Experimental Multi-dimensional Study on Corrosion Resistance of Inorganic Phosphate Coatings on 17-4PH Stainless Steel

Shuaihui Guo [a*] and Zhangming Yan [a]

[a] North China University of Water Resources and Electric Power, Zhengzhou, Henan, China.

Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

Original Research Article

ABSTRACT

The multidimensional corrosion resistance of chromium-molybdenum heat-resistant alloy steel was studied by using TW-7 and AHP inorganic phosphate as coatings. TW-7, AHP inorganic phosphate coatings are both water-based high temperature resistant coatings, which are composed of a bottom coating and sealing coating.

The main component of the sealing paint is a mixture of water, chromate, and phosphate. The single dry hot air resistance, resistance to continuously sprayed synthetic sea water, and composite heat resistance/synthetic sea water salt spray conditions were tested. The corrosion resistance of TW-7 and AHP inorganic phosphate coatings was studied. The results of the dry/hot air test showed that the TW-7 coating on the AHP cannot endure long-term high temperature corrosion from small to large degrees at 600 °C. The synthetic seawater spray test showed that the TW-7 coating had

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*Corresponding author: Email: guo17838355546@163.com;

the least amount of corrosion. No obvious corrosion spreading or shedding of the two coatings was observed under the combined conditions of heat resistance and sea water salt spray. The results show that the cyclic resistance of the two coatings to heat-resistant/synthetic seawater salt spray is consistent.

1. INTRODUCTION

Inorganic phosphate is a kind of inorganic coating because the form of phosphorus in the solution is phosphate ion. It is widely used in various industries [1-5]. Because it has good heat resistance, corrosion resistance, oil resistance, high temperature resistance and other characteristics [6-7]. The coating can be used as anti-corrosion material in high humidity, wear-resistant material, and Refractory in high temperature for mechanical metal equipment. At present, inorganic phosphate coatings have been widely used in aerospace development, marine shipbuilding, automotive new energy industry and other fields and have achieved good results [8].

At present, most of the equipment explored in the marine field are mainly metal mechanical equipment, and these mechanical equipment will inevitably be corroded to different degrees when they work in the harsh marine environment for a long time, at least, it will cause the corrosion of equipment and the damage of machinery. In the long run, it will not only consume time and energy but also restrict the development of marine economy in our country [9-10]. Therefore, the protection of metal equipment is imminent, and the anti-corrosion of mechanical metal equipment has been a hot research topic at home and abroad [11-13]. The Corrosion of metal material is a kind of damage phenomenon caused by the chemical or electrochemical action when metal material is in contact with the surrounding medium [14]. As far as metals are concerned, they mostly exist in the form of metal compounds in nature. From the thermodynamic point of view, all metals except a few precious metals (such as gold and platinum) have a tendency to change into ions. Therefore, metallic elements have a higher free energy than their compounds, and there must be a tendency to spontaneously revert to their more thermodynamically stable natural state, oxides, therefore, the metal corrosion is a kind of phenomenon that exists spontaneously and universally, and it is inevitable [15-16]. For metals, one of the effective ways to improve their corrosion resistance is to treat their surface substrates. The coating protection of substrate surface is one of the most important and effective methods for surface treatment [17-18]. So far, the current corrosion protection methods for metals include coating protection, changing metal structure, electrochemical protection, surface treatment, dielectric treatment, corrosion inhibitor protection, temperature protection and maintenance [19-20]. These methods can be used alone or in combination to protect metal effectively, thus reducing the harm of corrosion to metal, but in practical application should be based on specific circumstances to select the most suitable method of protecting metal. Phosphate-based heat-resistant materials after a series of operations such as substrate surface treatment, coating and curing, corrosion-resistant, salt-resistant, heat-resistant and other related excellent properties of the coating, after a series of tests has proved to be an excellent turbine protective coating of the primary material [21-22]. Phosphate surface treatment works by using the chemical reaction of phosphate in an aqueous solution to adsorb it onto the surface of the material to form a phosphate film. The formation of phosphate film can improve the corrosion resistance and heat resistance of the material surface, thus enhancing its stability and service life under specific conditions. The mechanism of degradation of surface-treated phosphate materials under oxidation is mainly two aspects, namely, the oxidation reaction of phosphate materials under oxidation, the phosphate film on its surface is oxidized to phosphate oxide, releasing some of the phosphate and oxide ions [23]. The release of these ions can further promote the accelerated oxidation of phosphate materials, so that the surface of thermochemical reaction occurs. The surface treatment materials of phosphate with free radicals are affected by oxidation, and a lot of free radicals, such as hydroxyl radicals and oxygen radicals, are produced on the surface. These radicals react with phosphate and phosphate membranes to form new chemical bonds and oxidizing substances that accelerate

