International Journal of ELECTROCHEMICAL SCIENCE www.electrochemsci.org

Short Communication

Preparation of Zn-Ca Phosphate Coating in Presence of PTFE on 45 Steel and its Corrosion Properties in Simulated Seawater

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Received: 4 June 2022 / Accepted: 5 July 2022 / Published: 7 August 2022

Zn-Ca phosphate coating in presence of PTFE was prepared on the surface of 45 steel by dipping method using a phosphate solution containing PTFE particles. By investigating the influence of the amount of PTFE particles added, stirring rate, phosphate solution temperature and phosphate time on the thickness and PTFE particles content of Zn-Ca phosphate coating in presence of PTFE, the optimal process parameters for preparing Zn-Ca phosphate coating in presence of PTFE were determined as follows: amount of PTFE particles added 16 g/L, stirring rate 200 r/min, phosphate solution temperature 65° C and phosphate time 18 min. The comparative experimental results show that the thickness of the optimized Zn-Ca phosphate coating in presence of PTFE reaches 9.5 μ m with the highest PTFE particle content (approximately 11%) which can slow down the phosphate coating corrosion process. After immersion in simulated seawater for different time, the corrosion current density of the optimized Zn-Ca phosphate coating in presence of PTFE gradually increases and then remains stable. The optimized Zn-Ca phosphate coating in presence of PTFE can inhibit the electrochemical reaction and provide ideal corrosion protection for 45 steel.

Keywords: Zn-Ca phosphate coating in presence of PTFE; Corrosion protection; PTFE particles; Stirring rate; phosphate solution temperature; phosphate time

1. INTRODUCTION

45 steel has high hardness, good toughness and excellent wear resistance, which is suitable for manufacturing shafts, gears, tie rods and bases of port mechanical brakes [1-4]. Since the port mechanical brake is generally used in marine environment, factors including temperature, humidity changes and chloride ions will affect the corrosion resistance of the components, thereby reducing the reliability of the mechanical brake. Therefore, it is hugely significant to take measures to improve the

corrosion resistance of 45 steel, as this will ensure the basic function of the mechanical brakes can be performed [5-8].

Many surface treatment technologies exist that are suitable for use in the improvement of the physical and chemical performance of metal materials, such as electrodeposition, electroless deposition, phosphate, passivation and so on [9-14]. Meanwhile, phosphate coating is generally used as a corrosion protection layer on the surface of steel and has been widely used, effectively slowing down the steel corrosion process. However, the phosphate coating has a microscopic porous structure, and the pores can easily become corrosion channels, resulting in a decrease in the corrosion resistance of the phosphate coating. To significantly improve the corrosion resistance of phosphate coating in a more severe environment (such as the marine atmosphere environment or the environment where various corrosive media coexist, etc.), some measures must be taken to fill or seal the pores of the phosphate coating. Relevant studies have found that insoluble particles accompanied by phosphate precipitation crystals can fill the pores in the phosphate coating, thereby effectively improving the corrosion resistance of the phosphate coating [15-17].

PTFE particles possess excellent chemical stability and corrosion resistance and they can withstand the corrosion of all strong acids, strong alkalis and chloride salts [18-19]. If PTFE particles are encapsulated in the phosphate coating together with phosphate precipitation crystals, the corrosion resistance of the phosphate coating will be significantly improved due to the role played by filling pores. In this paper, Zn-Ca phosphate coating in presence of PTFE was prepared on the surface of 45 steel by dipping method and the corrosion protection of the phosphate coating for 45 steel was studied.

2. EXPERIMENT

2.1 Materials

45 mm \times 22 mm \times 3 mm 45 steel samples were used as the substrate. The surface of samples was polished and immersed in the alkaline solution with sodium hydroxide (40 g/L), sodium carbonate (15 g/L) and sodium phosphate (8 g/L) at 60°C for 12 min to completely remove oil. After cleaning, it was immersed in dilute hydrochloric acid for 1 min to activate the surface, and finally the substrate was ultrasonically cleaned in ethanol and pure water for 5 min, and dried in cold air.

