Study on High Temperature Wear-Resistant Coatings on Surface of Special Steel by Orthogonal Test

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Abstract. In order to improve the friction and wear performance of steel under severe service conditions such as high temperature, epoxy resin E-51 resin is used as the base material, graphite, MoS₂ and other high temperature wear-resistant materials are used as pigments to prepare a kind of oil-coated high temperature resistant paint. The entropy weight method is used to comprehensively evaluate high temperature wear, thermal weight loss rate, and corrosion current density. The results show that the coating optimized by the entropy weight method has excellent performance. Among them, the high temperature wear rate of the paint film is 0.83%, the thermal weight loss rate at 200 °C is 4.56%, the thermal weight loss rate at 400 °C is 27.84%, and the corrosion current density is 9.31E-9. The alkali resistance of the paint film is far greater than the national standard.

1. Introduction
In industrial production, it is necessary to apply an organic coating on the steel surface to protect the steel from corrosion[1-3]. Yet, the organic coatings on the market cannot be used at high temperatures. At the same time, most coatings require complicated degreasing work before painting. This also increases the process flow and reduces efficiency. Therefore, it is priority to design a coating that has a good protective effect at high temperatures can be applied on oil-rich surfaces[4-7].

Orthogonal experimental design is a highly efficient experimental method. However, this method cannot perform multi-response analysis and cannot comprehensively evaluate the coating formulation. Entropy weight method is a comprehensive evaluation method based on orthogonal experiment combined with subjective scoring method. This method can comprehensively evaluate more than three response experiments and can optimize the overall performance of the formulation.

This article mainly studies a kind of coating suitable for the surface of special steel with oil. The coating has good comprehensive performance under high temperature conditions. Additionally, this article applies entropy weight method to the paint industry, which has certain scientific value.

2. Experiment

2.1 Materials and Equipment
The materials used in this article are: epoxy E-51 resin, water-based amine curing agent, graphite, MoS₂, glass flake, gas phase SiO₂, quartz sand (SiO₂), n-butanol, xylene, dimethyl silicone oil.
The instruments used in this article are: electro-hydraulic servo friction and wear testing machine (Jinan Shunmao Test Instrument Co., Ltd.), box-type resistance furnace, electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd. CHI660E), Fourier exchange infrared spectrometer (Thermo Scientific Nicoleti S50).

2.2 Preparation

In this paper, a round sample with a diameter of 5 cm and a square sample with a diameter of 50 mm×70 mm are used. After grinding, degreasing and derusting treatment, apply 0.15 g of dimethyl silicone oil on the round sample and 0.3 g of dimethyl silicone oil on the square sample. Try to apply evenly. After the oil is applied, the sample is placed in the air for more than 2 h to allow the surface to fully absorb the dimethyl silicone oil. Wipe off the excess dimethyl silicone oil on the surface of the sample with absorbent cotton before use, leaving a thin film of oil on the surface[8].

The preparation of the coating is divided into the following steps:

1. Configure the mixed solvent according to the recipe amount.
2. Configure curing agent solution, in which the mass ratio of aqueous amine curing agent to mixed solvent is 4:1
3. Dissolve all epoxy E51 resin with the remaining mixed solvent.
4. Grind the graphite, MoS2, glass flake, quartz sand (SiO2) and gas phase SiO2 together, and put them into the beaker.
5. Pour part of the resin solution into the beaker of step 4, stir with a glass rod to completely wet the filler, then add all the remaining resin solution, dilute, and add a stirrer to disperse.
6. After dispersing for a period of time, add the curing agent solution and continue to disperse until the dispersion is uniform, then roll coat.

2.3 Performance Evaluation

Basic performance of coating: GB/T 1728-1979(1989), GB/T 9286-1998, GB/T 9274-1988.

High temperature thinning of coating: Install the sample in the device, set the test temperature to 200 ℃, Erase the coating with hot air for 1 minute. Test the coating film thickness before and after the experiment, and calculate the coating loss before and after the experiment.

High temperature resistance: According to the requirements of GB/T 1735—2009. Put the cured paint film into the box-type resistance furnace. Warm to the required temperature and keep for 2h. Take it out after cooling to 25 ℃. The high temperature resistance of the paint film was evaluated by the thermal weight loss of the paint film.