oxidation [24]. Therefore, this paper mainly aims at TW-7 inorganic phosphate coating, AHP inorganic phosphate coating related samples, develop test methods to study the corrosion resistance of coating, the applicability evaluation is carried out for simulating the service environment of high temperature, high humidity and high salt, which provides supplementary data for the production of turbine blades and the safety index and reliability of machinery and equipment. At the same time, the new research method which simulates the single and compound harsh environment provides a reliable basis for the evaluation of the corrosion resistance of inorganic phosphate coatings.

2. METHODOLOGY

2.1 Experimental Materials

The material used in the experiment was 17-4PH steel. 17-4PH stainless steel is a common low carbon steel, and its yield strength is usually between 725Mpa and 1180MPa, it has high strength and corrosion resistance, and good weldability and processability, widely used in construction, machinery, roads and bridges and other fields, its chemical composition includes carbon, silicon, manganese, sulfur, phosphorus and so on. The specific components are shown in Table 1.

TW -7 coating and AHP coating are made of a high-temperature resistant topcoat that consists of silicone, high temperature resistant pigments and fillers, additives and organic solvents.

2.1.1 Experiment setup

The main equipment used in this experiment is shown in the diagram, Fig. a is Muffle Furnace used for drying hot air test, Figs. b c d are oven, Salt Spray Chamber and Humid Heat Chamber used for synthetic sea water salt spray test and heat resistance/synthetic sea water salt spray cycle test.

Table 1. Composition of experimental materials (%)

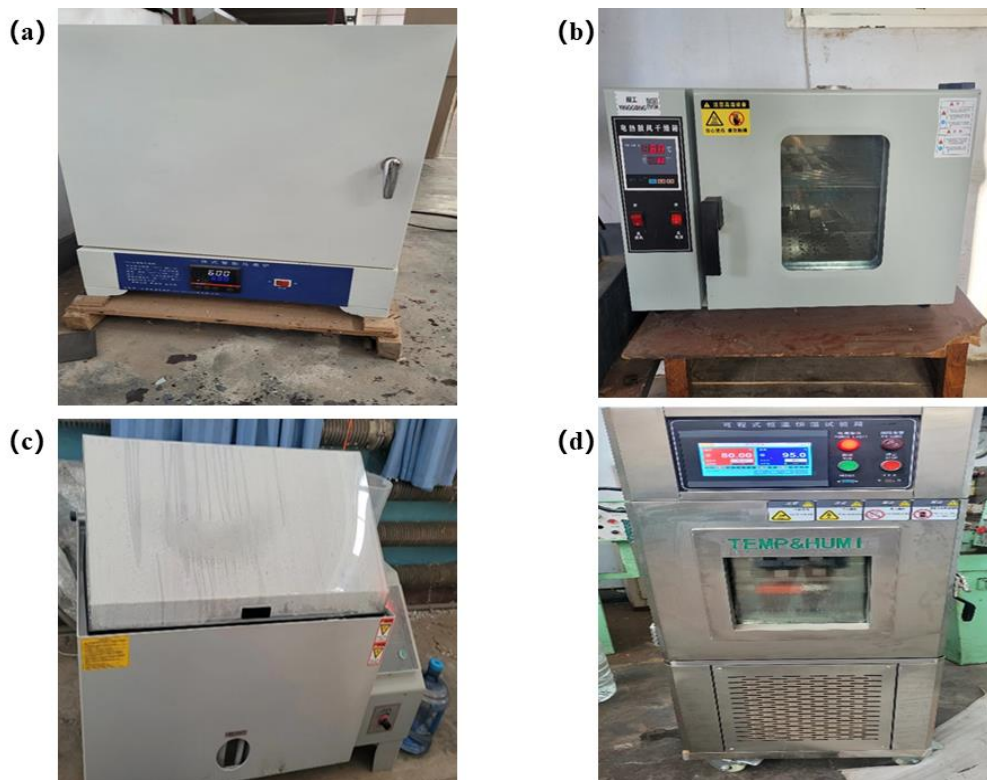| C	Si	Mn	S	P	Other
≤0.22	≤0.35	≤1.40	≤0.045	≤0.045	Fe