2.2 Preparation of Zn-Ca phosphate coating in presence of PTFE

Zn-Ca phosphate coating in presence of PTFE was prepared on the surface of the 45 steel by dipping method. The main composition of phosphate solution and process parameters were shown in Table 1. In particular, the steps for preparing the phosphate solution were as follows: (1) zinc oxide, phosphoric acid, calcium oxide, nitric acid, tartaric acid and nickel nitrate were used to prepare the basic phosphate solution in a certain proportion; (2) PTFE particles that were produced by DuPont of the United States were slowly added into the phosphate solution and stirred.

The phosphate solution temperature was maintained at the set value by heating in a water bath with integrated magnetic stirring function. PTFE particles were suspended and dispersed in the phosphate solution by stirring at a constant rate. During the experiment, five Zn-Ca phosphate coating in presence of PTFE samples were prepared respectively by changing the amount of PTFE particles added, stirring rate, phosphate solution temperature and phosphate time. At the same time, a Zn-Ca phosphate coating sample was prepared by using the basic phosphate solution for comparison.

Chemical agents and	Value
process parameters	
$ZnO / (g \cdot L^{-1})$	25
$H_{3}PO_{4} / (mL \cdot L^{-1})$	8
$CaO / (g \cdot L^{-1})$	13.5
$HNO_3 / (mL \cdot L^{-1})$	10
$C_4H_6O_6 / (g \cdot L^{-1})$	0.6
$Ni(NO_3)_2 / (g \cdot L^{-1})$	1.5
PTFE particles added/	2~20
$(g \cdot L^{-1})$	
stirring rate / $(r \cdot min^{-1})$	50~250
temperature / °C	45~85
time / min	8~32

Table 1. Main composition of phosphate solution and process parameters

2.3 Testing of Zn-Ca phosphate coating in presence of PTFE

2.3.1 Surface morphology, composition and PTFE particles content

The surface morphology of Zn-Ca phosphate coating in presence of PTFE was observed by Nova NanoSEM450 scanning electron microscope equipped with an energy dispersive spectrometer, and the mass fraction of each element was obtained by composition analysis. According to the mass fraction of F element, the content of PTFE particles in the phosphate coating was calculated.

2.3.2 Thickness

The thickness of Zn-Ca phosphate coating in presence of PTFE was obtained by using Mini TEST600B-FN thickness gauge according to the method of multi-point measurement and averaging, and the measurement accuracy was $0.1 \mu m$.

2.3.3 Corrosion resistance

Parstat 2273 electrochemical workstation was used to test the polarization curves of Zn-Ca phosphate coating in presence of PTFE after immersion in simulated seawater (containing 3.5%

sodium chloride + 2‰ sodium sulfate + 1‰ magnesium chloride) at 25°C for different time. The reference electrode, auxiliary electrode and working electrode are used as saturated calomel electrode, platinum electrode and Zn-Ca phosphate coating in presence of PTFE with exposed area of 1 cm², respectively. The scan rate was 1 mV/s, and the potential range was -250 mV to +250 mV. The test data were imported into PowerSuite software to get the corrosion current density and polarization resistance, and then the corrosion resistance of different Zn-Ca phosphate coating in presence of PTFE was evaluated.

In addition, Nova NanoSEM450 scanning electron microscope was used to observe the surface morphology of Zn-Ca phosphate coating in presence of PTFE in simulated seawater.

3. RESULT AND DISCUSSION

3.1 Effect of amount of PTFE particles added and stirring rate on thickness and PTFE particles content of phosphate coating

