Experimental Studies of Heat Transfer and Corrosion Resistance of Ni-W-P Coating under Supercritical Water

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Abstract. As the post-processing method of equipment and tubes is seldom used in the field of super-critical water (SCW), as well as the high cost and low heat transfer performance of some of the materials, more up-to-date methods were developed to inhibit oxidation of SCW. In this work, the ternary Ni-W-P coating was deposited on the sample surface. Structure and composition of ternary Ni-W-P coating were profoundly analyzed with scanning electron microscopy (SEM), energy disperse spectroscopy (EDS) and X-ray diffraction (XRD). Additionally, thermal conductivity meter was specially employed to study the performance of heat transfer behaviour, by means of heat-resistance method. After oxidation of SCW, amorphous Ni-W-P coating crystallizes to some extent and oxidation resistance of coating can be convincing. Nevertheless, the diffusion of metal cations and the growth of oxides are the vital factors inhibiting the oxidation of SCW. Furthermore, the thermal conductivity of ternary Ni-W-P coating decreases with the increase of tungsten content and the coating unquestionably shows better heat transfer performance in the field of nuclear waste disposal and transport.

1. Introduction

The rapid growth in world population and increased living standard has triggered exploration of long-term energy solutions such as those provided by nuclear energy. To enhance these current nuclear reactor technologies, improvements in four main categories are considered: sustainability, safety and reliability, economics, proliferation resistance and physical protection [1]. Among them, water is widely used as an excellent solvent due to the character of cheap, non-toxic, non-flammable, non-explosive and environmentally friendly. Interphase mass transfer resistance can be eliminated and the reaction rate can be improved [2]. Therefore, supercritical water is an attractive new reaction medium which can replace non-eco-friendly solvents [3]. Due to the strong oxidation characteristics of supercritical water, it has very great requirements on piping materials. After the pipe used for transporting SCW was oxidized, on the one hand, the metal wall of the pipes will become thin and the circumferential stress of pipelines will increase. On the other hand, creep failure will occur in pipes owing to the low thermal conductivity of the oxide layer. When the oxide layer spalls, accumulative products will block the pipelines, resulting in the implosion of pipelines and the erosion of equipment. Hence, it is necessary to reduce oxidation products as it can enhance heat transfer and lower the operating temperature of metal. Consequently, the safety and service life of pipelines will be improved. There are many factors affecting the growth rate of the oxide layer. Two main methods can be employed. One is to change the materials and enhance its surface properties. The other one is to purify the water quality. It is of great scientific significance and academic value to study the influencing factors of the oxidation process and discuss the growth mechanism of the oxide layer.

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On the previous studies, Zr alloys [4,5], ferritic/martensitic steels [6,7], austenitic steels [1,8], nickel-based alloys [9,10,11] and oxide dispersion strengthened steels (ODS) [6,12] are commonly used as the main materials for supercritical heat exchanger. In industry, electroless coating has been widely used as a diffusion barrier in conventional electronic devices [13,14,15] and its heat transfer efficiency is acceptable due to its low thickness [16]. It has been reported that the Ni-P coating exhibits surprising reliability when stored at 423K for a long time [17]. Nevertheless, due to its low crystallization temperature and poor thermal stability, the combination of transition metals and hard composites (such as Al₂O₃ [18,19], TiO₂ [20,21], ZrO₂ [22,23], CeO₂-SiO₂ [24,25], etc.) with binary Ni-P coatings has been further investigated in recent decades. Previous studies reflect that the addition of tungsten can significantly delay the crystallization of binary nickel-phosphorus coating [26,27]. Meanwhile, the combination coating of composite coating and hard composite material in the high temperature range of 673-1173K has also been extensively studied to prove its suitability under high temperature [13,20,21,23,24,28,29].

2. Experimental section

2.1. Materials and preparation

In this work, the samples were made of mild steel in size of 15 mm × 15 mm × 5 mm. After being polished by sandpaper. They were soon put into an ultrasonic cleaner filled with distilled water. Before the corrosion, the samples were degreased with the miscible solution of 25 g/L NaOH, 20 g/L Na₂CO₃, 35 g/L Na₃PO₄ and 5 g/L alkylphenol polyoxyethylene. Subsequently, we coated samples with the ternary Ni-W-P coating (Table 1). The plating time goes 3 hours. When completing the process, the samples were cleaned in KQ3200BE ultrasonic cleaner again.

