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OPTIMIZING OF PARAMETERS DIPPING METALLIZATION OF TAGUCHI METHOD

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Abstract

Zinc coatings obtained by immersion metallization are an effective and economical way to protect against corrosion also for high carbon steels, foundry iron alloys. However, the difficulties encountered in surface preparation due to oxide contamination, as well as the presence of carbon (graphite) from the casting process or cast iron sludge, make cast irons zinc-plated by manufacturers reluctantly. The technological properties of the zinc baths and the technological properties of the coatings obtained vary considerably depending on the various impurity and alloying additives. The effect of zincing time significantly affects the thickness of zinc coatings and is dependent on the mass and thickness of the sample. The submerged object heats up faster or slower, depending on the thickness, and its temperature first reaches and then exceeds the melting point of the zinc. However, to avoid zinc over time, it is appropriate to respond to the current ISO standard EN 1461:2009, which specifies the minimum thickness of zinc coatings on steel substrate. The subject of the study was the optimization of parameters metallization using the Taguchi Method. The analysis took into account the influence of the immersion time and temperature of the bath of molten zinc on the thickness of the zinc coating. The test was conducted for three different times, i.e. 30, 60 and 150 seconds and for three different values of the temperature of the bath: 440, 460, 480 [°C]. In order to predict the thickness of the coating, multiple regression analysis was applied, which showed that the experiment was carried out correctly and falling within the limits of tolerance error

Keywords: *metallization, galvanizing, Taguchi method, optimizing*

1. Introduction

One of the effective methods of protection against corrosion of metal structures is metallization. It involves immersing finished products with a suitably prepared surface in the molten coating metal. The material that will be subjected to the thermal coating method must have a significantly higher melting temperature than the coating material [1, 3].

The method for its simplicity is relatively cheap and does not require the use of expensive machinery and equipment. These coatings are remarkably thick and resistant to corrosion. Their distinctive feature is the diffusion bonding of the coating to the substrate [5]. This is due to the diffusion on the boundary of the coated and liquid metal, and the formation of intermediate layers composed of inter crystalline phases. The structure and thickness of the interlayer phase affects its mechanical properties. Excessive thickness of this phase lowers the plasticity of the coatings. The quality of hot-dip coatings is influenced by surface preparation, chemical composition of the coated metal, composition and temperature of the bath, bathing time, method and speed of bathing products [1, 4].

Optimization of the quality according to the Taguchi method is to minimize the volatility of the product due to the presence of noise factors, and while maximizing the volatility of the product in response to the signal factors. Interference factors (noise) remain out of control, while signal factors can be controlled by the operator. The primary effect of this optimization is to schedule the device/process to achieve the highest signal to noise ratio (S/N). In the planning of the experiment, this method helps to answer the question: "How to plan experience to get as much useful information as possible at the least cost of effort?" In this paper was applied the following optimization criterion: the thickest zinc dip coating for specific processing [2].

2. Material and method

In order to execution research on the influence of process parameters on the thickness of zinc immersion coating, steel samples S235 of dimensions 100×15×3 were used. The samples were mechanically treated to give a 2 μm pre-roughness. The prepared samples were fluxed in zinc chloride solution of ZnCl2. In the experiment was used an induction furnace UltraMelt S15 OUPT-016-450-04 company UltraFlex. A furnace equipped with an inert gas (argon) feed system, over the mirror of the melted material to limit oxidation of the metal in the crucible [6]. In the crucible was placed pure zinc [5]. Monitoring the temperature of the molten zinc was performed digital thermometer YF-160A intended to cooperate with the probe attached to the submersible working furnace to 1200°C. The accuracy of this thermometer in the range of 301°C to 1300°C is 1°C.

The objective of quantifying the influence of immersion metallisation parameters, i.e. zincing temperature and immersion time on the hardened layer thickness, was used with a randomized orthogonal experiment plan, taking into account the tri-valentity of the independent variables (inputs) that predicted the execution of nine experiments (Tab. 1) with five repetitions.

Tab. 1. An orthogonal experiment plan involving two triangles independent variables

Sample number	Galvanizing temperature [°C]	Time of immersion [s]
1	440	30
2	440	90
3	440	150
4	460	30
5	460	90
6	460	150
7	480	30
8	480	90
9	480	150

The method of measuring the thickness of the destructive coating was used for the test, which requires prior preparation of the sample (metallographic ink). During the study was used a scanning electron microscope Zeiss EVO MA 15. The device allows you to obtain electron images of 3 nm resolution at 30 kV. The range of possible magnifications is 5 to 1 000 000 times.

