The Enhancement Effect of Salt Bath Chromizing for P20 Steel

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Abstract: The TD (Thermal Diffusion) salt bath process is used to obtain a super hard carbide coating on the material surface by utilizing the mechanism of metal thermal diffusion. In this paper, chromium carbide coating was prepared on P20 hot-pressing die steel by the TD salt bath chromizing process. Characterization of the modified surface layer was made by means of scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDS), a micro-hardness tester and an automatic scratch tester. The influence rules of different salt bath times and temperatures on the growth thickness of the cladding layer were explored through experiments, and the optimum salt bath process scheme was determined as a temperature of 960 °C and time of 6 h. The chromium carbide coating with a thickness similar to that of chromium plating was prepared, and the average thickness of the coating was about 8–10 µm. The results showed that hardness and bonding strength of chromium carbide coating are higher than that of electroplated chromium coating. The combination of chromium carbide coating and matrix is metallurgical, while the electroplated chromium coating is physical. Immersion corrosion test results show that both coatings have good corrosion resistance in a 65% nitric acid solution.

Keywords: P20 steel; salt bath chromizing; chromium plating; microscopic analysis

1. Introduction

Salt bath chromizing is a kind of valuable chemical heat treatment technology that is widely used for steels to obtain the required properties [1–3]. It has some advantages over other chromizing technologies, such as a better surface quality, higher bonding strength and better wear resistance [4]. After salt bath chromizing, a compound layer is formed, which can significantly improve the performance of metals [5]. However, for obtaining the effective thickness of chromium carbide coating, a salt bath must be carried out at reasonable temperature and time [6].

Researchers have derived the growth kinetics equation of chromium carbide coating on high chromium steel surface by TD heat treatment, and proposed that the structure of carbide coating is several or one of Cr7C3, (Cr,Fe)7C3 and Cr23C6; however, there is a lack of research on the structure growth process of carbide coating [7]. Another researcher found that sub-micron CrN agglomeration area and CrN nano-crystal agglomeration area exist in the compound obtained by chromizing 20 steel in a low temperature composite salt bath for 12 h. There are a few defects in the CrN grain, but many (Cr,Fe)2(N,C)3 compounds with micro twins or stacking-fault are formed in the later stage of chromizing, which further supplement the structure of chromium carbide coating [8].

TD processing equipment is open type, without vacuum conditions. The equipment is generally an independently designed silicon carbide rod type high temperature resistance furnace, which only needs to provide a stable and controllable heat source, and can process many workpieces each time, with a high production efficiency.

Due to its excellent mechanical properties and excellent polishing properties, P20 steel is widely used in injection mold, which is often used to make the cavity and core of the
In the production of automobile friction plates, it is difficult to meet the needs for factory production to improve the surface properties of P20 die steel by the electroplating process, while the TD chromizing process has great advantages in improving the surface properties of the die.

When the hot-pressing die is used to process the automobile friction plate, there will be many failure forms on the die surface, such as the friction and wear of the die surface and the corrosion of the friction plate forming material. Secondly, the defects of the chromium electroplating process are obvious: the coating is easy to peel off, the adhesion phenomenon appears in the process of production and processing, as does the pollution problem of industrial electroplating. Therefore, a TD salt bath chromizing process was developed to solve the problem of insufficient surface performance of the mold and replace the existing polluting chromium plating process, in order to improve the service life of hot-pressing die and comply with the requirements of energy saving, environmental protection and industrial green development.

In order to improve chromizing efficiency, the optimum salt bath temperature and time for salt bath chromizing were explored and compared with chromium plating coating. Meanwhile, the structure growth process of chromium carbide was analyzed and the improvement of its matrix performance was discussed.

2. Experimental

2.1. Materials

P20 was selected as the matrix material with the chemical composition (wt.%) of: 0.38 C, 1.3 Mn, 1.85 Cr, Mo 0.4, 0.082 S and balance Fe. The specimens were machined into a size of 25 mm × 25 mm × 10 mm. Then the mechanically polished specimens were treated with emery papers of different granulometry to achieve a mirror finish. Finally, the specimens were ultrasonically cleaned in anhydrous ethanol and dried before salt bath chromizing treatment.

