CHEMICAL ENGINEERING TRANSACTIONS

VOL. 59, 2017

A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet

Guest Editors: Zhuo Yang, Junjie Ba, Jing Pan

ISBN 978-88-95608- 49-5; **ISSN** 2283-9216

DOI: 10.3303/CET1759005

Preparation and Experimental Study on Influencing Factors of Mechanical Grinding Chemical Composite Ni-P Coating

Lianwei Kang

School of materials science and engineering, Hebei University Of Engineering, Handan, Hebei 056038, China
kanglianwei@126.com

Ni-P-PTFE composite film was prepared on the surface of high-speed steel ring, and the preparation process of Ni-P-PTFE composite film was studied in order to improve the wear resistance of mechanical seal ring. The surface morphology of Ni-P-PTFE composite film was observed by scanning electron microscopy (SEM). The dry friction properties of Ni-P-PTFE coating/graphite were investigated on a pin-disc friction and wear tester. The results show that a large amount of PTFE particles are encapsulated in the skeleton of Ni-P in Ni-P-PTFE composite membrane. Compared with high-speed steel/graphite, the friction coefficient of Ni-P-PTFE coating/graphite the Ni-P-PTFE coating has lower average friction coefficient and stable, which indicates that the coating has good self-lubricating effect. The Ni-P-Ti (CN) composite coating and Ni-P plating are the second. The Ti (CN) particles dispersed in the composite coating have a very important role in reducing the friction coefficient and reducing the wear rate of the coating.

1. Introduction

Mechanical end of the dynamic seal is the most widely used in fluid machinery, a contact seal, sealing ring and the static ring of the end of the spring under the action of the preload force to form a friction. For the frequent start and stop of the mechanical seal, the friction in the start-stop phase often dry friction state, resulting in a greater friction and friction torque, resulting in severe wear (Balu et al., 2013). A higher starting torque can cause shock and shock during the start-up phase, resulting in greater wear and failure of the mechanical seal prematurely. In the process of mechanical seal, due to media participation, sealing surface in addition to friction and wear will occur corrosion; fatigue, etc., seriously affect the service life of the seal and the reliability of mechanical system operation (Li et al., 2014). Low temperature engine the mechanical seal of the turbo pump is often impregnated with graphite static ring and steel ring. The friction and wear properties of the impregnated graphite and the steel are often strongly influenced by the formulation of the impregnated graphite and the impregnation process, showing strong start-up torque and strong time-varying friction coefficient (Wang et al., 2013). In order to improve the wear resistance of the mechanical seal ring surface, often use a special sealing end material or surface treatment methods. In the mechanical seal on the face of surfacing carbide, the advantages of high hardness, wear resistance, temperature resistance and corrosion resistance, the disadvantage is easy to produce pores and slag and surface hardness uneven. In the mechanical seal dynamic ring sealing surface set of alumina ceramic, high hardness and wear resistance, the drawback is brittle and high hardness and difficult to process (An et al., 2013). Filled poly tetra fluoro ethylene (PTFE) sealing material seals have excellent chemical stability, high and low temperature resistance and chemical resistance, and has a very low friction factor, the disadvantage is low mechanical strength, and susceptible to thermal deformation. Electrolyses nickel-phosphorus alloy has high corrosion resistance, wear resistance and weld ability, uniform coating thickness, can be coated with complex parts, in the electronics, computer, chemical, medical and other industries are widely used. The addition of PTFE particles to the chemical bath to disperse and deposit in the nickel-phosphorus alloy layer imparts self-lubricating properties (Ying et al., 2013). The results show that Ni-P-PTFE composite films can obtain excellent mechanical properties and self-lubricating properties. In order to improve the friction and wear behaviour of impregnated graphite and high speed steel, the Ni-P-PTFE composite film was chemically deposited on the surface of high speed steel. The preparation of Ni-P-PTFE composite film on the surface of high-speed steel substrate was

Please cite this article as: Lianwei Kang, 2017, Preparation and experimental study on influencing factors of mechanical grinding chemical composite ni-p coating, Chemical Engineering Transactions, 59, 25-30 DOI:10.3303/CET1759005

discussed (Rasu E, 2015). The performance of Ni-P-PTFE composite film was studied. The friction properties of graphite and Ni-PPTFE composite film under different pv values were discussed.

