strict production process, results in the waste of copper resources, and it is easy to cause copper brittleness [4-6]. Therefore, the surface treatment method including electroplating is effective in improving the corrosion resistance of steel by plating copper on steel surface. However, the traditional copper plating process has a great risk of containing CN^- with high toxicity, so several non-cyanide copper plating processes are currently being developed [7]. Based on the previous studies, the corrosion behavior of the samples obtained by the non-cyanide copper plating processes such as citrate copper plating, citric acid-tartrate copper plating, HEDP copper plating, and electroless copper plating, were investigated using electrochemical impedance spectroscopy (EIS) and polarization curves, and the suitable plating method and parameters for enhancing corrosion resistance of steel were discussed.

Materials and Experiment Methods
A. Materials
The experiment material is carbon steel, whose chemical composition is C0.18, Si0.25, Mn0.5, S0.018, P0.016, and the balance is Fe. The size of the machined carbon steel according to the test requirements is 10mm×10mm×3mm. The wire is connected and except for the reserved working face (10mm×10mm), other surfaces were sealed with epoxy resin and curing agent with a mass ratio 3:1. The machined surface was treated by grinding, polishing and soaked in ethanol, ultrasonically washed for 30 min then washed with deionized water and dried.

B. Experiment Procedure
For the citric acid-tartrate copper plating, 60g/L $CuCO_3 \cdot Cu(OH)_2 \cdot H_2O$ + 250g/L $C_6H_8O_7 \cdot H_2O$ + 35g/L $C_4H_4KNaO_6 \cdot 4H_2O$ + 15g/L $NaHCO_3$ + 0.01g/L SeO_2 were selected as plating solution. Under the conditions of bath temperature 30~40 ℃, current density 1.5~2.5A/dm^2 and bath pH value 8.5~9, copper was electrodeposited on the surface of the selected carbon steel with copper plate as anode and carbon steel as cathode for 5~45min. In the case of the citrate copper plating, 30g/L $CuSO_4$ + 147g/L $Na_3C_6H_5O_7 \cdot 2H_2O$ + 30g/L $C_4H_4KNaO_6 \cdot 4H_2O$ + 20g/L $NaHCO_3$+8g/L $KHNO_3$ were selected as electrodeposition solution. Under the conditions of bath temperature 70 ℃, current density 1.5~5A/dm^2 and

bath pH value 11, copper was electrodeposited on the surface of the selected carbon steel with copper plate as anode and carbon steel as cathode for 15~30min. For the HEDP copper plating, 150g/L HEDP ($C_2H_8O_7P_2$) + 14g/L $CuCO_3·Cu(OH)_2·H_2O$ + 40g/L K_2CO_3 + 0.1g/L $C_{12}H_{25}NaO_3S$ were selected. Under the conditions of bath temperature 40~60 ℃, current density 1~2A/dm^2 and bath pH value 9, copper was electrodeposited on the surface of the selected carbon steel for 30~60min. For the electroless copper plating, plating solution was 40g/L $CuSO_4$ + 30mL/L H_2SO_4 + 0.2g/L CH_4N_2S + 0.24g/L $HO(CH_2CH_2O)_nH$ + 0.25g/L $FeSO_4·7H_2O$ + 25g/L KBr. Under the conditions of bath temperature 25 ℃, copper was chemically deposited on the surface of the selected carbon steel for 1min.

The plated samples were washed with deionized water, dried and used for polarization curve and electrochemical impedance spectroscopy (EIS) analysis in an electrochemical analyzer(CHI660E, China). Three-electrode system was used for the electrochemical corrosion test. The working electrode was the sample, the reference electrode was the saturated calomel electrode (SCE) and the auxiliary electrode was the platinum electrode. The corrosion solution, 0.168g/L $NaHCO_3$ + 1.42g/L Na_2SO_4 + 6.82g/L H_3BO_3 + 3.636g/L $Na_2B_4O_7$ + 1.775 NaCl was used. The electrochemical corrosion test was carried out at room temperature. The working electrode was immersed in the corrosion solution for 15min before test and corrosion test was performed after the open-circuit potential was stabilized. The polarization curves were obtained in the range of the final potential -1.0~1.5V at a scanning rate of 5mV/s. EIS analysis was performed at open-circuit potential, with a sinusoidal amplitude of 5mV and a frequency range of 0.01Hz to 100kHz.