Image 1. Instruments used for the study

2.2 Method for Preparing Coating

According to the technical standard of inorganic phosphate coating, the process is made of 17-4PH stainless steel was prepared and exposed to ambient temperature of 20℃-40 ℃, and the relative humidity is controlled to 40% -70%. Detailed operation as follows:

(1) Sandblasting. The samples were sandblasted with a 40 mesh white corundum test forging sample, and then the Compressed air was used to clean the external dust of the substrate and grind the material at the same time. When sandblasting a flat surface, the angle should be 65° ~ 75° and the distance between the nozzle and the workpiece should be 250 ~ 280 mm. The process should keep the environment clean to avoid pollution.

(2) Bottom slurry agitation spraying. Use mechanical agitator was used to mix the slurry for three hours, filter with a 150 mesh filter to remove impurities, to ensure that the slurry was uniform without layering and precipitation and shelf life of the slurry. A total of two times, ensure that the full spray and each spray interval of 20 minutes, ensure that the specimen completely dry before spraying.

(3) The substrate is solidified and shot-peened. After spraying, the sample is cured in a heat treatment furnace. The performance parameters are: keep at 200 ℃ for 70 minutes, then heat to 380 ℃ for 70 minutes. Cool the air in the heat treatment furnace to room temperature. Using spray pressure of 0.4-0.5 Mpa, after curing in the bottom spray particles. Turn on the Compressed air and let go for 10-15 minutes. The surface for Compressed air, and shot peening should only be done without water.

(4) The coating is sprayed and cured. After holding temperature at 100 ℃ for 50 min, it was heated to 400 ℃ for 50 min and then cooled to room temperature in a heat treatment furnace. Repeat the curing process. A total of two times, ensure that the full spray and each spray interval of 20 minutes, ensure that the specimen completely dry before spraying. According to the ambient temperature of 20-℃40℃, its relative humidity of 40% -70%, 17-4PH stainless steel as a substrate in which exposure to the standard.

2.3 Accelerated Corrosion Test

Four samples were used for each experiment.

2.3.1 Test for resistance to dry hot air

The specimen was kept at 600 ℃ for 100h. It is then cooled to room temperature and the paint layer is tested. The specimen was kept at 600 °C for 1000 h. It was then cooled to room temperature and the paint layer was tested. Scanning electron microscope, energy spectroscopy and X-ray diffraction were used to observe and analyze the structure, thickness and composition of the coating.

2.3.2 Resistance to synthetic seawater spray

The experiment was carried out with 35 ± 2℃ synthetic seawater salt spray for 100 h and 1000 h, respectively. Observe whether the coating (cross-cut sample: cut place) is damaged or seriously corroded, the structure, thickness, composition and corrosion products of the coating were observed and analyzed by SEM, EDS and X-ray diffraction.

2.3.3 Heat resistant/synthetic seawater salt spray cycle

The specimens were subjected to ten cycles, each of which was heated at 450 ℃ for 2 hours in an air-circulating oven, cooled to room temperature, and then exposed to 35 ± 2 ℃ synthetic seawater for 20 hours. The damage of the film surface and the extent of corrosion spread were observed, and the structure, thickness, composition and microstructure of the corrosion products were tested and analyzed by SEM and EDS to determine the effect of the above exposure environment on the coating.

3. RESULTS AND DISCUSSION

3.1 Analysis of Physical and Chemical Properties of Coating

Figs. 1a and b shows the EDX spectra of TW-7 and AHP coatings, respectively. The energy spectrum showed that the chemical element in TW-7 coating were mainly O, C, Cr, P, Mg, and Al. According to the content of metal elements, the content of CR is higher than that of Al and Mg. It is inferred that the original composition of the coating is aluminum-chromium phosphate and Monomagnesium phosphate composite [25].