2.2. Experimental

Before the experiment, the specimens should be polished, activated and preheated. The specimens were polished with water mill to ensure that the surface of the specimens is smooth without scratches, and then were dried and stored after alcohol cleaning. Before the experiment, 5% nitric acid solution was used to clean the surface of the specimens until gray white appeared on the surface of the specimens, and then the residual liquid was removed in alcohol. The rust and oil stains of specimens were removed in 5% hydrochloric acid solution, and then the residual liquid was removed in alcohol and washed and dried. The process is activation treatment, the purpose is to improve the surface activity of the specimens and it is easier to prepare the coatings in the salt bath treatment. The temperature of salt bath treatment is generally above 910°C. Preheating treatment can prevent the surface cracks of specimens after rapid heating.

The main treating process was composed of three steps: salt bath chromizing, oil quenching and tempering. Firstly, the specimens were chromized in salt bath chromizing medium at different temperatures for various times, and then were quenched in oil. Finally, the specimens were tempered at 220 °C to reduce the stress concentration after quenching [9]. The formula of salt bath chromizing was 80% anhydrous borax, 10% Cr₂O₃ powder, 5% Al powder and 5% NaF activator. The effective temperature of TD salt bath chromizing coating is generally between 910 and 1050 °C [10]. In order to explore the effect of salt bath temperature and time on salt bath chromizing, two groups of experiments were designed and compared with chromium plating coating. The process parameters are given in Table 1.

In Group 1, three temperature gradients of 930, 960 and 980 °C were set, and the salt bath holding time was set as 5 h. In Group 2, the three groups of specimens were kept in the salt bath for 3, 4.5 and 6 h, respectively, and the holding temperature of the salt bath was set as 960 °C. The above specimens and the specimens with electroplating coating were
cut into 10 mm × 8 mm small specimens. After polishing the coating section, 4% nitric acid alcohol solution was used for etching for 5–10 s, and then the specimens were rinsed with ethanol and dried by cold air.

Table 1. Process parameters of salt bath chromizing.

| Experiment Group | Time/h  | Temperature/°C |
|------------------|--------|----------------|
| Group 1          | 5      | 930, 960, 980  |
| Group 2          | 3, 4.5, 6 | 960          |

2.3. Characterization of Modified Surface Layers

The cross-sectional microstructure was observed by optical microscopy and the thickness of coatings was measured by scanning electron microscopy (SEM). Five points at different positions on the coatings were selected for thickness measurement and the average value was taken. The element contents were determined by energy dispersive X-ray analysis (EDS). The equipment of SEM-EDS is the field emission scanning electron microscope with X-MAX 50 X-ray energy spectrometer produced by Zeiss, Jena, Germany, and the model is Zeiss ultra plus. Hardness measurements were made in a HX-1000TM micro-hardness tester (Shanghai optical instrument factory, Shanghai, China), with the test load of 2.94 N and holding duration of 15 s. Each hardness value was determined by averaging at least 5 measurements.

The corrosion resistance of the specimens was tested by corrosion test. In the process of forming a friction plate, the punch and die are corroded by the decomposition products of chlorine, fluorine and their compounds. In the actual working condition, it is found that the die surface has a pitting corrosion phenomenon after a long time of use. Therefore, it is necessary to explore the corrosion resistance of the two kinds of coatings. Due to the long period of salt spray test, the corrosion resistance of the two coatings cannot be obtained in a short time. In order to speed up the experimental progress and obtain reasonable experimental data, a full immersion corrosion test was used. Because hydrofluoric acid has poor stability, has a low boiling point and is difficult to control, the corrosion effect of hydrochloric acid is similar to that of nitric acid, so nitric acid solution was used as a corrosive agent. At room temperature, the full immersion corrosion test was carried out on the chromium plated, salt bath chromized and non-chromized specimens in 65% nitric acid. The corrosion time was 6, 12, 24, 36 and 48 h. By comparing the corrosion of three groups of samples, the difference of corrosion resistance of the three specimens was obtained. The mass of the specimens before and after corrosion was measured by AL204-1C electronic balance (METTLER TOLEDO, Zurich, Switzerland, accuracy: 0.1 mg) for five times and then averaged.

