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The Research on Heat Treatment Technology of Ni - Mo - P Electroless Plating

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Abstract. In this paper, study on the Ni-Mo-P electroless plating which are heat treated with different time and temperature. Test and record the hardness of coatings by the micro-hardness tester. Modeling analysis and optimize the parameters of the coating hardness by uniform design methods. The results indicate that under the same experiment parameters, the maximum hardness of the Ni-Mo-P electroless plating are mainly effect by the heating time and temperature. The hardness decrease when the heat temperature is over high and heating over long time. Got the hardest coating after the heat treatment technology of 380 ~ 400 ℃ for 3 h.

Introduction

As a fine surface treatment technology, electroless plating can yield uniform deposits on surface of conductor and non-conductive material, and the operation is easy, so it has been got great attention by industry and academia[1].In the further improve the wear resistance and hardness of nickel-molybdenum phosphorus coating, the general nickel-molybdenum alloy coating for heat treatment, but in the heat treatment process parameter selection, access to literature found that different literature have different conclusions. It is believed that annealing at 400 ℃ × 3h get the maximum hardness[2,3]; there is that, in the heat treatment temperature of 250 ℃ by prolonging the heat treatment time can also increase the hardness of the coating[4]; there are some studies that the maximum hardness of the best heat treatment temperature should be 500 ° C. In order to find out the effect of heat treatment temperature and time on the hardness of Ni-Mo-P electroless plating, the optimum heat treatment process was selected. In this paper, the experimental points were selected most effectively through experimental design. Then the measured values of the responses are obtained by experiment, and the experimental conditions for obtaining the optimal response values are obtained by data analysis. UniformDesignVersion 3.0 uniform design software was used to build a mathematical model by computer. The average hardness value measured at different heat treatment temperature and time was analyzed and optimized, and the optimal heat treatment time and temperature were obtained.

Experimental Program and Step

Experimental Protocol. Experiments in the past on the basis of the proportion of chemical bath solution ratio, this formula plating speed fast, stable bath. In order to save material, reduce the number of experiments in this experiment using uniform design method [U9*(9^4)].So that nine samples were placed in the plating solution for 2 hours of electroless plating. The specimens were then heat treated at different temperatures and different heating times in a 2KW box-type electric furnace (with a temperature control accuracy of ± 2 ° C). After the heat treatment, the specimens were selected with a HX-1000 microhardness tester on each block Plating Hardness Measurements Remove the maximum and minimum values Select the average. The mathematical model and the regression analysis of the hardness values of the coatings under different heat treatment conditions were used to obtain the optimum heat treatment.

Electroless Plating Process. Electroless nickel-molybdenum phosphorus process: A3 steel sheet → sample → pre-grinding sample → cleaning → degreasing → cleaning → pickling → cleaning

→ electroless nickel plating → cleaning → drying → packaging[5-7].

Preparation of Plating Solution. Nickel sulfate, sodium citrate, lactic acid, sodium hypophosphite, sodium acetate, sodium moly date, and sodium dodecyl sulfate are mixed and dissolved in the order of once.

Hlating Condition. pH:8.0~8.2; Temperature:81~82℃; Time (t) :2h.

Experimental Design. The original value of the factors in the uniform design table in the way specified by the layout or uniform design of the requirements of the test parameters set and output for experimental reference data is called the establishment of experimental program. In the experiment, we choose "Unconstrained Unconventional Design" for experiment with single factor of 9 factors and "Interval of setting horizontal value by program" in "Experiment Design", and then input in the "upper and lower limit of each factor level" The upper and lower limit values of each factor level. Click on the "Create a Test Protocol" button to export the unconstrained recipe design test solution.

Heat Treatment and Hardness Testing. The specimens were heat-treated at different temperatures and different heating times in a 2KW box-type electric furnace (temperature control accuracy: ± 2 ° C). After heat treatment, the specimens were microhardened with HX-1000 microhardness hardness Measurement comparison, as shown in Table 1.

Table 1 test piece in a test program under the coating hardness

NO	Coating hardness					The average hardness	The heating temperature	Heating time
	1	2	3	4	5			
1	593	701	643	701	643	643	200	2
2	726	766	701	701	766	739	250	3.5
3	841	841	766	841	841	841	300	5.0
4	927	927	1027	825	825	927	350	1.5
5	927	1027	1027	1027	927	1027	400	3.0
6	766	841	927	841	927	841	450	4.5
7	548	508	643	701	548	701	500	1.0
8	666	584	726	677	584	666	550	2.5
9	623	574	689	643	584	623	600	4.0

Establishment of Mathematical Model and Regression Analysis.

UniformDesignVersion 3.0 uniform design software was used to build a mathematical model by computer. The average hardness value measured at different heat treatment temperature and time was analyzed and optimized, and the optimal heat treatment time and temperature were obtained.

