Preparation and properties of PTFE anti-friction coating

Polytetrafluoroethylene (PTFE) is considered the best solid lubricant, its friction coefficient is only 0.05 within 320 ℃ temperature, even smaller than the graphite friction factor 0.07. PTFE because its good lubricity and chemical stability, is widely used as industrial lubricants. Perfluoro surfactant is a type of fluorine-containing surfactant. Compared with the traditional surfactants, fluorosurfactants have high surface activity, high heat stability, high chemical stability, low surface tension, low use concentration and other excellent features. Most importantly, the fluorine-containing surfactant can be used as a dispersant, and the fluorine-containing surfactant can be used as a dispersant in the dispersion polymerization of various fluororesins.

Bonded solid lubricating film is organic and inorganic binder as the base material, the small friction coefficient solid as a solid lubricant, together with solvents and surfactants made of composite lubricating paint, by dip or spray to form coating.In this paper, epoxy resin as the base material, by adding polytetrafluoroethylene and perfluorinated surfactants (FC-400) made of self-lubricating friction-reducing coating. According to orthogonal experimental design to get different formulations of paint, and then coated on the polished iron which has been processed, at room temperature curing, get friction coating. After a series of tests, the best formula was screened out. The physical and chemical properties of the coatings were measured by a SpectrumOne infrared spectrometer, an MM-200 friction and wear tester, a Y-4Q X-ray diffractometer, a P / N2500052 SERIAL contact angle gage and a differential thermal analyzer.

Experimental method

The substrate used in the experiment is A4 steel sheet with size 20mm*20mm. The substrate was polished by model 200 #, 600 #, 800 #, 1500 # water-resistant sandpaper to remove the substrate surface oxide and impurities, with 15% hydrochloric acid and 15% sulfuric acid mixed solution pickling 2 min, with acetone and ethanol mixed solution in the beaker ultrasonic cleaning 10 min to remove the sample surface of organic matter, and then activated with 3% hydrochloric acid. According to orthogonal experiment design, select the amount of epoxy resin and curing agent are 3 g, the other reagents were tested according to a 4 factor 4 level orthogonal experiment table(see Table 1).

Table 1 4 factors 4 level orthogonal experiment table

Level	Acetone A	PTFE B	Graphite powder C	FC-400 D
1	5 mL	0.9 g	0	0.009 g
2	6 mL	1.2 g	0.1 g	0.012 g
3	7 mL	1.5 g	0.2 g	0.015 g
4	8 mL	1.8 g	0.3 g	0.018 g

According to the different formulations the orthogonal table, the weighed polytetrafluoroethylene powder and the graphite powder were mixed and shaken for 3 min in an ultrasonic oscillator, so that the polytetrafluoroethylene powder and the graphite powder are mixed uniformly. Take 4 mL of acetone, dissolve the dispersant FC-400, mix well, pay attention to slow stirring, to avoid excessive air bubbles. The mixed PTFE and graphite powder dissolved in acetone to dissolve the dispersant, the rapid mixing evenly. Pour the mixed mixture into a beaker with epoxy and EP curing agent, and mix well. The surface treatment of iron into the coating in the infiltration of 5 ~ 10 s, with the centrifuge to shake the sample, select the appropriate film thickness, that is the sample.

Results and discussion

Tribological properties

The friction coefficient of the coating was measured with a MM-200 friction and wear tester manufactured in Japan. The test was carried out at a load of 10 N at a speed of 180 r / min, the results of orthogonal experimental analysis are shown in Table 2.