Table 1 Main components of electroless Ni-W-P plating solution

| Specimen No. | NiSO₄ (g/L) | Na₃WO₄ (g/L) | Na₃C₆H₅O₇·3H₂O (g/L) | C₃H₆O₃ (g/L) | NaH₂PO₂ (g/L) | NH₄AC (g/L) |
|-------------|-------------|-------------|---------------------|--------------|--------------|------------|
| 1           | 18          | 25          | 35                  | 15           | 20           | 12         |
| 2           | 18          | 30          | 35                  | 15           | 20           | 12         |
| 3           | 18          | 35          | 35                  | 15           | 20           | 12         |
| 4           | 18          | 40          | 35                  | 15           | 20           | 12         |
| 5           | 18          | 45          | 35                  | 15           | 20           | 12         |

2.2. SCWO corrosion experiment

Figure 1 shows the SCWO test rig, including peristaltic pump, pre-heater, reactor with electrical heater, cooler, valve, etc. The high temperature zone (red) covered with insulating layer is separated from the low temperature zone (blue). Samples were hung in the cap with nickel wire. Firstly, mixtures of the water and 30% hydrogen peroxide solution were pumped to the pre-heater for preheating to more than 573K. The flow velocity of peristaltic pump is 60ml/min. Then the heated solution flowed to the reactor and takes action with samples at 773-823K, 23-25MPa. After SCWO for 40 hours, the post-reacted steam was cooled then discharging to the container.
The mass of the samples was weighted and surface morphology of them after SCWO corrosion was observed by scanning electron microscopy (QuantaTM250, FEI, USA). Additionally, energy disperse spectroscopy (EDS) was further carried out to analyze the element distribution of the corrosion products. After that, crystal forms of Ni-W-P coating and the corroded samples were studied with the method of X-ray diffraction (XRD, D8 ADVANCE, Brueke, Germany). Furthermore, the fractal method was investigated to evaluate the SCWO corrosion resistance of Ni-W-P coating.

After the experiment, the surface morphology of samples was monitored by metalloscope and scanning electron microscopy (QuantaTM250, FEI, USA).

2.3. Heat transfer performance

In order to measure the thermal conductivity of ternary amorphous Ni-W-P coating during its use. The thermal conductivity of samples were tested by thermal conductivity meter (DRL-III, Xiangtan instrument Co Ltd, China) and changes of substrate with Ni-W-P coating were further analyzed. Firstly, the thermal conductivity of bare substrate was tested.

\[
\lambda_1 = \Phi_1 \cdot \delta_1 \left( \frac{1}{A_1} \right) \tag{1}
\]

Where \( t_1 \) and \( t_2 \) are the hot side temperature and cool side temperature of the substrate. \( A \) is the area of heat transfer. \( \Phi_1 \) is the heat exchange amount of substrate and \( \delta_1 \) is the thickness of substrate. The thermal conductivity \( \lambda_1 \) of the substrate is measured under the set temperature. As the thickness of ternary Ni-W-P coating is thinner than that of substrate. The steady state method is employed to calculate thermal conductivity of coating. The characterization of thermal physical parameters of micro-scale Ni-W-P coating will be further carried out in our subsequent research. Now, take the thermal conductivity of Ni-W-P coating into the equation (2).

\[
\Phi_2 = A_2 \left( t_1 - t_3 \right) \left( \frac{\delta_1}{\lambda_1} + \frac{\delta_2}{\lambda_2} \right)^{-1} \tag{2}
\]

We can obtain the thermal conductivity \( \lambda_2 \) of Ni-W-P coating from equation (2). Where \( t_3 \) is the cool side temperature of Ni-W-P coating. \( \Phi_2 \) is the heat exchange amount of substrate with Ni-W-P coating and \( \delta_2 \) is the thickness of Ni-W-P coating. Then, the change of it with the variety of ambient temperature, as well as differences of tungsten element content at constant temperature can be analyzed.