3. Results

3.1. Effect of Temperature on Chromizing in Salt Bath

The microstructure and uniformity of the coating were observed under SEM, as shown in Figure 1. Comparing Figure 1a–c, when the salt bath temperature is low, the coating is very thin and there are obvious pits on the surface of the coating. With the increase of salt bath temperature, the coating thickness increases and becomes more uniform under the condition of a certain treatment time. It can be seen from Figure 1d that the chromium plating coating is very thick and uniform, but there are pits and impurities in the coating, and there is an obvious boundary between the coating and the matrix material.

The thickness of coatings treated at 930, 960 and 980 °C was 5.3 ± 0.3, 8.6 ± 0.3 and 9.5 ± 0.3 µm, respectively, and the thickness of chromium plating coating was 11.02 ± 0.3 µm. It can be seen that when the temperature is low, the curve rises rapidly with the temperature, then slowly rises and finally tends to be stable. The activity of molten salt is related to temperature [11]. At low temperatures, the activity of chromium ion in molten salt is poor. In addition, part of chromium ion will settle, resulting in salt bath segregation,
and the growth rate of coatings will slow down due to the decrease of chromium ion concentration [12]. At low temperatures, the activity of carbon element in the matrix is also very low, and the interaction between the carbon element and chromium element is reduced, which seriously hinders the coating growth process [13]. At high temperatures, the fluidity of molten salt is enhanced, the activity of elements is improved and the coating thickness is increased. However, the higher the temperature, the smaller the increase of thickness, which indicates that the increase of treatment temperature cannot make the coating thickness increase infinitely. The main reason for this is that the temperature is too high, which will prevent the carbon element in the matrix and the chromium ion in the molten salt from continuing to combine, and which makes it more difficult to form a new coating.

![Figure 1](image_url)

**Figure 1.** The cross-sectional microstructure of specimens chromized for 5 h at different temperature and chromium electroplated. (a) 930 °C; (b) 960 °C; (c) 980 °C; (d) chromium electroplated.

### 3.2. Effect of Time on Chromizing in Salt Bath

After three groups of specimens and chromium plating specimens were treated as described in Section 3.1., the coating morphology was observed by SEM, and the results are shown in Figure 2. Comparing Figure 2a–c, the longer the salt bath time is, the greater the thickness of chromium carbide coating is, and the coating quality is also improved.

The thickness of coatings treated for 3, 4.5 and 6 h was 4.7 ± 0.3 μm, 7.9 ± 0.3 μm and 10.4 ± 0.3 μm, respectively. It can be seen that the coating thickness increases with the salt bath time, but the growth rate slows down and finally tends to be stable. At 960 °C, the activity of molten salt is fixed, and the growth rate of chromizing in salt bath is constant. The shorter the salt bath time is, the smaller the coating thickness is. When the salt bath time reaches 5 to 7 h, the chromium carbide coating is basically formed [14]. When the treatment time continues to increase, the coating thickness will not increase significantly. On the contrary, in the long-term high-temperature molten salt, the workpiece is easy to deform and the surface coating will peel off [15]. Moreover, if the treatment time is too long, the specimen core will overheat, the austenite in the matrix will be coarse and the mechanical properties of the coating will be reduced.
Figure 2. The cross-sectional microstructure of specimens chromized at 960 °C for different time and chromium electroplated. (a) 3 h; (b) 4.5 h; (c) 6 h; (d) chromium electroplated.

3.3. Compositional Analysis

The chromium carbide cladding specimen treated in a salt bath at 960 °C and 6 h were selected for comparison with a chromium plating specimen. Figure 3 shows the selected positions for point scanning of the two specimens. Figure 4 shows the energy spectra of selected points of the specimen with chromium carbide coating. The detected elements were normalized according to the energy spectra of each point of the two specimens. The elements of each point are shown in Tables 2 and 3 below.

Figure 3. Electron probe microanalysis of chromium carbide and chromium plating coatings. (a) Chromium carbide coating; (b) Chromium plating coating.