3. Results

To evaluate the effects of technological parameters of the bath temperature and the time of metallization (immersion), the following measurements roughness of coating thickness were made. After making 5 point of thickness measurements, we can see that the individual results are close to each other. This shows that the coating is spread evenly over the surface of the steel sample.Tab. 2 shows the results obtained for measuring the thickness of zinc coatings for the individual points of the experiment plan.

Tab. 2. Results of coating thickness measurements taking into account the influence of immersion metallisation process parameters

Sample number	Galvanizing temperature [°C]	Time of immersion [s]	Thickness of the zinc [μm]
1	440	30	71
2	440	90	75
3	440	150	71
4	460	30	70
5	460	90	73
6	460	150	106
7	480	30	105
8	480	90	97
9	480	150	92

Figure 1 and 2 show the relationships between independent variables and dependent variables. These diagrams show that with increasing temperature of the zinc bath and the immersion time of the steel sheet increases the thickness of the zinc coating.

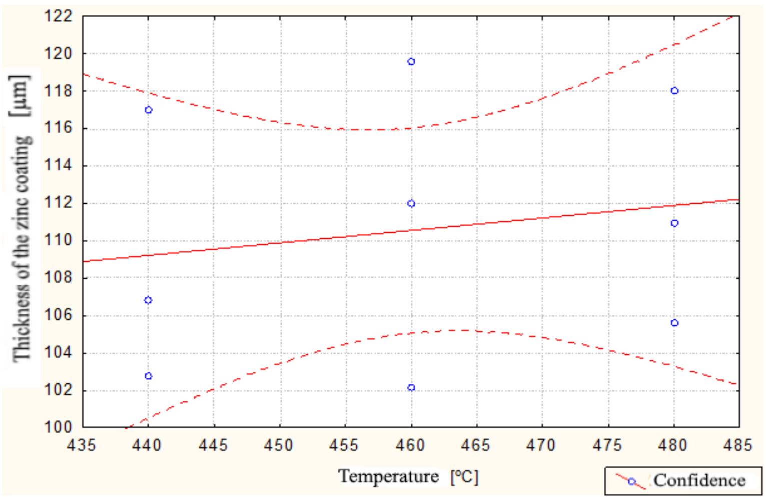

Fig. 1. Influence of temperature dependence on the thickness of zinc coating

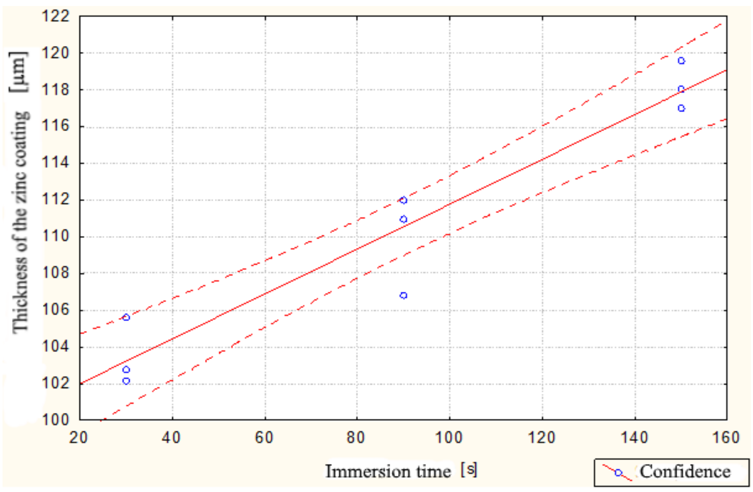

Fig. 2. Influence of time dependence on the thickness of the zinc coating

The purpose of quantitative "evaluation" of influence of input variables on the analysed characteristic dip zinc coatings were used two statistical methods: Mathematical optimization by Taguchi and multiple regression. Optimization was made for the purpose of "the bigger the better" by setting the process parameters to maximize the value of the S/N ratio (ETA) calculated from formula (1):

$$ETA = -10 \log [(1/n)\Sigma(1/y_i^2)] \quad \text{for} \quad i = 1 \text{ to } n, \tag{1}$$

where:
n – number of measurements,
y – value of analysed product characteristics.