PTFE particles are encapsulated in the phosphate coating together with the phosphate precipitation crystals, thereby forming the Zn-Ca phosphate coating in presence of PTFE. Figure 1 shows the thickness and PTFE particles content of the phosphate coating prepared when the amount of PTFE particles added is ranged from 2 g/L~20 g/L. It can be seen from Figure 1 that with the increase of the amount of PTFE particles added, the thickness of the phosphate coating first remains basically unchanged and then decreases, while the content of PTFE particles increases gradually. The reason is that when the amount of PTFE particles added is small, only a small amount of particles are suspended and dispersed in the phosphate solution, and fewer particles are encapsulated in the phosphate coating together with the phosphate precipitation crystals, so the phosphate coating contains fewer particles. As shown in Figure 2, PTFE particles in the phosphate coating can not only adhere to the surface of the grains, but also fill the gaps between the grains, which can prevent the penetration and diffusion of corrosive ions to the interface of the substrate/phosphate coating. Therefore, the content of PTFE particles has a certain influence on the corrosion resistance of the coating which is also reported by some researchers [20-22]. As a means of improving the corrosion resistance of the phosphate coating, the content of PTFE particles in the phosphate coating must be increased. Generally, the higher the content of PTFE particles, the more particles in the dispersed state in the phosphate coating, and the ability to block the penetration of corrosive ions into the interface of the substrate/phosphate coating and inhibition of the electrochemical corrosion is enhanced. Conversely, if the content of PTFE particles is low, and there are fewer PTFE particles in the phosphate coating, effectively blocking the penetration of corrosive ions into the interface of the substrate/phosphate coating will be difficult and electrochemical corrosion can easily occur.



Figure 1. Effect of amount of PTFE particles added on thickness and PTFE particles content of phosphate coating



Figure 2. Schematic diagram of Zn-Ca phosphate coating in presence of PTFE formed by PTFE particles accompanied with phosphate precipitation

Stirring plays an important role in the chemical reaction [23-24]. The liquid flow disturbance generated by stirring drives the PTFE particles to adsorb on the surface of the substrate and enter the phosphate coating through phosphate precipitation and crystallization. Figure 3 shows the thickness and PTFE particles content of the phosphate coating prepared when the stirring rate is ranged from 50 r/min to 250 r/min. It can be seen from Figure 3 that as the stirring rate increases, the thickness of the phosphate coating does not change much, while the content of PTFE particles increases first and then decreases. When the stirring rate is slow, the liquid flow disturbance strength is low, resulting in uneven dispersion of particles in the phosphate solution, and the amount of PTFE particles that are attached to the surface of the substrate and together with phosphate precipitation and crystallization is small, resulting in a lower number of PTFE particles in the phosphate coating. As the stirring rate increases, the liquid flow disturbance strength increases, so that the particles are in a better suspension and dispersion state in the phosphate solution, and more and more particles enter the phosphate coating along with the phosphate precipitation and crystallization. Therefore, the PTFE particles in the phosphate coating increases. However, when the stirring rate is too fast, due to the large scouring force, the particles attached to the surface of the substrate potentially returning to the phosphate solution and prevents the particles from becoming covered by the phosphate coating, resulting in a decrease in the PTFE particles content in the phosphate coating.



Figure 3. Effect of stirring rate on thickness and PTFE particles content of phosphate coating

3.2 Effect of phosphate solution temperature and phosphate time on thickness and PTFE particles content of phosphate coating

Phosphate solution temperature is an important factor that determines whether the phosphate coating can be successfully formed. It is reported that the temperature can affect the thickness, composition, morphology, structure of coatings [25-27]. Figure 4 shows the thickness and PTFE particles content of the phosphate coating prepared when the phosphate solution temperature is ranged from 45°C to 85°C. It can be seen from Figure 4 that as the phosphate solution temperature increases from 45°C to 65°C, the thickness of the phosphate coating increases, along with the increase of PTFE particles content. This is because the increase of the phosphate solution temperature promotes the hydrolysis and crystallization reaction of phosphate, shortens the induction period of coating formation, and accelerates the coating formation rate, which results in an increase of the thickness. In addition, the increase of phosphate solution temperature can also enhance the activity of free ions in the phosphate solution, while also promoting the entry of PTFE particles into the phosphate coating together with phosphate precipitation and crystallization, so that the PTFE particles content in the phosphate coating increases. However, as phosphate solution temperature continues to increase from 65°C to 85°C, the thickness of the phosphate coating is stable, but the PTFE particles content decreases. The reason is that the high phosphate solution temperature leads to an increase in the amount of sediment that is generated by the phosphate side reaction, thereby hindering the adhesion of PTFE particles and resulting in a reduction in particles covered on the phosphate coating.