3. Results and discussion

3.1. Oxidation resistance performance

Surface morphology of Ni-W-P coating after being SCWO was observed by scanning electron microscopy. As shown in Figure 2. It is found that the surface of Ni-W-P coating is distributed with nickel cell lines. Based on our previous studies [26,30,31], this is because of the amorphous solid solution formed by solid solution of tungsten and phosphorus elements in the nickel crystal cell. See Figure 2a, we can notice that the surface of the coating is dense without obvious defects before SCWO. The grooves on the coating surface are caused by the deposition of Ni[W,P] on the surface of mild steel with micro-scratch. There are bulges, nickel furuncle, on the surface of the coating and they are mainly caused by the partial uneven deposition of phosphorus. Through cross-sectional scanning (Figure 2b), it was found that the color of the tissue change significantly. The nickel furuncle is filled with Ni[W,P] structure. This is due to the local inhomogeneity of the bare substrate surface, which leads to the over-deposition of Ni[W,P]. According to the aforementioned studies, ternary Ni-W-P coating is a typical amorphous structure. With the increase of tungsten content, the amorphous degree of the coating is higher. In order to avoid the effect of high temperature crystallization on the oxidation resistance of the coating, we specially prepared Ni-W-P coating with high tungsten content, which is a typical mixed crystal structure [26].
Figure 2 Structure morphology of the ternary Ni-W-P coating. (a) Surface morphology of the ternary Ni-W-P coating; (b) Cross-section of the ternary Ni-W-P coating.

Figure 3 Surface morphology of the ternary Ni-W-P coating with a metalloscope. (a) (b) (c) Ternary Ni-W-P coating after SCWO; (d) Ternary Ni-W-P coating before SCWO.

In Figure 3, we can notice that the black spots are nickel furuncle compared with the surface morphology of the non-corroded surface in Figure 3d. In addition, through the SEM results in Figure 2a, it can be known that the surface morphology photographed shows no discernible pores or pits as well. There is no distinct desquamation on the surface. The surface is still dense without pores. Instead, some nickel furuncle were distributed on the surface of the sample. The area of black spots on the surface broadens, as the oxides accumulate on the nickel furuncle, leading to the increase of irregular nickel furuncle. Owing to the formation of oxides, the silver color of the sample surface has changed. Instead, yellow, puce and black oxides were formed.

After further investigating the surface morphology of the oxidized coating, as shown in Figure 4, it can be noticeable that the original smooth nickel furuncle became irregular due to the oxidation of SCW, and the edge line of the nickel cell also degenerated because of the action of high temperature. The surface becomes more uneven and the bulges show more. This is due to that a large number of
oxidation products accumulate on the surface. See Figure 4b, the oxidation products are more likely to adhere to the surface of the nickel furuncle and the edge of the sample, resulting in a more uneven surface of the nickel furuncle. However, ternary Ni-W-P coating does not show obvious spalling and corrosion pits, and the protective effect of ternary Ni-W-P coating is not significantly damaged by the deposition of oxidation products on the surface of samples during oxidation. In Figure 4c, the tiny oxidation products are dispersed around the nickel cell. But unlike the oxides on the surface of the nickel furuncle, the oxides here are smaller and sparse. Meanwhile, it can be observed from Figure 4d that the number of nickel furuncle is more and they show more irregularity. Furthermore, there are long strips of nickel furuncles on the surface of the sample, which may be caused by the connection of multiple nickel furuncles due to the excessive growth of nickel furuncles. Oxidation of SCW and oxygen to the matrix layer is affected by the adhesion of oxidation products on the surface of nickel furuncle. It has a certain inhibitory effect on the oxidation of the SCW and oxygen.

