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PREDICTION OF HARDNESS AFTER HOMOGENIZATION ANNEALING OF PdNi5 ALLOY BY USING STATISTICAL ANALYSIS

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ABSTRACT
This article presents the results of defining the mathematical model which describes the dependence of hardness after homogenization annealing of the major influencing factors in this process (temperature and time). Factorial design was used, type 3^2. The temperature and time of homogenization annealing were taken as the independent variables, while the hardness value after homogenization annealing was observed as response of the system. The observed levels of independent variables were: 30, 60 and 90 minute for time; 800, 900 and 10000C for temperature. Based on the results of statistical analysis (ANOVA) a conclusion can be made on the ability of such defined set of independent variables to predict the hardness values. The purpose of such a model is obtaining optimal states of the system that enable efficient operations management.
Keywords: modeling, factorial design of experiment, homogenization annealing, Pd-5Ni alloy

1. INTRODUCTION
Pd-Ni alloys are used in the production process of nitric acid as the Pd-catalyst-trap in a combination with conventional platinum catalysts. The role of Pd-catalyst-trap consists in reduction of volatile platinum oxide from gas flow to the metal form and retention of platinum metal on the surface of Pd catalyst-trap [1-4]. Pd-Au alloys were previously used for making Pd-catalyst-trap, until nowadays the alloys of Pd-Ni system are used [5,6]. The alloys of this system have a complete solubility of components in the solid state The structure of palladium-nickel alloy is single-phase and consists of polyhedral face-centered cubic grains of α-solid solution. Formation of this structure is possible only under conditions of slow crystallization because the concentration difference could equate by diffusion. Due to rapid solidification of the melt in graphite molds, as in real conditions, due to the rapid cooling of crystal, the crystal segregation is present. In order to eliminate segregation of Pd-5Ni crystal alloy, the samples were subjected to homogenization annealing. During homogenization, annealing, aging, and other forms of heat treatment of alloys can single out the operations in which the temperature and time are factors that influence the properties of alloys [7-10]. The choice of level the variation factors is limited by the laws of diffusion processes occurring in the alloy during heating prejudice to the fact that at high temperatures, these processes are complete within a relatively short time, while at low temperatures, they are sluggish and takes more time[8].

2. EXPERIMENTAL PART
Optimal conditions of the homogenization annealing process of PdNi5 alloys are investigated in this paper. All experimental investigations were carried out on PdNi5 samples with content of Ni in all samples as follows:5%mass, Pd-purity 99,99% and Ni-purity 99,95%. Palladium, used for making all the samples, originating from the RTB Bor production of electrolytic copper, was obtained as a by-product. The additional refining treatment in the Mining and Metallurgy Institute was carried out in order to increase the purity of palladium. Impurities in the samples were: Ag, Cu, Fe, As, Sb and Bi.

In order to achieve better compacting of materials, Pd-powder and Ni-sheet metal were pressed on a hydraulic press with force of 270 daN/cm^2. Melting of PdNi5 alloy was carried out in the medium frequency induction furnace, in MgO casting pot, size h1xh2 = 85x80 mm, d1xd2 = 65x55 mm. Casting temperature of PdNi5 alloy was 1520^{o}C. Molten batch was overheated before casting for 150-170^{o}C. Casting was done in graphite mold, pre-heated at temperature of 350-400ºC. Annealing of samples was carried out in an electric resistance furnace of chamber type LP08. The optimized parameter was Hardness (Vickers) PdNi5 alloy after homogenization annealing. Factors were temperature (T, ^{0}C) and time (τ, min.) of homogenization annealing. Levels of factors of homogenization annealing were determined on the basis of previous research in MMI Bor. Factorial design type 3^2 was used for a selection of optimal conditions. Microstructure testing of the alloy was done on samples with diameter of 20 mm and 5 mm in height. The samples were prepared according to standard procedure. Optical microscopy was performed on metallographic microscope EPYTIP 2, with magnification 200x.

3. RESULTS AND DISCUSSION

The structure of palladium-nickel alloy is single-phase and consists of polyhedral surface-centered cubic grains of α-solid solution. The appearance of this crystal structure is possible only when solidification is slow, while, in conditions of the rapid solidification of the melt in graphite molds, as in our case, there is a crystal segregation which in the metallographic cross section can be identified by stratified built inhomogeneous dendrites, as shown in Figure 1.