2. Experimental part

2.1 Experimental reagents

The main reagents used in the experiment are NiSO4•6H2O, NaH2PO2•H2O, CH3COONa, lactic acid, propionic acid, succinic acid, Pb (Ac)2•3H2O, PTFE emulsion (average particle size 0.2μm), N-ethyl-N-hydroxyethyl perfluorooctane sulfonamide (FC10, nonionic), perfluoroalkyl quaternary amine iodide (FC134, cationic).

The surface and cross-sectional morphology of the composite coating were observed by IE200M metallographic microscope and EVO-MA15 scanning electron microscope (ZEISS). The element composition was analysed by EDS energy spectrometer. 1000 X-ray diffractometer was used to analyse the phase composition before and after the heat treatment of the coating. The microhardness of the coating was tested by HXD-1000TMB digital microhardness tester. In order to minimize the influence of the matrix on the microhardness measurement of the coating, the experiment was carried out by using the cross-section hardness test method. The loading load was 25gf and the packing time was 10s. The average of the six test points was selected as the test result.

2.2 Preparation of Ni-P-PTFE composite film

High-speed steel surface followed by 500 mesh water sandpaper, 1200 mesh water sandpaper, W20 metallographic sandpaper, W5 gold sandpaper grinding treatment, through the TR2000 roughness measured surface roughness below Ra0.1μm (Arun et al., 2013).

Experiments were carried out using 20 steel (50mmx25mmx2mm) as a chemically deposited substrate. The steel sheet was ground with 1000 mesh SiC sandpaper and then subjected to degreasing treatment with an alkaline solution consisting of an organic solvent and deionized water and activated with a 10% (mass fraction) hydrochloric acid solution for 2 min. When the substrate surface was filled with bubbles, the surface was rinsed with deionized water and the surface was wetted with anhydrous ethanol to increase the wettability of the substrate in the bath.

Table 1: Formulation and conditions of composite plating solution

Ingredient	Value
Lactic acid	20~40 ml/L
Propionic acid	2ml/L
$NiSO_4 \cdot 6H_2O$	24g/L
Pb(Ac)	$2 \cdot 3H_2O$ 1~4 mg /L
PTFE	8g/L
Temperature	70~90 °C
PH value	3. 5~ 5. 0
Succinic acid	2g/L
CH_3COONa	3. 5g/L

The process formula and the coating conditions of the compound ferry are shown in Table 1. The results showed that the concentration of lactic acid was 20~40ml/L, pH 3.5~5.0, temperature was 75~95 °C, stabilizer lead acetate, 1~4mg/L. Surfactants are FC10, FC134 and FC134 + FC10.

2.3 Characterization of the sample

The appearance and microstructure of the composite film were observed by HITACHIS-3000N scanning electron microscope (Gong et al., 2014). The composite coating was analyzed by KEVEX type energy dispersive spectrometer (EDX) the friction and wear properties of the plating elements were evaluated using a CETRUMT-2 multifunctional friction and wear tester.

4. Results and discussion

4.1 Determination of the optimum process conditions

In this paper, orthogonal experiments are designed. The results of the evaluation test are mainly based on the plating speed, the appearance quality of the coating and the bath (Zhang et al., 2015). Plating speed according to the quality of the sample before and after plating:

$$u = 10000\Delta m / (Stp) \quad (1)$$

Where: u for the plating speed, μm/h. Δm is the coating quality g. S is the plating surface area, cm2. T is the plating time.
According to the results of the orthogonal table and the difference of the factors, it can be determined that the factors affecting the electro less nickel plating rate are: pH value, temperature, complexion agent lactic acid, stabilizer lead acetate, and get the best The technological conditions were pH=5.0, 90 °C, lactic acid content 40ml/L, lead acetate 1mg/L. With reference to the results of the orthogonal test, the experiment was repeated for each factor to examine the effect of a single factor on the bath.

4.2 Effect of fluoride surfactant on Ni-P-PTFE bath

In order to investigate the effect of fluorosurfactant on Ni-P-PTFE bath, the surface energy experiment was carried out under the following schemes (Jia et al., 2016), and then the composite plating was carried out:
(1) Add a fluorine-free surfactant to a PTFE emulsion of 15 ml/L.
(2) Add PTFE emulsion 15ml/L, add non-ionic FC10, no FC134.
(3) Add PTFE emulsion 15ml/L, add cation FC134, add non-ionic FC10.
Experimental results show that the surface free energy, coating weight gain and PTFE content in the composite film are shown in Table 2.
The FC10 and FC134 surfactants were added to the composite solution, the surface of the liquid was reduced to 18.15mN / m, the PTFE particles were well wetted and the stability of the bath was greatly improved. The cationic FC134 guaranteed the positive charge of the PTFE particles, With the NiP alloy to achieve the adsorption co-deposition, coating PTFE content of nearly 10%. During the whole experiment, the bath remained transparent and no aggregates and precipitates appeared. The single factor test method was used to determine the contents of FC134 and FC10 respectively, 0.01, 0.05 g/L.