For the specimen chromium carbide coating, the closer to the coating, the higher the content of Cr element and the lower the content of Fe. In the middle of the coating, the content of Cr element decreases from 81.81% to 52.83%, and the content of Fe element increases from 18.91% to 45.92%. This indicates that the chromium carbide coating contains part of Fe element, the closer it is to the matrix, the higher the content of Fe element. When chromium carbide is formed, the Fe atom also diffuses into the coating. It can be inferred that the growth position of the coating exists both on the surface and inside of the matrix. When the metal donor and the carbon in the matrix form a carbide coating, the Fe in the matrix will also enter the coating to form a mixed layer of iron and carbide [16]. Because
the relative atomic radii of iron and chromium atoms are similar, they are easy to diffuse at high temperatures [17]. The diffused chromium ions enter the cementite in the matrix and form \((\text{Fe-Cr})_3\text{C}\). Moreover, the cementite content in the matrix has little change, but the overall content of C atom is constant. Therefore, the excess chromium ions diffuse into the cementite to form more \((\text{Fe-Cr})_3\text{C}\) compounds, and a large amount of Fe appears in the chromium carbide coating of P20 steel with general carbon content.

According to the theory of thermal diffusion, Cr diffuses from molten salt to P20 matrix, and the content of Cr element is a gentle gradient from surface to core, which indicates that a chromium carbide layer is formed at the junction of Cr and C. When the chromium carbide layer reaches a certain thickness and density, it hinders the continuous binding reaction between Cr and C [18]. It is proven that when the temperature and salt bath treatment time increase to a certain extent, the coating will not increase.

Figure 5 shows the section line scanning of two specimens. For the specimen with chromium carbide coating, the content of elements in the matrix does not change much, but it is very different at the junction of matrix and coating. In the linear direction from the surface of the matrix to the coating, the content of Cr increases rapidly, while the content of Fe decreases sharply until it disappears. The change of C element is not obvious. The change trend of elements is basically consistent with that of element content in point scanning. For the specimen with chromium plating coating, it is found that the content of elements at the interface between the matrix and the coating changes greatly. It is also
found that the content of elements at the interface between the matrix and the coating changes, but only Cr and Fe elements are found. It can be seen from Table 3 that only chromium element exists in chromium plating coating. This shows that chromium carbide coating is formed by a chemical reaction of chromium and a carbon atom, and chromium plating coating is a physical deposition.

![Figure 5. Section line scanning of two specimens. (a) Chromium carbide coating. (b) Chromium plating coating.](image)

3.4. Micro-Hardness Profile

Table 4 shows the hardness of chromium carbide specimens with different salt bath time and chromium plating specimen, the microhardness of chromium carbide coating is 1500–1600 HV2.94, which is much higher than 700 HV2.94 of chromium plating coating. Since the temperature change of salt bath treatment is equivalent to one-time quenching process, the hardness of matrix also increases [2]. The plating process is only chromium plating on the surface, which has no effect on the matrix, so the hardness of the matrix is close to that of the untreated specimen. According to the hardness test of the first three groups, with the increase of the coating thickness, the hardness of the specimen increases slightly, but the change is not significant, indicating that the coating thickness is basically independent of the surface hardness of the coating. The main reason for this is that the coating is complete and there is no obvious uneven thickness. In the same way, the surface hardness of the chromium carbide coating shown in Table 5 is basically stable under the salt bath treatment at different temperatures.
Table 4. Average hardness of chromium carbide specimens with different salt bath times and chromium plating specimens.

| Hardness (HV2.94) | Chromium Carbide Coating | Chromium Plating Coating | Matrix |
|-------------------|--------------------------|--------------------------|--------|
|                   | 3 h          | 4.5 h        | 6 h    |              |              |          |
| Coatings          | 1561.8 ± 0.5 | 1595.8 ± 0.5 | 1640.5 ± 0.5 | 690.8 ± 0.5 | 297.5 ± 0.5 |
| Matrix            | 556.3 ± 0.5  | 562.4 ± 0.5  | 584.1 ± 0.5  | 320.4 ± 0.5 | –            |

Table 5. Average hardness of chromium carbide specimens with different salt bath temperatures and chromium plating specimens.

| Hardness (HV2.94) | Chromium Carbide Coating | Chromium Plating Coating | Matrix |
|-------------------|--------------------------|--------------------------|--------|
|                   | 930 °C                   | 960 °C                   | 980 °C |              |              |          |
| Coatings          | 1552.6 ± 0.5             | 1619.2 ± 0.5             | 1640.5 ± 0.5 | 690.8 ± 0.5 | 297.5 ± 0.5 |
| Matrix            | 552.7 ± 0.5              | 573.8 ± 0.5              | 590.2 ± 0.5  | 320.4 ± 0.5 | –            |