Figure 4. Effect of phosphate solution temperature on thickness and PTFE particles content of phosphate coating

Phosphate time is set based on the composition of phosphate solution, which affects both phosphate coating thickness and performance. Figure 5 shows the thickness and PTFE particles content of the phosphate coating prepared when the phosphate time is ranged from 8 min to 32 min. Regarding to Figure 5, with the extension of phosphate time, the thickness of the composite phosphate coating increases first and then decreases, but the PTFE particles content gradually increases and then remains basically unchanged. The reason is that when the phosphate time is proper, the phosphate is continuously precipitated and crystallized, which is manifested as a gradual increase in the thickness of the phosphate coating. At the same time, the number of PTFE particles content in the phosphate coating. However, the phosphate reaction is not continuous and when the phosphate time exceeds a certain limit, the phosphate coating formation rate will gradually slow down. After that, the PTFE particles content in the phosphate coating remains basically unchanged.



Figure 5. Effect of phosphate time on thickness and PTFE particles content of phosphate coating

In summary, under the condition of amount of PTFE particles added 16 g/L, stirring rate 200 r/min, phosphate solution temperature 65° C and phosphate time 18 min, the as-prepared Zn-Ca phosphate coating in presence of PTFE has a thickness of 9.5 µm and PTFE particles content of 11%. For the convenience of description, the as-prepared Zn-Ca phosphate coating in presence of PTFE is named as optimized Zn-Ca phosphate coating in presence of PTFE.

3.3 Morphology and composition of the optimized Zn-Ca phosphate coating in presence of PTFE

Figure 6 shows the morphology of 45 steel and the optimized Zn-Ca phosphate coating in presence of PTFE while Figure 7 shows the EDS spectrum of the optimized Zn-Ca phosphate coating in presence of PTFE. It can be seen from Figure 6 that a phosphate coating with good uniformity is prepared on the surface of 45 steel with large number of PTFE particles attached to the surface. PTFE particles have excellent chemical stability and corrosion resistance, and they can effectively prevent the penetration of chloride ions and water molecules by adhering to the surface of phosphate coating and play a role in slowing down the corrosion [28-29]. PTFE doped coatings with excellent corrosion resistance are also prepared by some researchers [30-31].



(a) 45 steel (b) optimized Zn-Ca phosphate coating in presence of PTFE

Figure 6. Morphology of 45 steel and the optimized Zn-Ca phosphate coating in presence of PTFE



Figure 7. EDS spectrum of the optimized Zn-Ca phosphate coating in presence of PTFE

3.4.1 Polarization curves analysis

In order to investigate the corrosion protection of the optimized Zn-Ca phosphate coating in presence of PTFE for 45 steel, the samples are immersed in simulated seawater for different time. Figure 8 shows the polarization curves of 45 steel and the optimized phosphate coating immersed in simulated seawater for different time, and Table 2 shows the fitting results of polarization curves. Combined with Figure 8 and Table 2, it can be seen that the polarization curves of the optimized phosphate coating all shift to the upper left, indicating that the corrosion potential of the optimized phosphate coating is positive and the corrosion current density is lower than that of the 45 steel. With the prolongation of immersion time, the corrosion current density of 45 steel and the optimized phosphate coating changes significantly. The corrosion current density of 45 steel shows a gradually increasing trend, and the increase amplitude is approximately linear. But the corrosion current density of the optimized phosphate coating gradually increases and then basically maintains a stable trend.

Relevant studies have shown that the corrosion potential is mainly related to the composition of the material surface [32-33]. During the corrosion process, corrosion products are formed and cover the surface of 45 steel and the optimized phosphate coating. Although the corrosion products are different in terms of composition, the amount of corrosion products is lower, meaning that the corrosion potential changes less. The corrosion current density is mainly related to the surface structure of the material. With the prolongation of immersion time, the damage caused by corrosive media such as chloride ions to the surface structure of 45 steel and the optimized phosphate coating increases, resulting in the significant change of corrosion current density. There is a large number of dispersed PTFE particles attached to the grain surface and grain gap of the optimized phosphate coating, which can effectively prevent the penetration of chloride ions and water molecules in simulated seawater, and show high electrochemical corrosion resistance. With the prolongation of immersion time, the surface structure of the optimized phosphate coating is not seriously damaged, and it is still able to prevent the penetration of chloride ions and water molecules of the grains and filling gaps between the grains can also inhibit the electrochemical reaction and reduce the corrosion rate.