Corrosion resistance: According to the requirements of GB/T 9274-1988. Use 5% H2SO4 aqueous solution to measure the acid resistance of the paint film. Use 5% H2SO4 aqueous solution to measure the acid resistance of the paint film. Use 5 % NaOH aqueous solution to measure the alkali resistance of the paint film. Use 3.5 % NaCl aqueous solution to measure the salt resistance of the paint film. The polarization curve of the coating was tested using an electrochemical workstation. The corrosion medium was 3.5% aqueous sodium chloride solution.

3 Results and Discussion

3.1 Orthogonal Experimental Scheme Design

Based on single factor experiment. PVC (A), graphite dosage (B), MoS2 dosage (C), and glass flake dosage (D) were selected as the four factors in the orthogonal experiment. Carry out a four-factor three-level test.

The design of orthogonal experimental scheme is shown in Table 1.
Table 1. Orthogonal test factor level table.

| Level | Factor |
|-------|--------|
|       | A  | B/% | C/% | D/% |
| 1     | 1:1 | 25  | 16  | 14  |
| 2     | 1:2:1 | 35  | 18  | 18  |
| 3     | 1:4:1 | 45  | 20  | 22  |

The high temperature wear, thermal weight loss rate and corrosion current density of the coating are used as the evaluation criteria. The test scheme and results are shown in Table 2.

Table 2. Orthogonal test results.

| Test | A  | B  | C  | D  | thinning of Coating/% | TG/% (250℃) | Icorr/A cm⁻² |
|------|----|----|----|----|-----------------------|-------------|-------------|
| 1    | 1  | 1  | 1  | 1  | 5.17                  | 12.21       | 6.65E⁻⁸     |
| 2    | 1  | 2  | 2  | 2  | 15.22                 | 14.45       | 3.21E⁻⁸     |
| 3    | 1  | 3  | 3  | 3  | 2.00                  | 10.19       | 9.97E⁻⁸     |
| 4    | 2  | 1  | 2  | 3  | 16.14                 | 9.43        | 4.71E⁻⁸     |
| 5    | 2  | 2  | 3  | 1  | 4.13                  | 8.46        | 2.62E⁻⁸     |
| 6    | 2  | 3  | 1  | 2  | 9.28                  | 5.94        | 1.02E⁻⁷     |
| 7    | 3  | 1  | 3  | 2  | 3.35                  | 11.53       | 3.55E⁻⁸     |
| 8    | 3  | 2  | 1  | 3  | 7.45                  | 12.01       | 4.55E⁻⁸     |
| 9    | 3  | 3  | 2  | 1  | 13.63                 | 6.71        | 1.25E⁻⁸     |

The evaluation results of other properties of the paint film are shown in Table 3.

Table 3. Other performance evaluation results.

| Test items     | 1   | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9   |
|----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Film thickness/μm | 68  | 74  | 69  | 77  | 58  | 61  | 88  | 70  | 61  |
| Dry time /min    | 47  | 52  | 55  | 43  | 53  | 28  | 27  | 30  | 33  |
| Adhesion /level  | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   | 0   |
| Acid resistance(5%H₂SO₄)/d | 15  | 15  | 15  | 15  | 15  | 15  | 15  | 15  | 15  |
| Alkali resistance(5%NaOH)/d | 15  | 15  | 15  | 15  | 15  | 15  | 15  | 15  | 15  |
| Salt water resistant(3.5%NaCl)/d | 30  | 30  | 30  | 30  | 30  | 30  | 30  | 30  | 30  |
| High temperature resistance/℃ | 300 | 300 | 300 | 300 | 300 | 300 | 300 | 300 | 350 |

It can be seen from Table 3 that as PVC increases, the surface drying time of the paint film becomes shorter. The coating adhesion, alkali resistance and wear resistance at normal temperature of the 9 groups of experiments are all excellent.