Table 2 Friction factor analysis orthogonal table

试　验	A	B	C	D	摩擦因数
1	1	1	1	1	0.144
2	1	2	2	2	0.055
3	1	3	3	3	0.122
4	1	4	4	4	0.054
5	2	1	3	4	0.039
6	2	2	4	3	0.137
7	2	3	1	2	0.12
8	2	4	2	1	0.185
9	3	1	4	2	0.041
10	3	2	3	1	0.103
11	3	3	2	4	0.097
12	3	4	1	3	0.048
13	4	1	2	3	0.051
14	4	2	1	4	0.095
15	4	3	4	1	0.137
16	4	4	3	2	0.068
K_1	K_1^A=0.375	K_1^B=0.275	K_1^C=0.407	K_1^D=0.569	
K_2	K_2^A=0.481	K_2^B=0.39	K_2^C=0.388	K_2^D=0.284	
K_3	K_3^A=0.289	K_3^B=0.476	K_3^C=0.332	K_3^D=0.358	
K_4	K_4^A=0.351	K_4^B=0.355	K_4^C=0.369	K_4^D=0.285	
k_1	k_1^A=0.094	k_1^B=0.069	k_1^C=0.102	k_1^D=0.142	
k_2	k_2^A=0.12	k_2^B=0.098	k_2^C=0.097	k_2^D=0.071	
k_3	k_3^A=0.072	k_3^B=0.119	k_3^C=0.083	k_3^D=0.09	
k_4	k_4^A=0.088	k_4^B=0.089	k_4^C=0.092	k_4^D=0.071	
R	R^A=0.048	R^B=0.05	R^C=0.019	R^D=0.071	

In table 2, K^i_j is the i-th factor of the j-level test results and the value of the sum. For example, K^A is the sum of the values of the first level of factor A for each test result, that is K^A =0.144 +0.055 +0.122 +0.054 =0.375. k^i is the k^i value divided by the number of occurrences of that level, for example, K^A means that the K^A value is divided by the factor A factor of the first occurrence of the number of times, that is k^A =K^A/4 =0.375 /4 =0.094. R^i is the maximum difference between the k^i values in the i-th factor, for example, the R^A value is the difference between the maximum value of the k^A value minus the minimum value, that is R^A =k^A -k^A=0.12 -0.072 =0.048.

As can be seen from Table 2, the influence of each factor on the friction coefficient is small. We can see from R^C <R^A <R^B <R^D, the influence of the friction factor from the main to the sub-order of factors are dispersant, PTFE, acetone, graphite. The k^i values in Table 2 reflect the effect of the i-th factor on the friction factor.

Surface wettability analysis

The higher the surface energy of the solid is easier to be wetted by the liquid, the smaller the contact angle of the liquid on the solid, the better the wetting performance. Table 3 shows the contact angle of each sample, and orthogonal experimental analysis.

Table 3 Contact angle analysis orthogonal table

试 验	A	B	C	D	接触角 /(°)
1	1	1	1	1	72.3
2	1	2	2	2	51.7
3	1	3	3	3	68.3
4	1	4	4	4	54
5	2	1	3	4	41.7
6	2	2	4	3	54.7
7	2	3	1	2	61
8	2	4	2	1	57.3
9	3	1	4	2	57.7
10	3	2	3	1	59.7
11	3	3	2	4	59.7
12	3	4	1	3	60
13	4	1	2	3	58
14	4	2	1	4	61.7
15	4	3	4	1	58.3
16	4	4	3	2	57.3
K_1	$K_1^A=246.3$	$K_1^B=229.7$	$K_1^C=255$	$K_1^D=247.6$	
K_2	$K_2^A=214.7$	$K_2^B=227.8$	$K_2^C=226.7$	$K_2^D=227.7$	
K_3	$K_3^A=237.1$	$K_3^B=247.3$	$K_3^C=227$	$K_3^D=241$	
K_4	$K_4^A=235.3$	$K_4^B=228.6$	$K_4^C=224.7$	$K_4^D=217.1$	
k_1	$k_1^A=61.6$	$k_1^B=57.4$	$k_1^C=63.8$	$k_1^D=61.9$	
k_2	$k_2^A=53.7$	$k_2^B=57$	$k_2^C=56.7$	$k_2^D=57$	
k_3	$k_3^A=59.2$	$k_3^B=61.8$	$k_3^C=56.8$	$k_3^D=60.2$	
k_4	$k_4^A=58.8$	$k_4^B=57.2$	$k_4^C=56.2$	$k_4^D=54.3$	
R	$R^A=7.9$	$R^B=4.8$	$R^C=7.6$	$R^D=7.6$	

Table 3 is the contact angle of each factor and the impact of various factors on the contact angle, we can see from RB <RC =RD <RA, the factors affecting the contact angle from the main to the sub-order of factors are acetone, dispersant and graphite, and polytetrafluoroethylene. From the calculation results, the influence of acetone on the contact angle was greater, while the influence of PTFE on the contact angle was the smallest. That maybe because the amount of acetone greatly affected the dispersion of PTFE. The amount of acetone is too small, PTFE dispersion was not uniform, more obvious reunion. The amount of acetone is too large, the amount of PTFE contained in the coating may be less. Due to the hydrophobic nature of PTFE, the actual effect on the contact angle should be the content of PTFE.