Figure 4 Surface morphology of the ternary Ni-W-P coating after SCWO

As shown in Figure 5, there are a large number of oxygen elements on the sample surface, indicating that more oxides are produced. At the same time, it is obvious from Figure 5 that the content of oxygen element mounts with the increase of tungsten content in the coating. On the one hand, the amorphous coating has better SCWO resistance effect and generates fewer oxides. On the other hand, due to the generation of crystallization, the proportion of nickel content in the sample gradually decreases. Build on the previous studies [30,31,32], the higher the tungsten content is, the worse the stability of the coating is. Therefore, the nickel, tungsten and iron elements in the coating with high tungsten content will be easily oxidized by SCW producing corresponding oxides, which will then be subsequently deposited on the surface of the coating. The oxidation of nickel and tungsten can produce corresponding oxides and dense oxide films will be formed. The NiO oxide film is generally known as the rock salt structure, and WO$_3$ is mainly in the form of Monoclinic structure. Both of the structures are relatively stable and, under the strong oxidation of SCW, the matrix will be protected by them [33,34]. Additionally, the reaction rate of oxidation corrosion will be further inhibited [8]. In Figure 5, with the increase of tungsten content in the coating, the content of nickel and phosphorus decreased concomitantly. This can be easily understanding. The content of nickel and phosphorus decrease due to the rapid mount of oxygen content. The tungsten element is further released by the tungsten and phosphorus elements in the Ni[W,P] crystal cell, leading to the
accumulation of oxides on the surface of nickel furuncle. However, during this process, the content of phosphorus decreases. As it can be known, the cell body of the matrix show dense because of the phosphorus element. Due to function of phosphorus, nickel and phosphorus ions were co-deposited on the surface of the matrix and gradually grew. Without phosphorus, nickel would grow orderly in accordance with the structure of face-centered cubic (FCC) crystal. Phosphorus will occupy the positions of some nickel atoms forming Ni-P supersaturated solid solution. Under the supercritical condition, the vaporization of phosphorus at high temperature and the formation of NiP metallic compounds on the surface by crystallization will lead to a significant decrease in phosphorus.

![Figure 5 Elements change on surfaces of samples after SCWO](image1)

![Figure 6 Elements change on surfaces of samples after SCWO](image2)

See Figure 6, it can be known that the surface of the coating is relatively dense and the element distribution is uniform. Oxygen elements are also evenly distributed on the surface of the coating, so the oxidation process on the ternary Ni-W-P coating surface is relatively uniform and the oxides
deposit. In order to determine the distribution of oxides on the surface of nickel furuncles, EDS analysis of nickel furuncle was further carried out. As shown in Figure 7a, we can observe that content of phosphorus element is relatively low on the surface of nickel furuncle, which is due to the fact that the phosphorus element promotes the adhesion of nickel cells on the surface of the matrix. The presence of nickel furuncle is because of the uneven distribution of phosphorus during the deposition process and the adhesion of oxides on the surface of nickel furuncles leads to a further decrease of phosphorus element content on the surface of nickel furuncles. It can be seen that nickel and tungsten elements on the surface of nickel furuncles are relatively dense, and the surface is mainly covered by nickel, tungsten oxides and metal compounds because of crystallization. In Figure 7b, phosphorus element is similarly unevenly distributed on the surface of nickel furuncles. Moreover, due to the oxidation of SCW, the Ni[W,P] crystal cells are damaged, and nickel and tungsten oxides accumulate on the surface of the nickel furuncle, resulting in the irregularity of the nickel furuncle.

![Surface morphology and energy spectrum of nickel furuncle after SCWO](image)

In order to determine the surface composition of oxidation products, XRD analysis was conducted before and after SCWO. Coatings before SCWO show diffuse diffraction peaks at 45 degrees. The diffraction peaks are amorphous structure at low tungsten content, and the intensity of the diffraction peaks is small. The width of the half-peak of the diffraction peaks is large. At high tungsten content, the XRD diffraction peak intensity of the coating is larger and sharper, and the width of the half-peak decreases. The coating is in the form of a mixed crystal structure at this time. It can be known that the structure of the coatings changes from amorphous structure to a crystalline structure with the increase of tungsten content. Grain size of Ni-W-P coating was further studied based on Shelley's formula. It was found that the grain size of Ni-W-P coating samples before oxidation is 1.241 nm to 2.613 nm. Correspondingly, grain size of Ni-W-P coating samples increased with the rising of tungsten content in the sample.
After SCWO, in Figure 8, there are several relatively sharp crystal peaks in the original sample. Coupled with the overall diffraction curves, it was found that, besides the Ni (111) peak, there were obviously other diffraction peaks, which indicated that the coating crystallized under the action of SCWO, and the content of crystal phase increases. According to Scherrer’s formula, grain size now is 33.7 nm, which is much larger than that of coating before SCWO, but the grain size is still in the range of nanocrystals. Additionally, there are NiP and metal compounds generated besides Ni phase. This is due to the crystallization of the coating at high temperature, and the corresponding intermetallic compounds precipitate under the supercritical condition. This is also consistent with the results of previous studies on Ni-W-P coating [26,29]. Furthermore, there are tungsten oxide produced on the surface of coating.