Table 2: The effect of different surfactants on experimental results

Program type	Liquid surface energy E/(mN. M^{-1})	PTFE quality w/%	Coating weight gain m/mg
PTFE+base bath	41.76	Less than 1	7.96
PTFE+base bath+FC10	20.54	Less than 1	27.31
PTFE+base bath+FC10+FC134	19.07	9.32	142.63

4.3 Effect of process conditions on composite plating speed

With reference to the results of the orthogonal test, the effect of the single factor on the bath was examined by repeating the experiment for each factor. The results are shown in Figure 1.

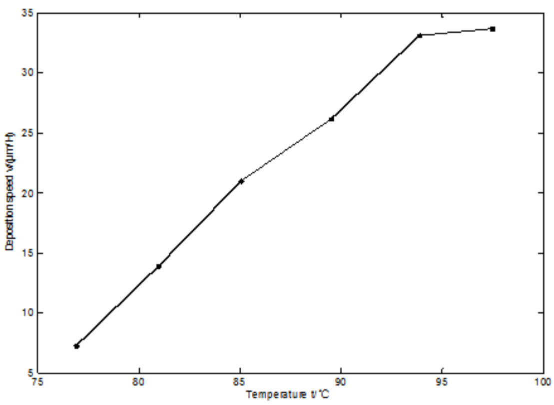

Figure 1: Deposition speed of the Ni-P-PTFE coating under temperature

As can be seen from Figure 1, with the increase of temperature, the plating rate increases abruptly. When the temperature is lower than 75 °C, the deposition rate is very slow (Wohlwend et al., 2012), and the plating rate increases slowly after 90 °C. The volume of the solution is reduced by about 30 ml.
It can be seen from Figure 1, in the pH range of 4.0 to 5.0, with the increase in pH, the plating rate is almost linear increase. When the pH is less than 4.0, the reaction is almost free and the plating rate is small. The

higher the pH, the more intense the reaction (Wu et al., 2012; Abu Bakar et al., 2017; Ahmad et al., 2017), the faster the plating rate, or even 60μm/h, but with the increase in pH, the quality of the coating has declined.

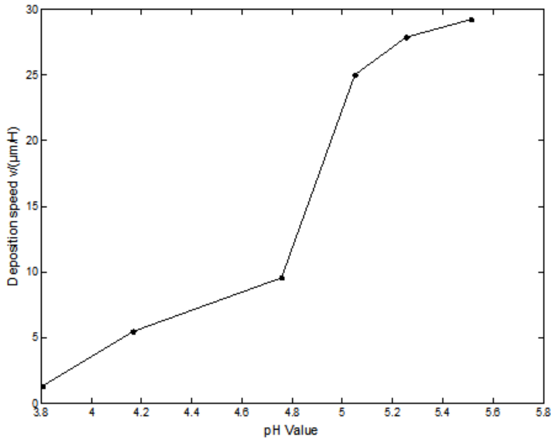

Figure 2: Deposition speed of the Ni-P-PTFE coating under pH value

It can be seen from Figure 2, the more the amount of stabilizer added, the slower the plating rate. When the stabilizer content is greater than 4mg/L, the plating rate drops rapidly and close to zero. Stabilizer with anti-catalytic effect, the amount of too large will inhibit the chemical plating reaction.
It can be seen from Fig. 2 that with the increase of the content of complexion agent, the plating rate increases rapidly, and when the content of complexion agent is 30ml/L, the cooling rate is slowed down and finally becomes stable.