At a certain temperature, the coating is decomposed into P_2O_5, Al_2O_3, Cr_2O_3 and MgO composite coatings. The main chemical element in AHP coating are O, C, Cr, P, Mg and Al. According to the content of metal elements, the content of CR is higher than that of Al and Mg. It is inferred that the original composition is aluminum-chromium phosphate, phosphorus and magnesium dihydrogen phosphate composite coating [26]. At a certain temperature, the coating is decomposed into P_2O_5, Al_2O_3, Cr_2O_3 and MgO composite coatings.

(a)

Element	Wt%	At%
CK	05.63	11.30
OK	33.88	51.04
NaK	01.09	01.14
MgK	06.13	06.07
PK	18.30	14.24
CrK	34.97	16.21
Matrix	Correction	ZAF

(b)

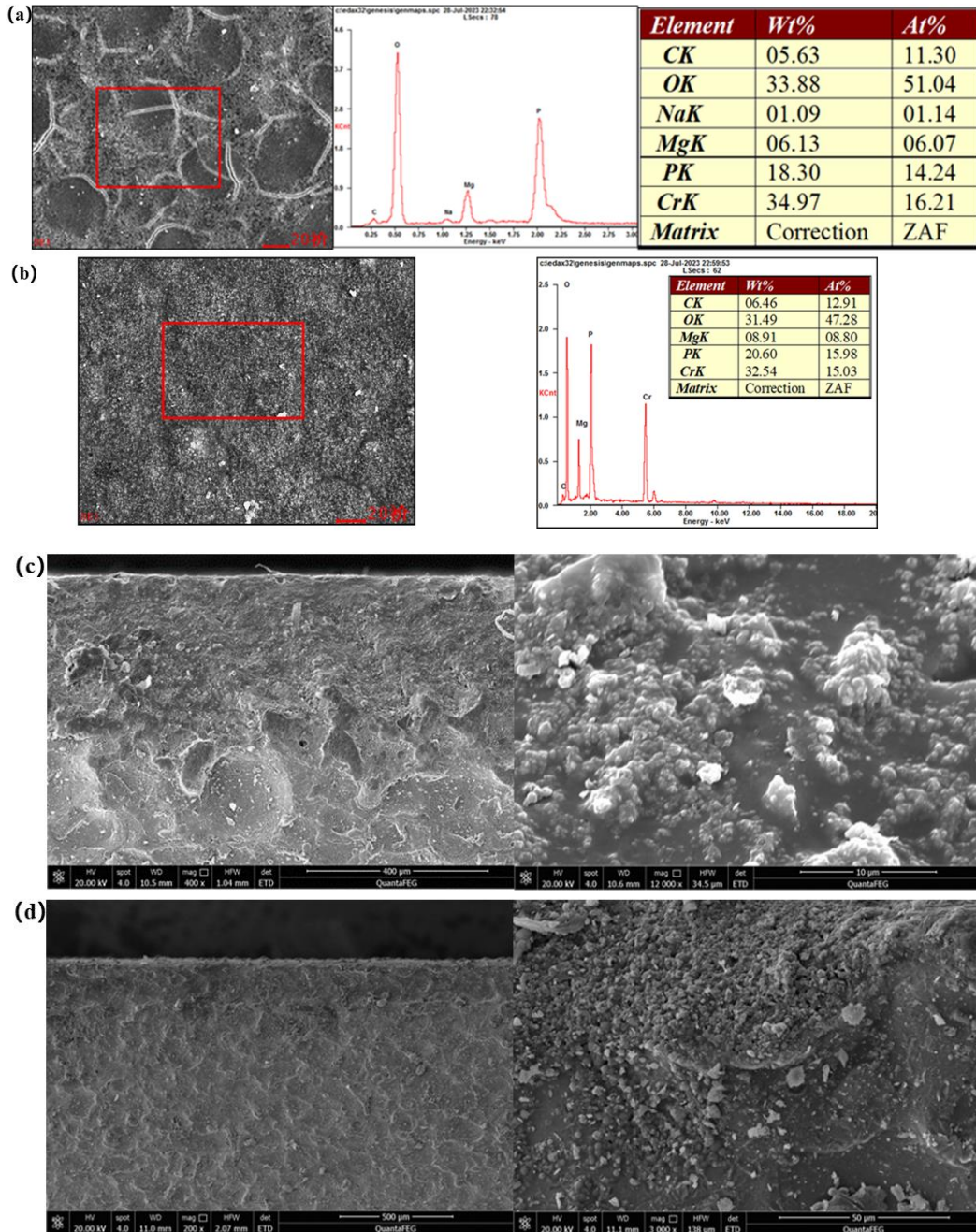| Element	Wt%	At%
CK	06.46	12.91
OK	31.49	47.28
MgK	08.91	08.80
PK	20.60	15.98
CrK	32.54	15.03
Matrix	Correction	ZAF