X-ray diffraction analysis

The crystal structure of the coating was analyzed by Bruker D8 Advance18 KW X - ray diffractometer, The diffraction pattern is shown in Fig 1. In Fig. 1, a is the X-ray diffraction spectrum of the sample # 5, b is the X-ray diffraction spectrum of the best sample, and c is the X-ray diffraction spectrum of the sample # 12. It can be seen from Fig. 1 that the peaks of the respective samples are substantially identical. Among them, the 12 # sample is not added graphite powder epoxy resin - PTFE composite coating. Comparison of X-ray diffraction standard PDF card can be seen, the main peak appearing at 2θ=17°is a peak of polytetrafluoroethylene (PTFE), the peaks appearing at 2θ=26°and 2θ=45°are carbon peaks. This is because PTFE is a crystalline structure and the content is more, graphite content is less. No graphite was added to sample # 12, and no carbon peak appeared. In the below of PTFE, the envelope peak is the peak of epoxy resin package, epoxy resin is amorphous structure, there is no crystalline peak.

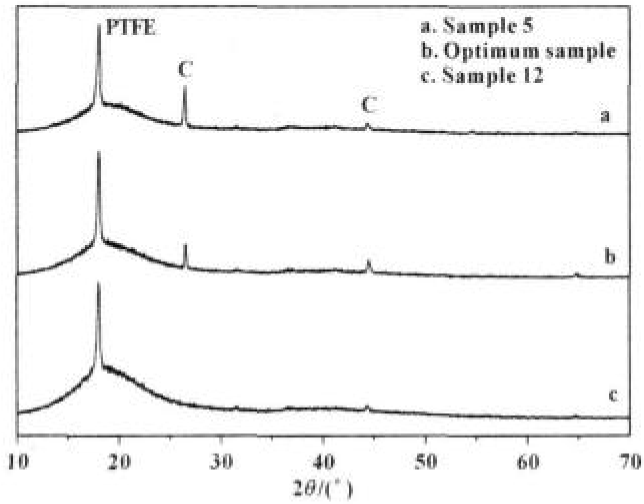

Fig.1 X-ray diffraction spectrum of epoxy resin-PTFE composite coating

Infrared spectroscopy

The bond length and bond angle of the molecule can be determined by infrared spectroscopy, and thus infer the three-dimensional configuration of molecules. By infrared spectroscopy, which can determine the presence of organic functional groups in the sample, can determine the chemical structure of the coating.

