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PREDICTION OF MECHANICAL CHARACTERISTICS AFTER RECRISTALIZATION ANNEALING OF PdNi5 ALLOY BY USING STATISTICAL ANALYSIS

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ABSTRACT

In order to supplement the existing database of platinum metals, certain tests were carried out on samples of alloys PdNi5 melted and casted in vacuum. Vickers hardness (HV) values, tensile strength (Rm) and elongation (A) after recrystallization annealing were determined in the function of annealing temperature (200-1000^{0}C) annealing time (20, 30 and 40 min) and deformation degree (60, 85 and 97%). Full factorial experimental design at three levels was applied. The temperature, time and deformation degree of recrystallization annealing were taken as the independent variables, while the hardness, tensile strength and elongation value after recrystallization annealing were observed as responses of the system. Based on the results of statistical analysis (ANOVA) mathematical models for all mechanical characteristics will be defined. These defined mathematical models will allow the selection of the operating parameters of the manufacturing process of PdNi5 alloys, for catalysts-catchers, which can be used for reliable predictions of the values of mechanical properties such as hardness, tensile strength and elongation, which is a key factor in the application of palladium catalysts to catch platinum metals in high-temperature catalytic processes.

Keywords: PdNi5 alloy, modeling, recrystallization annealing, catalysts-catchers

1. INTRODUCTION

In order to recover the lost platinum, catchment gauzes made of Pd-Ni alloy have been introduced into ammonia oxidation reaction in a combination with conventional platinum catalysts [1,2]. Keeping in mind the complex influence of deformation degree, annealing time and temperature on mechanical characteristics, the experiments with annealing time and annealing temperature change on alloy Pd-5%mas Ni with different degree of deformation, was carried out in this work[3].

2. EXPERIMENTAL PART

PdNi5 wires produced in three levels of deformation (60, 85 and 97%) were used in the experiment. After rolling samples were exposed to the recrystallization annealing at the annealing temperature (200-1000^{0}C) for three levels of annealing time (20, 30 and 40 min) in laboratory conditions. The investigation include a testing the influence of annealing parameters and deformation degree on changes in mechanical characteristics of rolled PdNi5 wires, because the mutual dependence was

observed. For fardness testing, PdNi5 plates 0,4 mm thick were used and for Rm and A testing, PdNi5 wire 1,7 mm in diameter was used. Tensile strength and elongation were measured using a universal device for tensile testing, type „Mohr + Federhaf + Losenhansen“ – Manheim. Before testing all samples were cut to a length of 150 mm.

3. RESULTS AND DISCUSSION

Table 1 shows the input factors, their levels, and the responses of the system in the form of descriptive statistics.

Table 1. Descriptive statistics of the input (Xi) and output Y(A, Rm, HV) values of annealing process parameters of PdNi5 alloy after the rolling process

Descriptive Statistics

	Mean	Std. Deviation	N
A	22.041975	18.8556680	81
X1	600.00	259.808	81
X2	30.00	8.216	81
X3	80.67	15.509	81

а) A, %

Descriptive Statistics

	Mean	Std. Deviation	N
Rm	446.872469	134.9407401	81
X1	600.00	259.808	81
X2	30.00	8.216	81
X3	80.67	15.509	81

б) Rm, MPa

Descriptive Statistics

	Mean	Std. Deviation	N
HV	145.0315	57.63245	81
X1	600.00	259.808	81
X2	30.00	8.216	81
X3	80.67	15.509	81

в) HV

For the modeling of all the responses linear dependence $Y_{(HV,Rm,A)} = f(X_1, X_2, X_3)$ was used. The regression coefficients were determined using the well known method of least squares using the software package SPSS Statistics. On the basis of those results mathematical models of all three responses that describe the influence of temperature and time of recrystallization annealing and the deformation degree on each of the observed responses, have been established:

$$Y_A = -30{,}72 + 0{,}066X_1 + 0{,}051X_2 + 0{,}142X_3 \quad (1)$$

$$Y_{Rm} = 653{,}541 - 0{,}472X_1 - 0{,}135X_2 + X_3 \quad (2)$$

$$Y_{HV} = 215{,}7 - 0{,}197X_1 - 0{,}046X_2 + 0{,}606X_3 \quad (3)$$

Based on the defined mathematical dependence of the output variables on the input variables, it is possible to predict the value of the response if input values are known. VIF values at $p<0.05$, which are equal to 1, indicate satisfactory statistical reliability of the results [4]. The adequacy of the model was tested using ANOVA test. Results of ANOVA test of the developed models for each of the responses are shown in Table 2.

Table 2. Results of ANOVA test of the finite model for each of the responses

ANOVA[b]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	24089.977	3	8029.992	142.045	0.000[a]
	Residual	4352.920	77	56.531		
	Total	28442.897	80			

a. Predictors: (Constant), X3, X2, X1

ANOVA[b]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1222692.279	3	407564.093	134.097	0.000[a]
	Residual	234027.988	77	3039.325		
	Total	1456720.267	80			

a. Predictors: (Constant), X3, X2, X1

b. Dependent Variable: Rm

ANOVA[b]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	216487.074	3	72162.358	112.862	0.000[a]
	Residual	49232.853	77	639.388		
	Total	265719.927	80			

a. Predictors: (Constant), X3, X2, X1

b. Dependent Variable: HV

b. Dependent Variable: A

Table 3. Evaluation of the model

Model Summary[b]

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	0.920[a]	0.847	0.841	7.5187389

a. Predictors: (Constant), X3, X2, X1

b. Dependent Variable: A

Model Summary[b]

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	0.916[a]	0.839	0.833	55.1300691

a. Predictors: (Constant), X3, X2, X1

b. Dependent Variable: Rm

Model Summary[b]

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	0.903[a]	0.815	0.808	25.28612

a. Predictors: (Constant), X3, X2, X1

b. Dependent Variable: HV

Analysis of variance of the regression equations, confirms that at a significance level of 95% (α=0,05), by using the selected regression equations the mechanical properties of the alloy PdNi5 during recrystallization annealing, when changing the annealing time and temperature as well as the degree of prior cold deformation, can be predicted (Calculated F > tabular F(0,05; 77;3)=2,74) . The coefficients of determination R^2 are 0,847 ; 0.839 and 0.815 respectively for the A , Rm and HV (Table 3) , ie. over 80 % of the variance in the results of the change of mechanical properties of alloy PdNi5 is explained by the models (1-3) .

These coefficients of determination indicate an excellent agreement between the experimental and by models (1-3) predicted values.

Certain mechanical properties are changed variously under the influence of the diffusion processes that take place during annealing [5,6]. Based on the regression models, the parameters of recrystallization annealing which will provide the required values of the mechanical characteristics of the alloy PdNi5 for the application of palladium catalysts to catch platinum metals in high-temperature catalytic processes, can be easily set.

4. CONCLUSION

Using a full factorial design of the experiment it was examined and compared the impact of changes of process variables (degree of cold deformation, annealing temperature and time) on the formation of the response models for all tested mechanical properties. Linear members of the input variables have statistically significant effect (at level $p<0,05$). The results obtained using the modelling show that dependence for A, Rm and HV can be described by a linear equation, based on which it is possible to predict the value of these mechanical characteristics for different values of deformation degree, temperature and time during recrystallization annealing.

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