Table 3 shows the ETA values for the individual measurements performed according to the planned experiment.

Tab. 3. Factor values ETA according to the planned experiment

Sample number	Galvanizing temperature [°C]	Time of immersion [s]	ETA [–]
1	440	30	39.87
2	440	90	40.22
3	440	150	41.08
4	460	30	40.02
5	460	90	40.70
6	460	150	41.44
7	480	30	40.28
8	480	90	40.54
9	480	150	41.11

Figure 3 shows the mean values of the ETA.

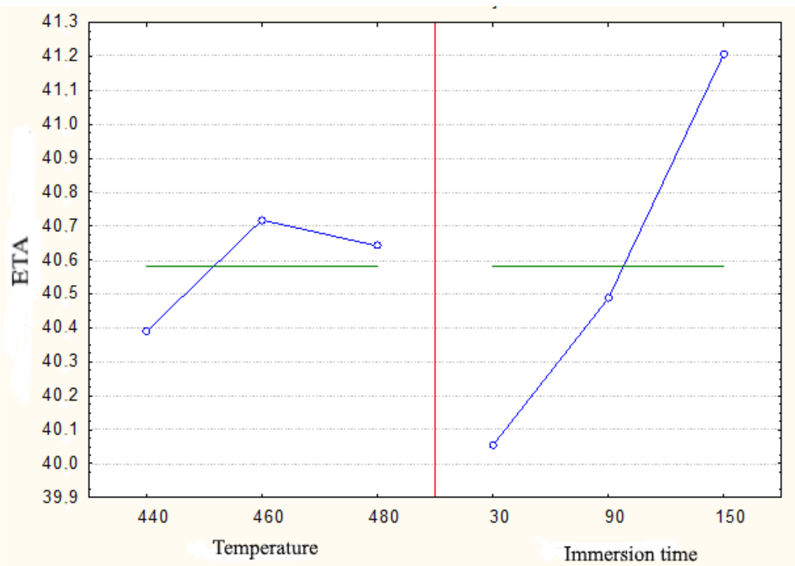

Fig. 3. The average values of the coefficient η

The highest value of the signal (S) to noise (N) was obtained for measurement of six. The value of the ETA was 41.44. On the basis of the average coefficient of ETA, it can be concluded that the largest effect on the maximum value is equal to the immersion time of 150 s. The optimal temperature of the zinc bath, after taking into account the influence of the often non-measurable interfering variable, is 460°C.

Tab. 4. Analysis of variance for the data in Tab. 3

Analysis of mean variances = 40.583 sigma = 2.289					
	SS	df	MS	F	p
Temperature	0.89	2	0.44	0.08	0.92
Time of immersion	10.18	2	5.09	0.93	0.40
Rest	219.62	40	5.50	–	–

The obtained results of the variance analysis (Tab. 4) confirm the earlier observation that the immersion time affects the thickness of the zinc coating more than the bath temperature – the higher value of the squares (SS). It is worrying that much of the variance of the dependent variable it is not explained FOR IN considered independent variables (SS ~ 220). Therefore, considering the result of the F variance test and the calculated significance levels (p) for this analysis, it can be concluded that the variables: temperature and immersion time do not have a statistically significant effect on the thickness of the zinc coating – at a determined significance level $p = 0.05$. The results of this analysis were influenced by large scattering of individual thickness measurement results (Fig. 4).