4.4 Microstructure of composite film

Under the microscope, you can see the high-speed steel surface polishing left the fine scratches. Ni-P-PTFE composite film surface was gray and have no metallic luster. Ni-P-PTFE composite film surface is spherical and uniform, the surface roughness of about 0.7μm, far greater than the matrix roughness (0.016μm). A large number of particles on the surface of the coating can be observed by experiment. Focus on the surface black spot enrichment, using EDX energy spectrum components analysis, black spots for the wrapped PTFE. The energy fraction of PTFE was 12% and the volume fraction was about 35%.
The results of energy spectrum analysis showed that the addition of Ti (CN) particles could decrease the content of phosphorus in the composite coating by 6.4%~8.2% (mass fraction). The increase in phosphorus content in the Ni-P alloy coating is result in irregular arrangement of Ni atoms, which changes the microstructure of the crystal. Therefore, the XRD pattern of Ni-P-Ti (CN) composite coating shows that the co-deposition of Ti (CN) particles leads to the decrease of phosphorus content and the relative content of Ni in the composite coating. During the heat treatment, Ni And the diffraction peak of the composite coating has a strong Ni diffraction peak at 2θ values of 44 ° and 54 ° after the heat treatment.

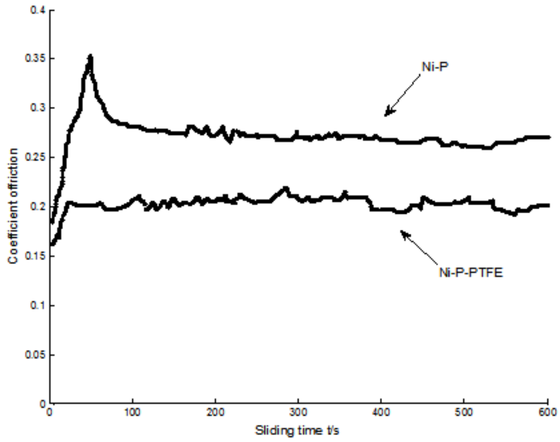

Figure 3: Variation of friction coefficient with sliding times

4.4.1 Friction properties of composite films

The results show that the friction coefficient of the high-speed steel matrix and its coated with graphite is measured under the condition of different pv values under the condition of dry friction. Average friction factor. The linear velocity is 0.6m/s, the specific pressure is 8~50MPa, and the pv value is 5~30MPa•m/s.

It can be seen from Fig. 3 that the Ni-P-PTFE coating exhibits a low average friction factor (between 0.16 and 0.18) under the experimental pv value. In the range of pv selected by the experiment, the average friction coefficient of the coating and impregnated graphite remained stable with the change of pv value.

Under the experimental conditions of 8.3MPa, 0.6m/s, pv=5MPa•m/s, the friction coefficient of the matrix and the sample after grinding with graphite is shown in Fig.

It can be seen that the high-speed steel samples and graphite in the friction start stage. The friction factor first rose sharply to close to 0.3, this short peak occurred in the run-in period, the peak reached 0.1. The initial friction coefficient of Ni-P-PTFE composite film is smaller than that of high-speed steel, and the composite film has almost no running-in phase. The lower friction factor can be maintained for 10min and there is no increasing trend.

Ni-P-Ti (CN) composite coating after heat treatment is produced compared with the composite coating before heat treatment, and there is no abrasive wear with large width. It has a more smooth and smooth wear surface and a large particle size is produced. Through the composite coating wear surface 1500 times. SEM image analysis shows that the surface of Ni-P-Ti (CN) composite coating has been punctured after 150 m sliding friction, but the coating is not penetrated, and the surface of Ti-CN (CN) Particles, wear the pit produced more debris. Similar to that of Ni-P-Ti (CN) composite coating, Ti (CN) particles protruded on the surface of the composite coating prevent direct contact with the surface of the coating. Therefore, the composite coating has a low coefficient of friction. Composite coating is denser, nickel-phosphorus matrix and Ti (CN) particles of greater binding force, so the heat treatment after the composite coating wear rate is small and the quality of wear with the vice. The Fe content of the wear surface after heat treatment is 13.68% higher than that before heat treatment, and the content of Ti, C and N is basically the same as that before heat treatment. It can be seen that the surface of Ni-P-Ti (CN) composite coating after heat treatment is mainly abrasive wear.

Ni-P-PTFE composite coating has excellent thermal stability. The experimental pair of counter-graphite is a high temperature resistant material, at room temperature with excellent lubrication performance. High-speed steel ring surface coated with Ni-P-PTFE coating than the non-coated high-speed steel and graphite with a pair of friction factor is smaller. This is due to the friction from the beginning that occurred between the two lubrication layer, that is, graphite and Ni-P-PTFE coating between, even in the high-speed rotating machinery to form a transfer film, but also to achieve good lubrication. This will help protect the ring seal surface, improve the sealing performance.