Figure 8. Polarization curves of 45 steel and the optimized Zn-Ca phosphate coating in presence of PTFE immersed in simulated seawater for different time

Different Corrosion potential/ V Corrosion current density/ $(A \cdot cm^{-2})$ samples immediately immersed immediately immersed immersed immersed immersed after for after for 6 days for 6 days for 2 days for 2 days immersion immersion 10 days 7.67×10-5 5.81×10-5 45 steel -0.498 -0.517 -0.534 -0.545 1.72×10-5 optimized -0.461 1.87×10⁻⁶ 3.32×10⁻⁶ 4.86×10⁻⁶ phosphate -0.422 -0.435 -0.450

Table 2. Fitting results of polarization curves

coating

Figure 9 shows the variation trend of protection efficiency of the optimized Zn-Ca phosphate coating in presence of PTFE on 45 steel after immersion in simulated seawater for different time. It can be seen from Figure 9 that the protection efficiency of the optimized phosphate coating on 45 steel is 89.1% immediately after immersion. Relevant studies have shown that higher protection efficiency means that the coating can resist corrosion better to provide ideal corrosion protection for 45 steel [34-35]. With the prolongation of immersion time, the continuous penetration of chloride ions and water molecules in the simulated seawater leads to a gradual decline in the corrosion resistance of 45 steel. However, due to the excellent chemical stability and corrosion resistance of a large number of PTFE particles attached to the grains surface and filling the grains gaps, the electrochemical corrosion can be effectively inhibited, and the corrosion rate of the phosphate coating can be reduced.

immersed

for

10 days

 1.08×10^{-4}

5.40×10⁻⁶



Figure 9. Variation trend of protection efficiency of the optimized Zn-Ca phosphate coating in presence of PTFE on 45 steel after immersion in simulated seawater for different time

3.4.2 Corrosion morphology analysis

Figure 10 shows the corrosion morphology of 45 steel and the optimized Zn-Ca phosphate coating in presence of PTFE after immersion in simulated seawater for different time. It can be seen from Figure 10 that after immersion in simulated seawater for 2 days, some corrosion products form on the surface of 45 steel, while few corrosion products form on the surface of the optimized phosphate coating. As the immersion time is extended to 10 days, the corrosion products on the surface of 45 steel increases significantly, thereby indicating that the surface structure of 45 steel is severely damaged, resulting in a significant decrease in its corrosion resistance. The immersion time on corrosion products on the surface of the optimized of the optimized phosphate coating in presence of PTFE are still few and the grains are not significantly damaged. This provides further confirmation that the surface structure of the optimized phosphate coating has not been seriously damaged. It can still effectively inhibit the electrochemical reaction, and can provide ideal corrosion protection for 45 steel within a certain period.



(a) 45 steel immersed for 2 days (b) optimized phosphate coating immersed for 2 days



(a) 45 steel immersed for 10 days (b) optimized phosphate coating immersed for 10 days

Figure 10. Corrosion morphology of 45 steel and the optimized Zn-Ca phosphate coating in presence of PTFE after immersion in simulated seawater for different time

4. CONCLUSIONS

(1) The optimal process parameters for preparing Zn-Ca phosphate coating in presence of PTFE are determined and investigated in the paper which is amount of PTFE particles added 16 g/L, stirring rate 200 r/min, phosphate solution temperature 65° C and phosphate time 18 min. The optimized phosphate coating with a thickness of 9.5 µm, PTFE particles content of approximately 11%, and possessing excellent corrosion resistance was prepared on the surface of 45 steel. The optimized phosphate coating can effectively inhibit the electrochemical corrosion, and can provide ideal corrosion protection for 45 steel within a certain period.

(2) After immersion in simulated seawater for different time, the corrosion current density of 45 steel shows a gradually increasing trend and the increase amplitude was approximately linear, while the corrosion current density of the optimized phosphate coating shows a gradual increase and then keeps stable. A large number of PTFE particles attached to the surface of the optimized phosphate coating can effectively prevent the penetration of chloride ions and water molecules to inhibit the electrochemical corrosion.

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