Based on the experience to complete the establishment of mathematical models, according to the heat treatment time and temperature on the hardness of metal values of the law, the experiment to establish the following mathematical model:

$$y = b + b1x_1 + b2x_2 + b3x_1^2 + b4x_2^2 \tag{1}$$

Where: y for the coating Vickers hardness; x1 for the heat treatment temperature; X2 is the heat treatment time; b0, b1, b2, b3, b4 are the regression coefficients.Regression equation significance test:

Table 2 Variable analysis

Mean square of the variation sources	Sum of squares	Degrees of freedom	mean square	mean square ratio
Regression	U=1.70e+5	K=4	U/K=4.24+4	F=11.62
Remainder	Q=1.46e+4	N-1-K=4	Q/(N-1-K)= 3.65e+3	
Total	L=1.84e+5	N-1=8		

Sample size: N = 9, the significance level: α = 0.05, test values: Ft = 11.62, critical value: F (0.05, 4, 4) = 0.05, Ft > F (0.05, 4, 4), the regression equation is significant.

Residual standard deviation s = 60.4

Regression coefficient test value:

T test value (df = 4):t(1)= 6.104; t(2)= 1.856;t(3)=-6.468; t(4)=-1.815.

F test value (df1 = 1, df2 = 4):F(1) = 37.71; F(2)= 3.444;F(3)= 41.83; F(4)= 3.294.

Partial regression square sum U (i):U(1)=1.38e+5; U(2)=1.26e+4; U(3)=1.53e+5; U(4)=1.20e+4.

Partial correlation coefficient ρ (i):ρ1,234= 0.9508; ρ2,134= 0.6802; ρ3,124=-0.9554; ρ4,123=-0.6720.

The contribution of each equation to regression (ranked by partial regression and descending order):U(3)=1.53e+5,U(3)/U=90.0%;U(1)=1.38e+5,U(1)/U=81.1%;U(2)=1.26e+4,U(2)/U=7.41%; U(4)=1.20e+4, U(4)/U=7.09%.

The contribution of the fourth equation [X (2) * X (2)] to the regression is the least, and the significance test is performed:The test value F (4) = 3.294, The critical value F (0.05, 1, 4) = 7.709,F (4) ≤ F (0.05,1,4), this factor (equation) is not significant.

Table 3 Residual analysis

number	Observed value	Regression value	Regression value-observed value X	(Regression value-observed value X) / 100 (%)
1	643	625	18.0	-2.80
2	739	804	-65.0	8.80
3	841	816	25.0	-2.97
4	927	897	30.0	-3.24
5	1027	974	56.0	-5.44
6	841	883	-42.0	4.99
7	701	727	-26.0	3.71
8	666	702	-36.0	5.41
9	548	508	40.0	-7.30

The regression equation of each heat treatment coating was analyzed by software and the t-test and F-test of the equation were obtained. The regression equation of different coating was obtained after removing the non-significant equation. The results are as follows:

$$y = -678 + 7.29x_1 + 166x_2 - 0.0951x_1^2 \tag{2}$$

From the residual analysis table to see the experimental observations and regression values, the deviation is not within the normal range, indicating (hardness) experimental data is basically reliable, and some of the experimental process of the phenomenon, the deviation mainly from the naked eye and The contribution of the fourth equation term [X (2) * X (2)] to the regression is the least, and the significance test is made: the test value F (4) = 3.294, The critical value F (0.05,1,4) = 7.709, F (4) ≤F (0.05,1,4), this factor (equation) is not significant to analyze the impact of time on the test block is small.

F (4) ≤ F (0.05, 1, 4), this factor (equation item from the equation of the contribution of the regression of U (3) contribution, indicating that the equation in the process of factor 1 that the temperature of the test block the impact is greater.) Not significant. In order to get the optimal heat treatment scheme, the parameters are optimized.

Parameter Optimization

Test optimization parameter settings: In the test optimization program settings window, there are factors on the lower limit, the value of the desired direction, the weight of three items, respectively, in the upper and lower limits of the input temperature and time limit, according to the program in the desired direction and the right Values do not need to be entered.

The setting of the parameters is divided into the grid parameter setting and the simplex method parameter setting. In this experiment, the parameters of the grid try parameter are set. Only one step is needed to set the parameters. The normal stride in the default stride.

After the completion of the above settings and click on the "automatic test optimization" button to start the automatic test optimization analysis, the results are as follows.

Table 4 conditions optimization Settings

factors	The upper bound	The next	The initial steps	The initial step
1	600.0	200.0	1.0e-3	0.400
2	5.00	1.00	1.0e-3	4.000e-3

Table 5 optimized test conditions

factors	The optimal conditions	The maximum value of expected indicators	The measured values
1	383.1	977(±168)	1027
2	3.116		

From the table to see the coating hardness measured values in the optimal analysis of the expected indicators, indicating that the uniform design optimization is reasonable, the establishment of the corresponding mathematical model is also appropriate.

From the design optimization results can be optimal test conditions for 3 hours 383 ℃, with the previous study of the temperature at 400 ℃ or less, can explain the further enhance the hardness of the coating, its optimal process should be 380 ℃ ~ 400 ℃ , The time is 3 hours.

Conclusion

It can be seen from the regression equation that the heat treatment time has little effect on the test piece, but the temperature has a great influence.

Optimization analysis of nickel-molybdenum phosphorus coating hardness and temperature and heating time. But the hardness will drop when the temperature is too high.

According to the experimental and data analysis when the temperature is heated to 380 ~ 400 ℃ heating time 3 hours, the coating hardness of the maximum.

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