5. Conclusion

(1) Ni-P-PTFE composite coating was prepared on the surface of high speed steel, and the microstructure of the coating was tested. The optimum process parameters were obtained by orthogonal test, and the effect of different experimental conditions on the composite plating was studied by single factor experiment.

(2) The friction coefficient of Ni-P-PTFE/graphite pair under dry friction is significantly lower than that of high speed steel/graphite, and the friction factor of the coating is stable at different pv values. Maintained at a lower value (0.16~0.18), and remained constant for a certain period of time.

Reference

Abu Bakar S.H., Hamid M.K.A., Wan Alwi S.R., Manan Z.A., 2017, An Industrial Case Study Application in Synthesizing a Feasible Heat Exchanger Network, Chemical Engineering Transactions, 56, 775-780, DOI: 10.3303/CET1756130

Ahmad Z., Bahadori A., Zhang J., 2017, Prediction of Combustion Efficiency Using Multiple Neural Networks, Chemical Engineering Transactions, 56, 85-90, DOI: 10.3303/CET1756015

An Z., Li M.Y., Ge N.H., 2013, Study on the Fabrication and Properties of Ni-P Composite Coating. Key Engineering Materials, 537, 265-268, DOI: 10.4028/www.scientific.net/KEM.537.265.

Arun K.V., Satish K.S., 2013, Influence of Electroless Ni-P Coating Bath Parameters on the Strength and Surface Finish, 1(2), 15, DOI: 10.12966/ram.05.02.2013.

Balu P., Hamid S., Kovacevic R., 2013, An Experimental Study on Slurry Erosion Resistance of Single and Multilayered Deposits of Ni-WC Produced by Laser-Based Powder Deposition Process. Journal of Materials Engineering and Performance, 22(11), 3398-3413, DOI: 10.1007/s11665-013-0620-1.

Gong Y.D., Wen X.L., Cheng J., 2014, Experimental study on fabrication and evaluation of a micro-scale shaft grinding tool, Journal of Mechanical Science and Technology, 28(3), 1027-1037, DOI: 10.1007/s12206-013-1176-6.

Jia D., Li C., Zhang Y., 2016, Experimental research on the influence of the jet parameters of minimum quantity lubrication on the lubricating property of Ni-based alloy grinding, The International Journal of Advanced Manufacturing Technology, 82(1), 617-630, DOI: 10.1007/s00170-015-7381-y.

Li Y.M., Zhang X., Wang A., 2014, Research on Preparation and Properties of Antiwear and Anticorrosion Composite Coating Ni-P-SiC, Advanced Materials Research, 988, 117-120, DOI: 10.4028/www.scientific.net/AMR.988.117.

Rasu E., 2015, Study on Structural Characterization of Surfactants Influence on Electroless Ni-P Deposits, British Journal of Applied Science & Technology, 5, 250-257, DOI: 10.9734/BJAST/2015/12640.

Wang H., Shi P., Yu H., 2013, Preparation and Micro Mechanical Properties of Nano-Al2O3 Particles Strengthened Ni-based Composite Coatings, Physics Procedia, 50:225-230, DOI: 10.1016/j.phpro.2013.11.036.

Wohlwend J.L., Amama P.B., Shamberger P.J., 2012, Effects of Titanium-Containing Additives on the Dehydrogenation Properties of LiAlH4: A Computational and Experimental Study, Journal of Physical Chemistry C, 116(42), 22327–22335, DOI: 10.1021/jp3050109.

Wu Y., Lototskyy M.V., Solberg J.K., 2012, Effect of microstructure on the phase composition and hydrogen absorption-desorption behaviour of melt-spun Mg-20Ni-8Mm alloys, International Journal of Hydrogen Energy, 37(2), 1495-1508, DOI: 10.1016/j.ijhydene.2011.09.126.

Ying L.X., Liu Y., Yang J.T., 2013, The preparation and properties study on antiwear and antifriction composite coating Ni-W-P-SiC-WS2, Journal of Functional Materials, 44(22), 3247-3250, DOI: 10.3969/j.issn.1001-9731.2013.22.008.

Zhang J.H., Zhao Y., Tian F.Q., 2015, Kinematics and experimental study on ultrasonic vibration-assisted micro end grinding of silica glass, The International Journal of Advanced Manufacturing Technology, 78(9), 1893-1904, DOI: 10.1007/s00170-014-6761-z.