Results and Discussion

Table 1 shows the thickness measurements of copper layers obtained by different plating methods, and Figure 1 shows the photographs of the plated samples obtained by different plating methods.

Table 1: Thickness of copper coatings

Sample	Thickness/㎛
Citric acid-tartrate copper plating	8.15
Citrate copper plating	6.25
HEDP copper plating	6.9
Electroless copper plating	4.9

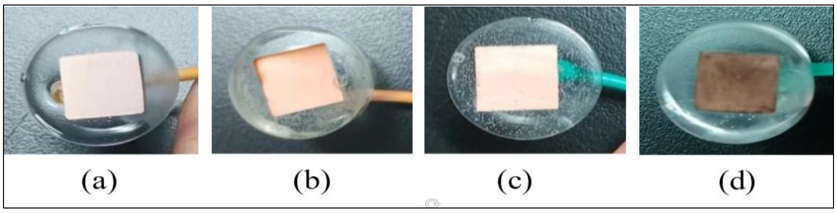


Fig 1: Photograph of the plated samples; (a) citric acid-tartrate copper plating; (b) citrate copper plating; (c) HEDP copper plating; (d) electroless copper plating

A. Polarization Curve

Figure 2 shows the polarization curves of copper coatings with different plating methods.

The corrosion potential Ecorr and corrosion current density Icorr were obtained by Tafel extrapolation and the results are shown in Table 2.

Icorr is an important parameter to evaluate the kinetics of corrosion reactions, usually proportional to the rate of corrosion of the material in the corrosive medium. The smaller the Icorr is, the lower the corrosion reaction rate is and the stronger the corrosion resistance of the material is. As can be seen from Figure 2 and Table 2, the corrosion current density of the citric acid-tartrate copper plated sample is the lowest and the corrosion potential is low. Next, HEDP copper plating had a low corrosion current density. The polarization behavior of citric acid-tartrate copper plating, citrate copper plating, and HEDP copper plating is similar, but the electroless copper plating has the highest corrosion potential and the corrosion current density is much higher than the other plated samples.

