

RESEARCH ARTICLE



Optimization of Corrosion Behaviour of Blended TiO₂ /ZnO Coating on SUS 304 using Taguchi Method

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Abstract

Objectives: To determine the best combination of blends of TiO₂ /ZnO coated on SUS for obtaining a minimum corrosion rate. **Methods:** Different TiO₂ /ZnO thin film blends are coated on SUS 304 substrates. The sol-gel dip-coating process is used for coating application. Also, withdrawal rates, calcination temperature, and doping percentage are the parameters used for the study of the anti-corrosion property. L9 array, using the Taguchi method, is applied for the design of the experiment. P.G-Lyte 1.0 model was used for the anticorrosion test. The Potentiostatic polarization method was applied for anti-corrosion testing. **Findings:** The results show that the percentage of ZnO doping (60%, 40%, and 20%) is the most significant factor in controlling the corrosion of the sample with plate number 1. The withdrawal rate (0.5 mm/sec, 1 mm/sec, and 1.5 mm/sec) has a minimal impact on the corrosion rate of the coated sample. The calcination temperature (300°C, 400°C, and 500°C) has a greater impact than the withdrawal rate. There is a good agreement between the predicted and actual corrosion current. **Novelty:** Investigation of mechanical properties like corrosion resistance is essential for increasing the durability of automobile components by applying a coating of blends of TiO₂ and ZnO with a rare blending combination that was not applied before for the investigation. In this research, efforts are made to reduce the corrosion rate by applying different blends of TiO₂ and ZnO. Also, withdrawal rate and calcination temperature are varied in the process which was also not considered in the earlier studies.

Keywords: Taguchi method; Corrosion current; Signal to Noise ratio; Coating; Design of experiments

1 Introduction

Steel degradation in corrosive environments is an essential concern in the industries. To handle this problem, it is necessary to understand the interaction between corrosive electrolytes and steel surfaces. Furthermore, the corrosion process will vary depending on the nature and the properties of steel surfaces for example 304 SS, 316 SS, and carbon steel⁽¹⁾.

Many options are available to avoid the corrosion of metals like alloying, painting, etc. But these methods are not always effective, practical or economical. Coating on the contrary is a non-destructive quick fix for many machines and hence is particularly attractive. Therefore, in this paper, the Sol-Gel Dip Coating option is undertaken for study as it is an effective and economical way instead of replacing the whole material.

Investigations of nanomaterial coatings are very important and all automobiles use metallic components that are susceptible to corrosion⁽²⁻⁴⁾. So the attention of the research is focused on the use of nanomaterials for coatings to minimize the corrosion in automobile parts, especially in the exhaust system components.

Several experiments have been conducted to reduce the corrosion rate on a lot of base materials and different methods have been suggested by various scientists to overcome the corrosion problem. Some of the base materials on which the coating is done are low carbon steel, 304L, PMS (Polished Mild Steel), etc.⁽⁵⁻⁸⁾. Glass substrate is also used as the base material⁽⁹⁾.

Zn–Ni–TiO₂ on PMS by electrodeposition showed that TiO₂ contributed more to corrosion resistance⁽⁷⁾. Bio-ZnO and CuO/ ZnO spherical nanofillers on steel showed better anti-corrosion properties⁽¹⁰⁾. Two ZnO nanorods having different surface chemistry were fabricated using hydrothermal methods: at pH6, and high pH (OH[–] rich) to study structural properties. However, in this study, the corrosion rate is not studied⁽¹¹⁾.

TiO₂ and ZnO in different combinations are selected to coat on a metallic substrate. It is easy to control the doping mechanism and film microstructure, and hence the final properties can be achieved easily by the sol-gel method. So, the Sol-gel method was selected for the synthesis of nanoparticles⁽¹²⁾. Different combinations of material coatings were suggested and analyzed by scientists.

Taguchi method can be an effective method for the optimization of different parameters to minimize the corrosion behavior of materials.

Some scientists worked on the Zn–Ni–TiO₂ on PMS, ZnO, Al₂O₃, CuO and TiO₂ on Copper, ZnO on glass TiO₂ and ZnO on A-36, TiO₂/ZnO(0.1 and 1.0%) on 304L for anticorrosion property^(5,7-9).

It is found that the selected combination of blend of TiO₂/ZnO, calcination temperature and withdrawal speed using Taguchi method has not been researched earlier so it was taken for study for the present work.

The research aims to find out the effect of different coating combinations of all the three variables including blends of TiO₂ and ZnO, calcination temperatures and withdrawal rates on estimating the corrosion behavior of SUS 304 using Taguchi method and determining the optimum combination of said variables which will provide the best anticorrosion property.

2 Methodology

2.1 Raw Materials:

Titanium Isopropoxide [Ti(OiPr)₄] (97%), Zinc acetate dehydrate (ACS reagent $\geq 98\%$), Ethyl acetoacetate[EAA], 2-Methoxyethanol (99%) and Mono ethanolamine (99%) all were purchased from Sigma Aldrich. SUS 304 substrate was used as base material. The material composition is shown in Table 1.