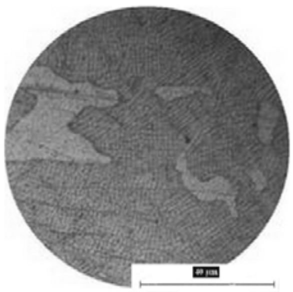

Figure 1. 95%Pd+5%Ni. Cast state. Inhomogenous α-solid solution. Crystal segregation.

Homogenization annealing regime and systems response are shown in Table 1. Results of statistical analysis of experimental research are presented in Table 2and Table 3.
Both variables (temperature and time) significantly correlate with the output variable Y_hardness (-0.773 and -0.509). The correlation value between independent variables is <0.7, and then the all independent variables were retained, otherwise from two variables, which strongly correlate, should make one common variable. As a part of the multiple regression procedure , the SPSS performs “collinearity diagnostics “ of variables. It often indicates the problems with multicolinearity. The results of this diagnosis are given in Table 2.

Table 1. Experimental conditions and system response

Nº	x1_temp.	x2_time	Y_hardness
1	800	30	81.60
2	800	60	79.30
3	800	90	77.50
4	900	30	77.10
5	900	60	76.20
6	900	90	75.30
7	1000	30	76.30
8	1000	60	75.90
9	1000	90	74.50

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Table 2. The regression coefficients[a]

	Unstandardized Coefficients		Standardized Coefficients			95,0% Confidence Interval for B		Correlations			Collinearity Statistics	
Model	B	Std. Error	Beta	t	Sig.	Lower Bound	Upper Bound	Zero-order	Partial	Part	Tolerance	VIF
1 (Constant)	97.194	3.598		27.017	0.000	88.391	105.997					
x1_temp.	-0.019	0.004	-0.773	-5.017	0.002	-0.029	-0.010	-0.773	-0.899	-0.773	1.000	1.000
x2_time	-0.043	0.013	-0.509	-3.302	0.016	-0.074	-0.011	-0.509	-0.803	-0.509	1.000	1.000

a. Dependent Variable: Y_hardness

In order to test the regression model as a whole, a hypothesis about the (non) existence of a linear relationship between the observed variables is placed , and it is necessary to perform an analysis of variance (ANOVA).

Table 3. ANOVA[b]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	32.697	2	16.348	18.035	0.0003[a]
	Residual	5.439	6	0.906		
	Total	38.136	8			

a. Predictors: (Constant), x2_time, x1_temp b. Dependent Variable: Y_hardness

Equation model based on the data in Table 2 as follows:

$$Y=97,194-0,019X_1-0,043X_2 \quad (4)$$

For a graphical representation of optimization the experimental conditions of homogenization annealing for PdNi5 alloy, MATLAB R2012b software was used [23] (Figure 2). Metallographic photos of structure of sample PdNi5 after homogenization annealing is shown in Figure 3.

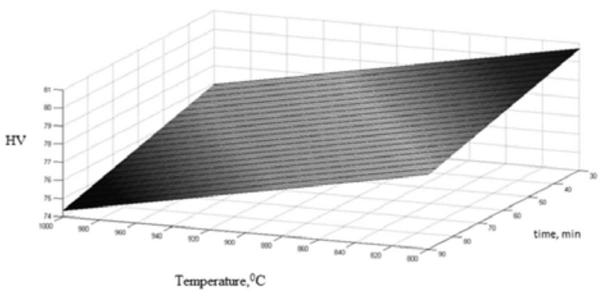

Figure 2. Surface diagram according to the equation (4)

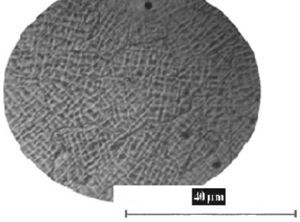

Figure 5. 95%Pd+5%Ni.After homogenization annealing at 1000^{0}C and 90 min. Solid solution grain boundaries are clearly marked.

4. CONCLUSION

This paper it is shows that with a relatively small number of experiments, using factorial design, a reliable model can be obtain describing the effect of temperature and time of homogenization annealing on PdNi5 alloys hardness . Hardness of PdNi5 alloy, melted and casted in vacuum, with increasing temperature and time of homogenization annealing slightly decreases. The results, obtained using the modeling, show that this dependence can be described by a linear equation, based on which it is possible to predict the value of alloy hardness for different values of temperature and time at homogenization annealing.

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6. LITERATURE

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