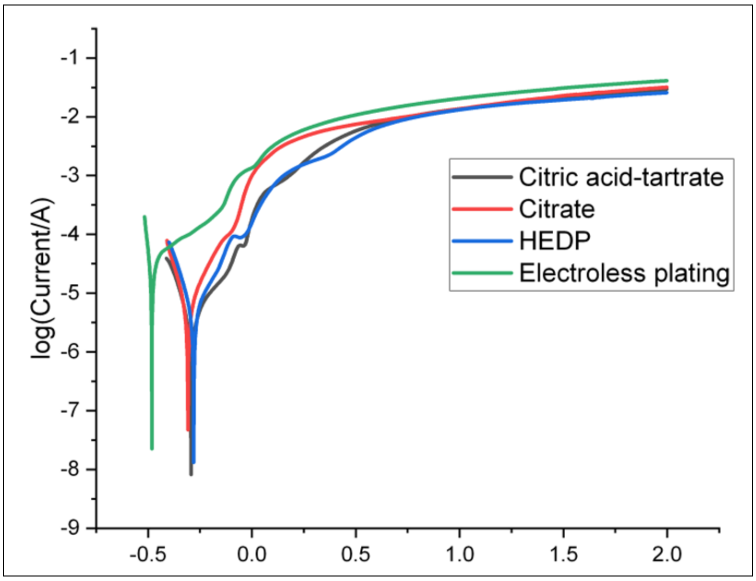


Fig 2: Polarization curves of different copper coatings

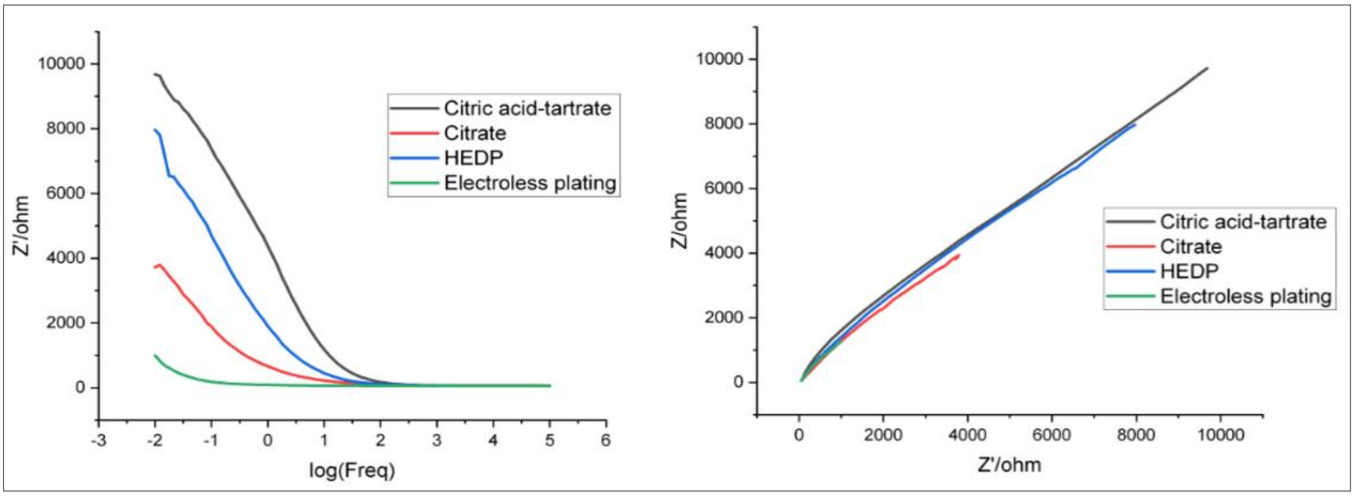
Table 2: Corrosion potential and corrosion current density

Sample	E_{corr} / mV(SCE)	I_{corr}/(μA/cm^2)
Citric acid-tartrate copper plating	-293	3.98
Citrate copper plating	-308	7.94
HEDP copper plating	-280	5.62
Electroless copper plating	-482	35.48

B. EIS

Figure 3 shows Bode plot and Nyquist plot of different copper coatings.

The larger the radius of the capacitive resistance arc in the Nyquist plot is, the better the corrosion resistance is. The arc radius of the citric acid-tartrate copper plated sample is the largest, followed by the HEDP copper plated sample.



A B

Fig 3: EIS plots of different copper coatings; (a) Bode plot; (b) Nyquist plot

Bode plot shows similar performance for the four plating methods in the high-frequency region, but there are some differences in the low-frequency region. The citric acid-tartrate copper plated sample has the largest distance of the curve in the Y-axis, followed by the HEDP copper plated sample. As the distance of the curve in the Y-axis represents the magnitude of the resistance, the corrosion resistance of the citric acid-tartrate copper plated sample is the best. The results of the Bode plot are also consistent with those of the Nyquist plot and with the polarization curve. Generally, the corrosion potential of carbon steel Ecorr is about -600mV and its corrosion current density Icorr is about 53μA/cm^2 [8]. The corrosion potential of the citric acid-tartrate copper plated sample Ecorr is 293mV and 280mV, which is higher than carbon steel and their corrosion current density Icorr is 3.98μA/cm^2 and 5.62μA/cm2, which is much lower than carbon steel. Thus, the corrosion resistance of steel with citric acid-tartrate copper plating and HEDP copper plating is obviously enhanced.

Conclusion

From the above discussion, the following conclusions can be drawn:

The copper coating on the surface of carbon steel can significantly improve corrosion resistance by increasing corrosion potential and corrosion current density. Among the four different non-cyanide copper plating processes considered in this study, the citric acid-tartrate copper plating process is the best in terms of corrosion behavior. And the following one is HEDP copper plating. The optimum conditions for citric acid-tartrate copper plating are temperature 40 ℃, current density 1.5A/dm^2, pH value of plating solution 8.5~9 and plating time 30~40 min.

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