3.2 Comprehensive Weighting Method

Based on the above orthogonal experiment results, the fuzzy evaluation matrix \(X=(x_{ij})_{n \times m}\) of the plan set versus the index set is obtained. Among them, \(n\) is the number of designed experimental schemes, \(m\) is the three indicators of high temperature wear, TG and Icorr, that is \(n=9, m=3\).

\[
X = \begin{bmatrix}
5.17 & 12.21 & 0.66 \\
15.22 & 14.45 & 0.32 \\
2.00 & 10.19 & 1.00 \\
16.14 & 9.43 & 0.47 \\
4.13 & 8.46 & 0.26 \\
9.28 & 5.94 & 1.02 \\
3.35 & 11.53 & 0.36 \\
7.45 & 12.01 & 0.46 \\
13.63 & 6.71 & 0.12 \\
\end{bmatrix}
\]

Due to the incommensurability of each index, the matrix cannot be processed. Therefore, it is necessary to normalize the fuzzy matrix \(X\) to obtain a normalized matrix \(Z=(z_{ij})_{n \times m}\). Since the three evaluation criteria have the characteristics of being as small as possible, the normalized formula is:
According to the working conditions and storage methods of the designed coatings, by inquiring and surveying relevant experts and workers, we designed the subjective weights of the indicators and obtained the subjective weight vector \( \alpha = [0.5, 0.3, 0.2]^T \).

The objective weights of various indicators are obtained from the formulas:

\[
Z_{ij} = \frac{(x_j)_{\text{max}} - x_{ij}}{(x_j)_{\text{min}} - (x_j)_{\text{min}}} \\
Z = \begin{bmatrix}
0.78 & 0.26 & 0.4 \\
0.06 & 0 & 0.78 \\
1 & 0.50 & 0.02 \\
0 & 0.59 & 0.61 \\
0.85 & 0.70 & 0.84 \\
0.48 & 1 & 0 \\
0.90 & 0.34 & 0.73 \\
0.61 & 0.29 & 0.62 \\
0.18 & 0.91 & 1
\end{bmatrix}
\]

Finally, the overall weight is obtained from formula (2).

\[
\min F(w) = \sum_{i=1}^{n} \sum_{j=1}^{m} \left[ \mu (w_j - \alpha_j)z_{ij} \right]^2 + (1 - \mu) \left[ (w_j - \beta_j)z_{ij} \right]^2 \\
\text{s.t.} \left\{ \begin{array}{l}
\sum_{j=1}^{m} w_j = 1 \\
w_j \geq 0, j = 1...m
\end{array} \right.
\]

In the formula, \( \mu \) is the preference coefficient, take \( \mu = 0.5 \) to get the comprehensive weight vector \( w = [0.415, 0.318, 0.266]^T \).

From the weighting formula \( f_i = \sum_{j=1}^{m} z_{ij} w_j \), the comprehensive score vector of the final indicators is \( f = [0.513, 0.232, 0.579, 0.350, 0.799, 0.517, 0.676, 0.510, 0.630]^T \).

The range analysis based on the comprehensive weighting method under multiple indicators is shown in Table 4.

| A   | B   | C   | D   |
|-----|-----|-----|-----|
| \( K_1 \) | 1.32 | 1.54 | 1.54 | 1.94 |
| \( K_2 \) | 1.66 | 1.54 | 1.21 | 1.42 |
| \( K_3 \) | 1.82 | 1.73 | 2.05 | 1.44 |
| \( k_1 \) | 0.44 | 0.51 | 0.51 | 0.65 |
| \( k_2 \) | 0.56 | 0.51 | 0.40 | 0.48 |
| \( k_3 \) | 0.61 | 0.58 | 0.68 | 0.48 |
| \( R \) | 0.16 | 0.06 | 0.28 | 0.17 |

Factor(main→secondary) Optimization: CDAB

C3D1A3B3

It can be seen from Table 4 that the amount of MoS2 has the greatest influence on the comprehensive weighted score. The ranking of the influence of various factors on the comprehensive weighted score is: MoS2 dosage > Glass flake dosage > PVC > Graphite dosage. It is concluded that the optimal combination of factor levels for each factor is C3D1A3B3. That is, PVC is 1.4: 1, graphite ratio is 45%, MoS2 ratio is 20%, and glass flake ratio is 14%.