(c)

(d)

Fig. 1. (a) EDX spectrum of TW-7. (b) EDX spectrum of AHP. (c) Comparison of section morphology of TW-7 coating before and after corrosion （d） Comparison of section morphology of AHP coating before and after corrosion

3.2 Analysis of Dry Hot Air Resistance

The specimens were kept at 600 ℃ for 100h and 1000 h, respectively, and then cooled to room temperature. Fig. 2 a b after continuous heating in 600 ℃ dry air for 100 hours, TW -7 coating did not change significantly, only the edge of the visible slight burn marks. AHP coating ablation appears in the lower part of the sample. After continuous heating in 600 ℃ dry air for 1000 hours, a large area of oxidation appeared on the surface of TW-7 sample, and so did AHP sample, but the oxidation area was smaller than that of TW-7 sample, and the coating cracked, this indicates that TW-7 is best tolerated by short-time heating in 600℃ dry air, Next is AHP sample, with the heating time prolonged, TW -7 sample can still maintain a good tolerance, paint layer is still retained, non-oxidation discoloration. Fig. 2 c and d are TW-7. The surface of AHP sample after 100h and 1000h is compared with the original sample. It can be seen that the surface of TW-7 AHP sample before being heated by high temperature dry air is dense pits, but it is still relatively flat, after 100h high-temperature drying hot air, the surface of the samples have traces of oxidation and peeling off the coating [27]. As can be seen in the figure after 1000 hours of continuous heating, the surface of all samples appear more serious oxidation and coating shedding. This shows that the coating can not withstand long-lasting high temperature effect, high temperature corrosion [28]. Zhang [9] et al studied the high temperature corrosion resistance of 50CrVA coating, and found that the high temperature corrosion of 50CrVA coating had occurred after two hours at the temperature of 1050 ° C, the high temperature corrosion resistance of TW -7 and AHP coatings is more excellent.
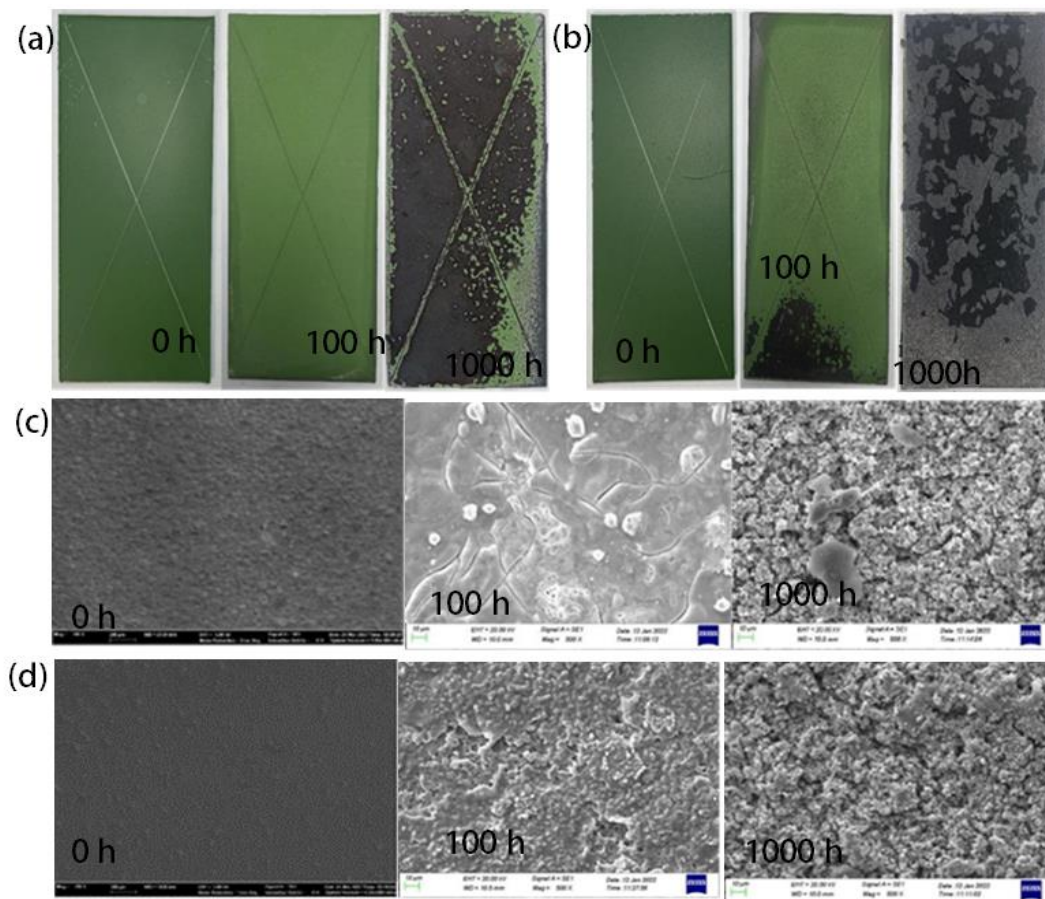