3.2. Heat transfer behavior

When the ternary Ni-W-P coating is applied to the surface of supercritical heat exchanger, the thermal conductivity of material can be vital to the heat transfer performance. The overall heat transfers coefficient of the coated sample changes because of its thermal resistance. Therefore, we need to compare and analyze the thermal conductivity of coated samples with different plating parameters in the same period of time. At the same time, the thermal conductivity of ternary Ni-W-P coatings was measured to determine the relationship between thermal conductivity changing and tungsten content. Then the optimum coating parameters can be identified.

The thermal conductivity of the samples coated with ternary Ni-W-P coating was measured with a thermal conductivity meter (DRL-III, Xiangtan instrument Co Ltd, China). The thickness of ternary Ni-W-P coating is 20±5um. As shown in Figure 9a, it can be noticed that the thermal conductivity of the ternary Ni-W-P coating decrease with the increase of tungsten content. On the one hand, excessive sodium tungstate solution in the bath results in the more tungsten element depositing on the surface of the substrate. Thus more tungsten element co-deposit in amorphous Ni solid solution, as shown in...
Figure 9b. The phonon vibration is suppressed in the coating, and the average kinetic energy of the heat transport process of electrons as hot carriers reduce during thermal conduction [35,36,37]. Therefore, when more tungsten element deposit, the heat transfer process of the coating is inhibited.

See Figure 10, it shows the thermal conductivity of the coating changes with temperature and tungsten content. The thermal conductivity of the coating increases gradually with the mounts of the temperature of the heat source, which is also compatible the heat transport mechanism [35,38]. The average velocity of thermal carriers in amorphous Ni solid solution increases as the temperature increase. Since the collision process of energy exchange among hot carriers is intense and more frequent, which leads to the acceleration of the heat transfer process. Accordingly, the thermal conductivity of the coating increases. For those the tungsten content increasing in the coating, oxides under supercritical conditions will grow rapidly, resulting in more oxide deposition on the surface of the coating. Correspondingly, the surface of coating will be covered by more oxides and the coating will become thicker. Meantime, the thermal conductivity of ternary Ni-W-P coating decreases with the increase of tungsten content, which makes the heat transfer performance of the coating worse. To put it into a nutshell, those Ni-W-P coating with less tungsten can be more effective to the SCWO.

4. Conclusions
In this work, samples with Ni-W-P coating were prepared for the purpose of corrosion resistance of SCW and its heat conductivity was measured by means of heat-resistance method simultaneously. After oxidation of SCW, amorphous Ni-W-P coating crystallizes to some extent and oxidation resistance of coating can be convincing. Specifically, the diffusion of metal cations and the growth of oxides are the vital factors inhibiting the oxidation of SCW. Meanwhile, the thermal conductivity of ternary Ni-W-P coating decreases with the mounts of tungsten content and the coating unquestionably shows better heat transfer performance in the field of nuclear waste disposal and transport.

Acknowledgment
This work was supported by the National Natural Science Foundation of China (NO.51676205), Natural Science Foundation of Jiangsu Province for Youths (BK20170286), Xuzhou Science Foundation Project (KH17008), the Future Scientists Program of "Double First Rate" of China University of Mining and Technology (2019WLKXJ030) and a Project Funded by the Priority Academic Program Development of Jiangsu Higher Education Institutions.

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