3.3 Experimental Results

Orthogonal optimization scheme experimental results are shown in Table 5.
Table 5. Orthogonal optimization scheme experimental results.

| Test items | Test results |
|------------|--------------|
| Film thickness /μm | 81.3 |
| Dry time /min | 31 |
| Hard time /h | 12 |
| (Before sintering) Adhesion /level | 0 |
| (After sintering) Adhesion /level | 0 |
| (before sintering) Acid resistance(5%H2SO4) /d | 15 |
| (after sintering) Acid resistance(5%H2SO4) /d | 15 |
| (before sintering) Alkali resistance(5%NaOH) /d | 30 |
| (after sintering) Alkali resistance(5%NaOH) /d | 30 |
| (before sintering) Salt water resistant(3.5%NaCl) /d | 15 |
| (after sintering) Salt water resistant(3.5%NaCl) /d | 15 |
| $I_{corr} /A \cdot cm^2$ | 9.31E-9 |
| High temperature resistance/℃ | 400 |
| 200℃ thermal weight loss rate % | 4.56 |
| 250℃ thermal weight loss rate % | 8.81 |
| 300℃ thermal weight loss rate % | 11.62 |
| 350℃ thermal weight loss rate % | 23.78 |
| 400℃ thermal weight loss rate % | 27.84 |
| 450℃ thermal weight loss rate % | 36.18 |
| High temperature wear % | 0.83 |

It can be seen from Table 5 that under the optimal combination conditions, the high temperature wear rate of the paint film is only 0.83%, the thermal weight loss rate at 200 ℃ is only 4.56%, the thermal weight loss rate at 400 ℃ is 27.84%, and the corrosion current density is only 9.31E-9. The other properties of the paint film meet national standards.

4. Conclusion
(1) The range analysis shows that the amount of MoS2 has the most obvious effect on high temperature wear performance. PVC has the greatest influence on the thermal weight loss rate of the paint film. The amount of graphite has the greatest influence on the corrosion current density.
(2) Through the analysis of the orthogonal weighting method, the optimized formulation of the coating is 1.4: 1 for PVC, 45% for expandable graphite, 20% for MoS2 and 14% for glass flake. Under optimal conditions, the high temperature thinning of coating of the paint film is only 0.83%, the thermal weight loss rate at 200 ℃ is 4.56%, the thermal weight loss rate at 400 ℃ is 27.84%, and the corrosion current density is 9.31E-9. Other properties are in line with industry standards.

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References
[1] DOUCETTE J J J, FRANCHITTO A R. Three dimensionally reinforced ablative/insulative composite: U.S.,5985405[P]. 1999-11-16
[2] TANAKA R. Research and development of ultra-high temperature materials in Japan[J]. Materials at high temperatures, 2000, 17(4): 457—464.
[3] GIAVERI S, GRONCHI P, BARZONI A. IPN polysiloxane-epoxy resin for high temperature coatings: structure effects on layer performance after 450 ℃ treatment[J]. Coatings, 2017, 7(12): 213.
[4] LIAN Zhao-hua, ZHANG Qiang, HUO Sheng. Performance test for coating of low surface treatment [J]. Ship & ocean engineering, 2019, 48 (02): 135-137 + 141.
[5] LIU Wei-min, XU Jun, FENG Da-peng, et al. The research status and prospect of synthetic lubricating oils [J]. Tribology, 2013, 33(01):91-104.

[6] BAI Jing-jing. Study on heat resistant anti-wear nano-composite coating [D]. Dalian university of technology, 2012.

[7] WU Lin-lin. Study on surface temperature field and its similarity of aircraft [D]. Harbin institute of technology, 2018.

[8] WU Lin-lin. Study on surface temperature field and its similarity of aircraft [D]. Harbin institute of technology, 2018.

[9] Apostolos Krallis, Dimitris Meimaroglou, Vassilis Saliakas, et al. Dynamic optimization of molecular weight distribution using orthogonal collocation on finite elements and fixed pivot methods: An experimental and theoretical investigation [J]. Computer aided chemical engineering, 2006, 21.

[10] WANG Li-hua, ZHANG Ying-zhi, SHEN Gui-xiang, et al. Optimization of machining parameters of wedm based on weighted mean method [J]. Machine tool & hydraulics, 2014, 42(17): 81-83.

[11] LI Feng, CHU Man-sheng, TANG Wei, et al. Optimization of high chromium vanadium-titanium magnetite electric melting process by comprehensive weighted scoring method [J]. Journal of northeastern university, 2016, 37(09):1327-1331.

[12] TAO Ju-chun, WU Jian-min. New study on determining the weight of index in synthetic weighted mark method [J]. Systems engineering-theory & practice, 2001(08):43-48.