Fig. 2. (a) (b) TW -7, AHP heat-resistant dry air macro-contrast diagram. (c) Comparison of scanning electron microscopy (SEM) morphology of TW-7 specimen. (d) Comparison of SEM morphology of AHP sample surface

3.3 Experimental Analysis of Resistance to Continuous Spray of Synthetic Seawater

In 35 ± 2 ℃ synthetic seawater spray for 100 h and 1000 h, respectively, It can be seen from Fig. 3a that there are many speckled corrosion marks on the surface of TW -7 coating, and the coating does not show obvious rust wear [29]. From Fig. 3b can see there is no obvious corrosion mark on the surface of AHP coating, and there is corrosion mark on the edge [30]. Fig. 3c and d are the scanning electron microscopy (SEM) images of 17-4PH stainless steel samples coated with TW-7 after 100 hours and 1000 hours of sea water spray test were obtained [31]. Whether the spray lasts 100 hours or 1000 hours, the surface of the sample is coated with dissolved salt from seawater. When the synthetic seawater was sprayed for 100 hours, the cracks on the surface of the AHP coating sample expanded and the coating bulged, and when the synthetic seawater was sprayed for 1000 hours, there were more salt deposits on the surface of the sample and cracks on the coating [32]. There was no obvious corrosion on the surface of the samples after 1000h continuous spray of synthetic seawater. After 1000h continuous spray of synthetic seawater, the surfaces of the two samples were covered by a thick layer of sedimentary salt, there are yellow-brown rust marks on the edge of the samples [33].