3.5. Corrosivity Analysis

In this experiment, the corrosion rate was characterized by weighing the weight difference before and after corrosion, that is, the ratio of the mass change of metal before and after corrosion to the mass per unit surface area and time. It can be described by the following equation:

\[ v = \frac{m_1 - m_2}{s \cdot t}, \]

where \( v \) is the corrosion rate (g (m^2·h)^{-1}); \( m_1 \) and \( m_2 \) are the weight before and after corrosion (g); \( s \) is the surface area of the corrosion specimen (m^2); \( t \) is the corrosion time (h).

Table 6 shows the corrosion amount and corrosion rate of the three specimens. Figure 6 shows the relationship between the corrosion amount and time of three groups of specimens. It is obvious that the corrosion resistance of the specimens with chromium carbide coating or chromium plating coating is better than that of the untreated specimens. Both salt bath treatment and electroplating treatment can form a dense and corrosion-resistant film on the surface of the material, which can effectively prevent the matrix from contacting with corrosive objects such as acid or alkali, so as to improve the corrosion resistance of the specimens [19]. For the corrosion rate, the corrosion rate of untreated P20 steel is about 15 times of that of chromium plating coating and 18 times of that of chromium carbide coating. After 48 h, the corrosion amount of the two kinds of coatings increased, indicating that part of the coating was damaged, and some pitting corrosion was found on the surface of the specimen. In the defective and uneven coating of the specimen, corrosion was more likely to occur, and the corrosion depth would deepen with the increase of time.

Table 6. The corrosion amount and corrosion rate of the three specimens.

| Corrosion Time (h) | Corrosion Amount Per Unit Area of Three Groups of Specimens (mg/cm^2) | Corrosion Rate of Three Groups of Specimens (g(m^2·h)^{-1}) |
|--------------------|-------------------------------------------------------------------------|----------------------------------------------------------|
|                    | Untreated                  | Chromium Plating                          | Chromium Carbide                          | Untreated                  | Chromium Plating                          | Chromium Carbide                          |
| 6                  | 0.0732                    | 0.0138                                    | 0.0112                                    | 0.1220                    | 0.0023                                    | 0.0019                                    |
| 12                 | 0.2137                    | 0.0362                                    | 0.0312                                    | 0.1781                    | 0.0031                                    | 0.0026                                    |
| 24                 | 0.9874                    | 0.0618                                    | 0.0557                                    | 0.4114                    | 0.0258                                    | 0.0232                                    |
| 36                 | 2.1674                    | 0.1583                                    | 0.1149                                    | 0.4515                    | 0.0329                                    | 0.0239                                    |
| 48                 | 2.7538                    | 0.3867                                    | 0.2513                                    | 0.3671                    | 0.0537                                    | 0.0349                                    |
Figure 6. The relationship between the corrosion amount and time of three groups of specimens.

4. Conclusions

The temperature and time of salt baths have important influences on the growth of coating. The thickness of the coating increases with the increase of the treatment temperature and the treatment time, and the thickness of the coating does not obviously increase to a certain extent. The optimum process of salt bath heat treatment for P20 steel is 960 °C and 6 h. The hardness of chromium plating coating is less than 700 HV, and that of chromium carbide coating is 1500 to 1600 HV. The bonding mode of chromium carbide coating and substrate is matrix bonding, but the electroplating chromium coating is physical bonding. Therefore, the adhesion of chromium carbide coating is stronger than that of electroplating coating, so it is not easy to peel off in practical use, and the matrix is less likely to be corroded. The corrosion resistance of the specimens with chromium carbide coating or chromium plating coating is obviously better than that of untreated specimens. The corrosion resistance of the two coatings is basically the same in the early stage of the corrosion test, and the corrosion resistance of the chromium plating coating decreases with the corrosion time. In terms of the corrosion amount, the corrosion per unit area of untreated P20 steel is 15 times as much as that of electroplated specimen and more than 18 times that of Chromizing coating, which indicates that the coating can effectively protect the matrix and improve the corrosion resistance of the specimen.

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