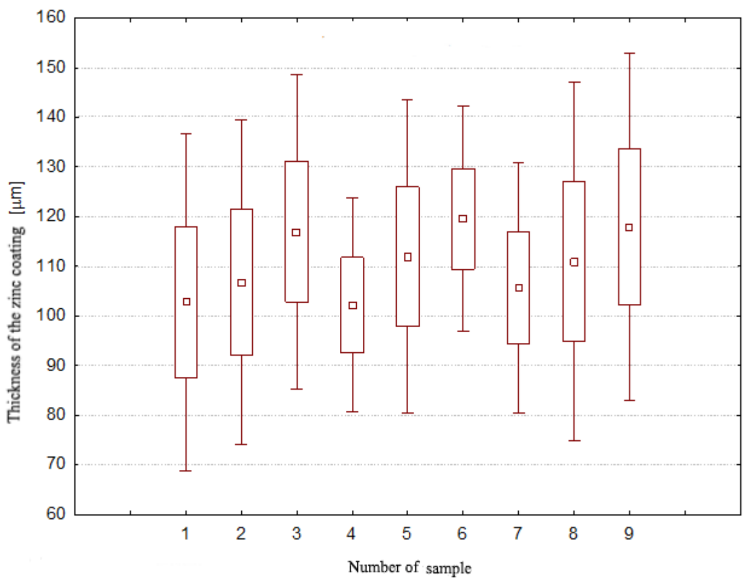

Fig. 4. Thickness measurement with respect to statistical error and standard deviation

In order to predict the thickness of the hardened layer, a multiple regression analysis was used, which the results are given in Tab. 5. The results of the F test, for multiple regression analysis, give the 99% probability of a statistically significant impact of the input variables on the dependent variable. The value of $R = 0.98$ indicates a very high correlation between the adopted input variables and the dependent variable. Considering the values of standardized regression coefficients (BETA) it can be concluded that the immersion time has a greater effect on the thickness of the zinc coating than the bath temperature. Moreover, at the assumed significance level $p = 0.05$ it can be inferred that there is no statistical significance of the variable bath temperature on the value of the parameter being evaluated [5]. This is evidenced by the results of the t-Student test. It can therefore be assumed that this variable can be removed from the model.

In order to verify this hypothesis, the coefficients determining the redundancy of the independent variables of tolerance and the CIW-coefficient of variance inflation (Tab. 6) were also determined, and the test autocorrelation d Durbina-Watson was performed 1.96. The calculated coefficients and the test t do not allow the bath temperature variable to be removed from the statistical model.

Tab. 5. Results of multiple regression analysis

Summary regression dependent variable: The thickness of the coating [µm] R= 0.976 R^2= 0.953 F(2.6)=61.241 p < 0.01, Standard error of estimation: 1.649						
	BETA	Standard error – BETA	B	Standard error – B	t(6)	level p
Free term			68.889	15.533	4.435	0.004
Bath temperature [°C]	0.175	0.088	0.067	0.034	1.980	0.095
Time of immersion [s]	0.961	0.088	0.122	0.011	10.889	0.010

where:
BETA – standardized regression coefficient,
B – regression coefficients,
t(6) – t-Student Significance Test,
p – calculated level of significance.

Tab. 6. Redundancy of independent variables

Redundancy of independent variables: DV: Coating thickness [µm]				
	Tolerance	R-square	Partial correlation	Semi partial correlation
Bath temperature [°C]	1.00	0.00	0.63	0.17
Time of immersion [s]	1.00	0.00	0.97	0.96

The thickness prediction of zinc coatings can be made using the following multiple regression equation (2):

$$\text{Coating thickness} = 0.067 \text{ bath temperature} + 0.122 \text{ immersion time} + 69. \quad (2)$$

Therefore, the predicted coating thickness for optimum parameters (determined by the Taguchi method) calculated from the equation should be 118 µm. Taking into account a 95% probability of estimation error, the expected average zinc coating thickness will be between 116 and 120 µm if the steel sheet S235 is immersed in a zinc bath at 460°C for 150 s (Tab. 7).

Tab. 7. Calculation of variable value: thickness of coating [µm] for optimum conditions

	Weight B	Value	Weight B – Value
Temperature [°C]	0.066	460.000	30.670
Time of immersion [s]	0.122	150.000	18.340
Free term	–	–	68.890
Predicted	–	–	117.890
–95.0%GU	–	–	115.770
+95.0%GU	–	–	120.020

4. Conclusions

Accepted parameters of the process of immersion metallization in the conducted tests Bath temperature 440°C, 460°C, 480°C and galvanizing time: 30 s, 60 s, 150 s have a significant influence on the thickness of zinc coatings. Dipping time has a greater effect on the thickness of the zinc coatings than on the bath temperature. In order to obtain the thickest possible dip zinc coating, while ensuring the minimum influence of confounding factors, the metallization process must be carried out under the following conditions: bath temperature: 460°C, immersion time 150 s. The thickness of the coatings obtained under optimum conditions should be in the range of 116 to 120 μm if the sheet is made of S235 steel and is immersed in a zinc bath at 460°C for 150 s.

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