3.4 Experimental Analysis of Heat-resistant/synthetic Seawater Salt Spray Cycle

The heat-resistant/synthetic sea water salt spray cycle is a cycle of Heating at 450 ℃ in an air cycle oven for 2 hours, air cooling to room temperature, and then exposure to 35 ± 2 ℃ synthetic sea water salt spray for 20 hours", 10 consecutive cycles. Fig. 4a b is TW-7, and the surface of AHP coating is macroscopically photographed before and after the test of heat resistance/synthetic seawater salt spray cycle. It can be seen that after the compound alternating action of high temperature and seawater salt spray, the surface of the sample appears more obvious corrosion marks, especially the edge part of the sample corrosion is more serious, the coating corrosion exposed the substrate, there is a distinct rust color on the base. The corrosion products were obviously distributed along the cross-cut of AHP coating. Fig. 4c d is a SEM image of the surface of TW-7 and AHP coating. After 10 cycles of heat-resistant/synthetic seawater salt spray cycles, the surface cracks of TW-7 coated samples deepened and shed, and more salt in seawater dissolved and deposited on the surface of samples. The crack on the surface of AHP coating deepens and falls off, and the coating bulges.
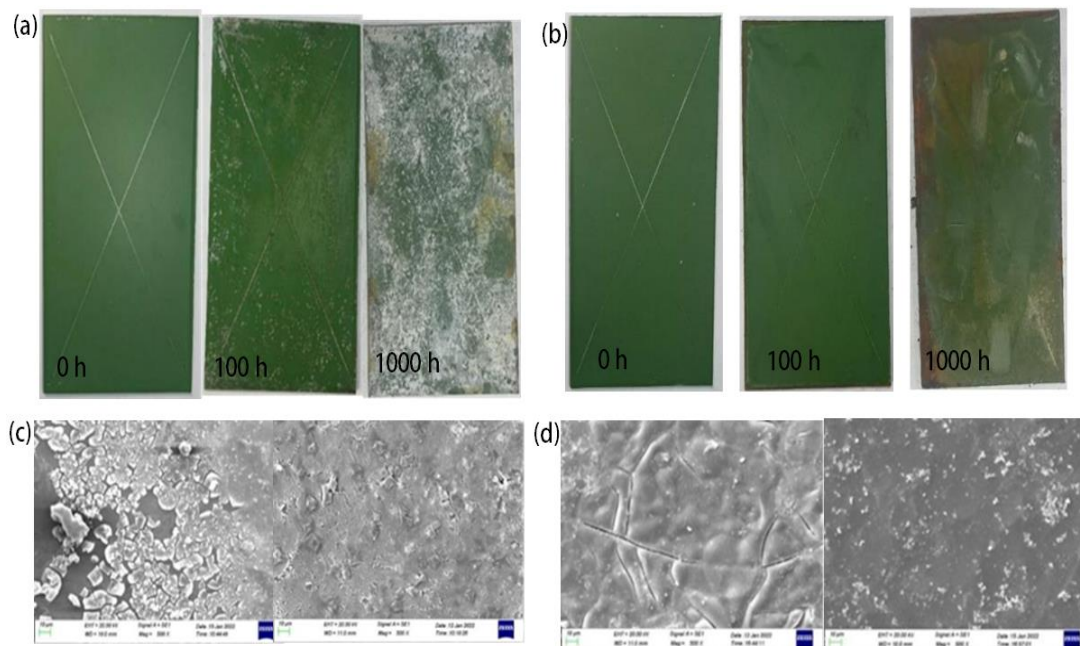

Fig. 3. (a) (b) Comparison of TW-7 AHP resistance to synthetic seawater continuous spray. (c) (d) (SEM) images of spray test of TW -7 AHP synthetic sea water (100h 1000h)
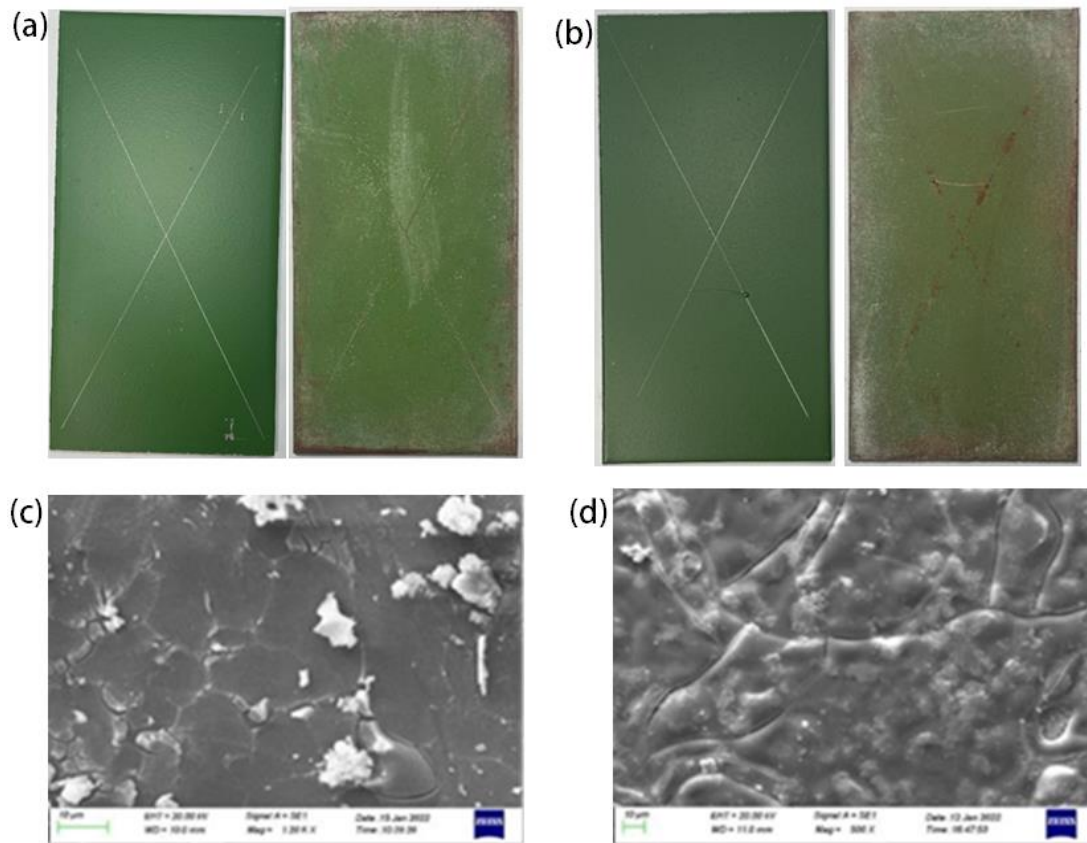
Fig. 4. (a) (b) TW-7, AHP coating by heat resistance/synthetic sea water salt spray cycle before and after the macro-photos. (c)(d) SEM photographs of the surface of TW -7 and AHP coatings