Table 1. Chemical composition of SUS 304

C%	Mn%	S%	P%	Si%	Ni%	Cr%	N%
0.060	0.84	0.03	0.043	0.29	0.01	18.13	0.043

2.2 Fabrication of coated sample:

2.2.1 Cutting of the samples:

The steel plate of material SUS 304 of size 500 mm × 500 mm × 2 mm was cut into pieces of size 70 mm × 20 mm × 2 mm. The extra length was kept to hold the workpiece. The substrates are polished by using polish paper.

2.2.2 Ultrasonic Cleaning:

The main purpose of ultrasonic cleaning was removing dirt, dust particles post the polishing of substrate. The samples (substrates) were dipped in ethyl alcohol/ethanol and then kept in ultrasonic cleaner for 10 minutes at room temperature (at 26°C).

2.2.3 Sol preparation:

The TiO₂ films were prepared by sol-gel dip-coating method. Inparticular, Ti(OiPr)₄ was added to EAA at room temperature under continuous stirring. The prepared solution was dissolved in 2-Methoxyethanol and the resulting solution was kept at room temperature for at least 2 hours. After sol preparation Zinc Acetate Dihydrate was mixed with Ti sol. The weight percentage of TiO₂/ZnO was taken by molar ratio. Mono ethanolamine was added to Zinc Acetate Dihydrate. The gel was allowed to mix in Ti sol for about 1 hour by using magnetic stirrer.

2.2.4 Dip coating of substrate:

The dip coating of substrate (SUS 304) was done by automatic dip-coating designed set up. The coating of different blending combinations of Ti and Zn was done on the substrate at different withdrawal speed.

2.2.5 Calcination process:

Muffle furnace was used for heating the substrate a specified temperature. The coated substrates were heated at different temperatures as 300 °C, 400 °C, 500 °C by using muffle furnace.

2.3 Parameters identification and their levels:

Following parameters were decided for the study: Doping Percentage of ZnO (60,40 and 20%), Calcination Temperature (300°C, 400°C and 500°C), Withdrawal rate (0.5,1 and 1.5mm/sec). The selected parameters and levels are shown in Table 2. After deciding parameters and levels, L9 array was finalized. The observation table is shown in Table 3. It indicates the process parameters like Doping Percentage, Calcination Temperature, Withdrawal Speed, S/Ratios (SNR1) as well as corrosion current (I_{corr}).

Table 2. Process parameters and their levels

Level	Doping Percentage	Calcination Temperature	Withdrawal Speed
1	2/3	300°C	0.5 mm/sec
2	3/2	400°C	1 mm/sec
3	4/1	500°C	1.5 mm/sec

Table 3. Observation table

Experiment	Doping Percentage	Calcination Temperature	Tem-perature	Withdrawal Speed	I _{corr} .	SNR1
1	1	1		1	0.19	14.4249
2	1	2		2	0.16	15.9176
3	1	3		3	0.18	14.8945
4	2	1		2	0.22	13.1515
5	2	2		3	0.23	12.7654
6	2	3		1	0.22	13.1515
7	3	1		3	0.26	11.7005
8	3	2		1	0.25	12.0412
9	3	3		2	0.23	12.7654

2.4 Potentiosatic Polarization Test:

This test was carried out to measure the corrosion rate of bare and coated samples. P.G-Lyte 1.0 model was used for the anticorrosion test. Three electrode cells were set up, comprising of one Platinum electrode as counter electrode, one Ag/AgCl

electrode as reference electrode and the Substrate as working electrode. Swipe rate was 50 mV/sec. The corrosion performance of coated and bare samples was determined by dipping the three electrodes in 3.5 wt.% of NaCl solution⁽¹³⁾ and by doing the proper set up of electronic connections.

3 Results and Discussion

Analysis of mean value and S/N Ratio:

The thin film coating of blended TiO₂/ZnO was studied using L9 orthogonal array in Taguchi technique^(14,15). Corrosion current was evaluated from Tafel tests. The Design of Experiment (DoE) and the analysis were conducted by using Minitab 21 statistical software. The influence of doping percentage, withdrawal rate, calcination temperature on the corrosion characteristic (corrosion current) of coated substrate in 3.5% wt. NaCl solution was evaluated⁽¹⁶⁾. Table 4 shows analysis of variance for the corrosion current against three factors varied at three levels.

Table 4. Analysis of Variance

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Doping percentage	2	0.007622	88.40%	0.007622	0.003811	343.00	0.003
Calcination Temperature	2	0.000289	3.35%	0.000289	0.000144	13.00	0.071
Withdrawal speed	2	0.000689	7.99%	0.000689	0.000344	31.00	0.031
Error	2	0.000022	0.26%	0.000022	0.000011	–	–
Total	8	0.008622	100.00%	–	–	–	–

The factors having P-values less than 0.05 were taken as statistically significant towards calculation of performance measures. Anova was applied to find the significance for control factors. As S/N ratio is the ratio of the expected response to the unwanted response, higher the value of S/N ratio better is the response. The main effect plot for mean is shown in Figure 1. The S/N ratio plot is shown in Figure 2. S/N ratio was calculated using smaller is better criterion and is given by:

$$\frac{S}{N} = -10 \times \log \left(\frac{\sum Y^2}{n} \right)$$

Where,

Y- Responses for the given factor level combination,

n - Number of responses in the factor level combination.