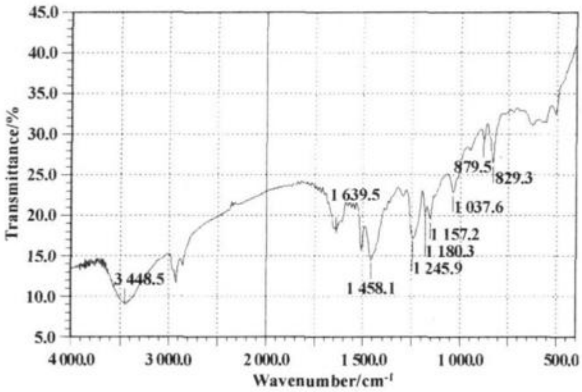

Fig.2 Infrared spectrum of epoxy resin-PTFE composite coating

Fig.2 is infrared spectrum of epoxy resin-PTFE composite coating. The strongest band of PTFE appeared in the frequency range of 1 250 to 1 110 cm-1, the characteristic frequencies of 1 157 .2, 1 180 .3 and 1 245.9 cm-1 are in the strongest spectral band of PTFE, the characteristic absorption peak is mainly caused by the stretching vibration of C-F bond, which indicates the presence of PTFE in the composite coating. And an absorption peak at the frequency of 3 448.5 cm-1, the absorption peak is in the frequency range of 3000 ~ 4000 cm-1, which is caused by the absorption of O-H bond stretching vibration, indicating that the composite coating contains - O-H bond material, inferred that the epoxy resin curing agent in the curing chain by the chain generated. The characteristic frequencies of 1 245 cm-1 and 879.5 cm-1 are between 870 and 1 280 cm-1 of cyclic ether bond, from this inference that it contain ether bond. And the absorption peak appeared at the frequency of 829.3 cm-1, and these absorption peaks appeared in the frequency range of epoxy compound, which indicated the existence of epoxy compound in the coating.

Differential thermal analysis

Using the differential thermal analysis, the melting point, decomposition temperature and so on of the samples can be obtained, and the thermal stability of the samples in a certain temperature range can also be seen.

Figure 3 is the differential thermal analysis of the epoxy resin -PTFE composite coating at temperature below 600℃. As can be seen from Figure 3, there is an endothermic peak at 381.34℃, which may be caused by the decomposition of epoxy resin. There is also an endothermic peak at 541.60℃ which is due to the decomposition of PTFE. Indicating epoxy resin PTFE composite coating at 300℃ is a stable state, can work properly.

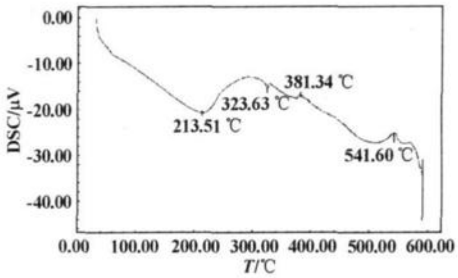

Figure 3 Differential thermal analysis of epoxy resin - PTFE composite coating

Scanning electron microscopy and energy spectrum analysis

The surface of the sample and the surface composition of the sample were analyzed by scanning electron microscopy. The results are shown in Table 4 and Fig.4.

Table 4 Relative proportions of coating surface composition

Element	Mass fraction /%	Atom fraction /%
CK	87.73	91.88
FK	12.27	8.12
Totals	100.00	

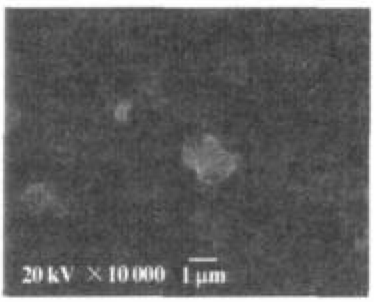

Figure 4 Epoxy resin - PTFE composite

It can be seen from Figure 4 that the surface of the epoxy resin- PTFE coating is relatively uniformly smooth and level. You can also see a small amount of white particles, it is caused by small amount of PTFE gathered. As can be seen from the relative proportions of the surface compositions of Table 4, the surface of the composite coating mainly contains F and C2 elements, and the mass ratio of F to C is 1: 7.15. The F element is one of the light elements, and the F element can be analyzed by electron microscopy. It is shown that the content of F in the composite coating is larger, that is, the content of PTFE is more.

Conclusion

Using the orthogonal experiment and the range analysis, the formula of epoxy resin- PTFE composite coating with the smallest friction factor was obtained, that is, epoxy resin and solid agent 3 g, PTFE 0.9 g, graphite 0.2 g, acetone 7 mL, dispersant FC-400 0.012 g, the minimum friction factor 0.037, the average thickness of about 300 micron. X-ray analysis showed that PTFE in coating is crystalline structure, epoxy resin is amorphous structure, the emergence of carbon peak shows the presence of graphite. FTIR and DTA showed that PTFE, graphite etc. are present as monomers in the coating, and in the stable below 300 ℃. Electron microscopy and energy dispersive X-ray spectroscopy (EDS) show that the surface of the composite coating is uniformly smooth and level, with a small amount of PTFE gathered, the mass ratio of F to C is 1: 7.15, indicating that the content of PTFE in the composite coating is higher, which plays a decisive role in reducing the friction. From the wetting properties of the coating and the medium water, due to the presence of PTFE in the coating, the contact angle is generally larger than without PTFE, indicating that the coating is more suitable for dry friction in the environment.