After the compound alternating action of high temperature and seawater salt spray, the corrosion marks on the surface of the sample appear obviously, especially on the edge of the sample, there is a distinct rust color on the base [34]. The corrosion resistance of TW-7 coating and AHP coating under high temperature and seawater salt spray cycling conditions is acceptable, and there is no obvious difference in surface state between the two coatings.

4. CONCLUSIONS

Inorganic phosphate-based composite coating is an important development direction of surface protective coatings, based on its unique properties such as radiation resistance, high temperature resistance, space environment and weather resistance, etc. people pay more and more attention to it, and it shows a very broad application prospect in the field of surface protection technology. However, with the progress of human science and technology and the exploration of unknown fields, especially the rapid development of modern aerospace, ships,

weapons and nuclear technology and other high-tech equipment, the related parts are all faced with extremely complex operating conditions (such as high and low temperature cycle, space environment, nuclear radiation environment, strong particle flow environment, multi-phase or special liquid medium environment, etc.) , it is a great challenge for the development and application of inorganic phosphate-based composite coatings, and it also brings more stringent application requirements for surface protective coatings. At present, the research and application of inorganic phosphate-based composite coating in surface protection field are still lack of a complete theoretical basis, all these problems need to be solved urgently. Therefore, it is necessary to focus on the key common scientific problems of coating composition, structure design, structure-activity relationship and multi-factor coupling, such as the multi-element, multi-phase, multi-scale interface effect and evolution law of coating materials, advanced design methods and preparation techniques, such as base resin modification, fine structure design and control of coating surface and

controllable preparation, functional filler synergy, etc. In this paper, TW-7 inorganic phosphate coating, AHP inorganic phosphate coating, dry hot air test, synthetic seawater continuous spray test were carried out, heat resistance/salt spray cycle test of synthetic seawater. The problems found in the research process are discussed in detail. At 600 ℃, after 1000 hours of hot air drying test, the coating has serious peeling off, most areas of the top coat and primer have peeling off, the exposed metal substrate has serious oxidation. This shows that the coating can not withstand long-lasting high temperature effect, high temperature corrosion. After 1000h continuous spray of synthetic seawater, the surface of the samples was covered by a thick layer of sedimentary salt, and the edges of the samples showed yellow-brown corrosion marks. After the compound alternating action of high temperature and seawater salt spray, the corrosion marks on the surface of the sample appear obviously, especially on the edge of the sample, there is a distinct rust color on the base. The study of corrosion and wear resistance of inorganic phosphate coatings can provide guidance and reference for the preparation and application of phosphate coatings in industry, is of great significance.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of manuscripts.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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