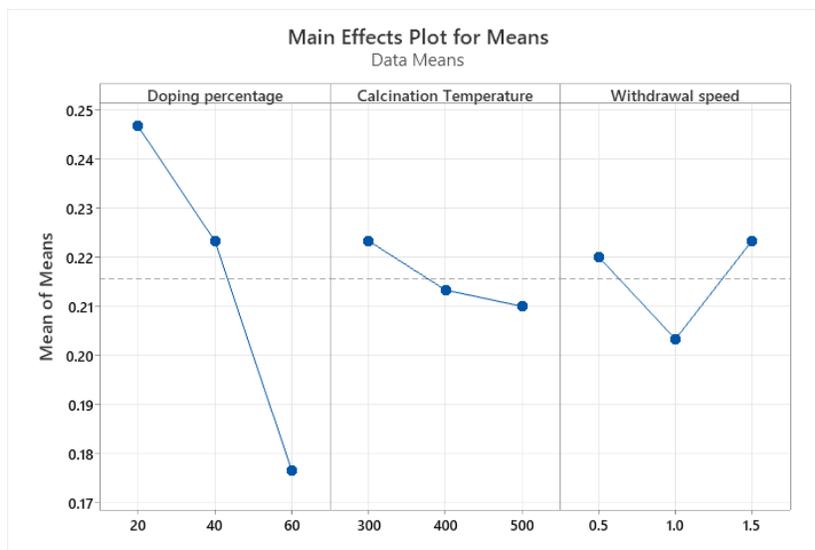


Fig 1. Main effect plot for means

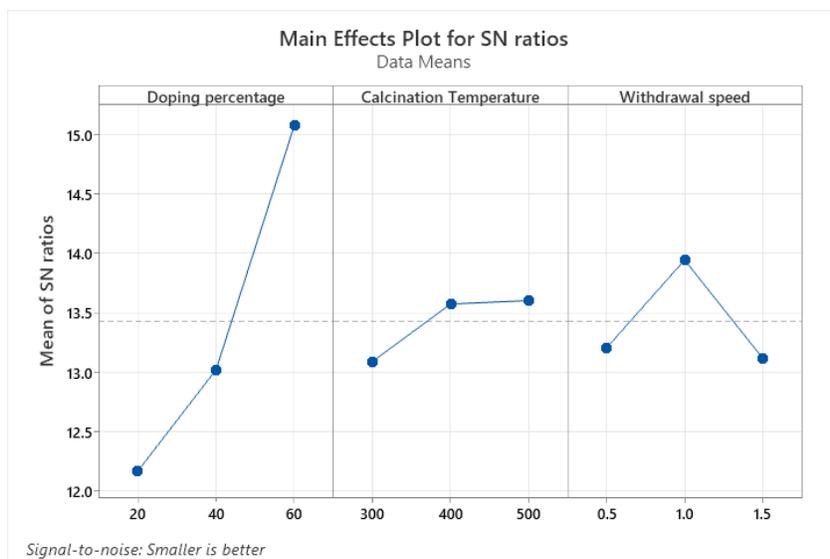


Fig 2. Main effect plot for S/N ratios

From Figures 1 and 2, it is observed that 60 percent of Zn indicating level 1, level 3 of calcination temperature that is 500°C and level 2 of withdrawal speed (1 mm/sec.) had the maximum S/N ratio values and were the optimized parameters. The results in Table 4 indicates that, doping percent is the most significant factor, whereas withdrawal speed is more significant than calcination temperature. The combination of factors considered in this work have not been studied before. Upon further exploration of this technique, the analysis can yield different corrosion current values if the testing is carried out in other electrolyte solutions.

4 Conclusion

The literature review suggested that there is no research work done to find out the properties of TiO₂/ZnO coating considering doping percentage, calcination temperature, withdrawal speed on SUS 304 respectively as the parameters. The impact of study is showcased by comparing the final output against bare sample of SUS 304.

The electrochemical corrosion testing confirmed the enhanced corrosion resistance of blended TiO₂/ZnO coatings on SUS304 at 60 percent ZnO doping, withdrawal speed of 1 mm/sec and calcination temperature at 500°C. The optimized coating had the minimum corrosion current of 0.14 μ A which is higher than any combination of the coated materials and bare sample as discussed in this paper. By visual inspection too it was further reinforced as the sample was in sound condition. Use of the optimized combination of blended TiO₂/ZnO on SUS304, fabricated by Sol-Gel method is an economical and recommended solution to increase the life of the automobile components by effectively preventing corrosion.

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