# Corrosion Behaviourof Sol-Gel Derived Nano-Alumina Film

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**Abstract-** In this paper, the effect of nano-alumina coating on the corrosion behavior of IS 2062 low carbon steel was investigated. The coating was achieved by sol-gel dip coating method. The coated specimens were subjected to Neutral Salt Spray (NSS) technique and the corrosion resistance was measured in terms of the corrosion time. The experiments were designed using the Taguchi methodology of Design of Experiments (DoE). Results revealed 80% increase in the corrosion time for the coated specimen compared to the uncoated one. Statistical analysis revealed that dipping time was the most influential in increasing the corrosion time followed by sintering temperature and molar concentration.

**Index Terms**: corrosion, nano-alumina, sol-gel, Neutral Salt Spray, corrosion time, Taguchi method, DoE,

## 1. INTRODUCTION

Corrosion is a natural process, which converts a refined metal to a more stable form, such as its oxide or hydroxide. It is the gradual destruction of materials (usually metals) by chemical reaction with their environment causing the impairment of the metal function .Corrosion resistance is the ability of a metal to maintain operational capability in a given corrosion system. . To protect a metal surface from corrosion, it is isolated from the corrosive medium by the application protective layers. There are various corrosion protection measures available in practice. These can be classified into two broad categories. Metalliccoatingand nonmetallic coating.In metallic coating, the metal surface is coated with a layer of other metal or its oxide which may be either morenoble than the structure or less noble than it. . In practice metallic coating can be carried out by different methods such as electro plating, hot dipping, thermal spraying, sol-gel dip coating the sol-gel process may be described as the formation of an oxide network through poly condensation reactions of a molecular precursor in a liquid. A sol is a stable dispersion of colloidal particles or polymers in a solvent. A gel consists of a three dimensional continuous network, which encloses a liquid phase. The idea behind sol-gel synthesis is to “dissolve” the compound in a liquid in order to bring it back as a solid in a controlled manner. Multi component compounds may be prepared with a controlled stoichiometry by mixing sols of different compounds. The sol-gel method prevents the problems with co-precipitation, which may be inhomogeneous, in a gelation reaction. It enables mixing at an atomic level, results in small particles which are easily sinterable.In the sol-gel dip coating process, the deposition proceeds in a series of steps which starts with the dipping and withdrawal of a substrate from a fluid sol. This is trailed by gravitational draining and solvent evaporation accompanied by further condensation reactions, resulting in the deposition of a solid film.With the advent of Nano technology, Nano materials, Nano films and Nano fluids are finding its applications in various spheres of mechanical engineering. Nanostructured materials are those materials having an average particle size of 1–100 nm known for their outstanding mechanical and physical properties due to their extremely fine grain size. Nano sized materials can be used for corrosion protection also ***[2, 3]***. There are several methods to achieve a Nano film coating over a substrate metal. Among these, the sol-gel dip coating is the most economical and efficient method to achieve a thin film coating ***[4, 5,6]***. Though, several references reporting the corrosion protection of mild steel using other methods like thermal spraying and hot dipped galvanizing are available ***(7,8)***, those relating to sol-gel dip coating technique are scanty.

Alumina ($Al_2O_3$) is one among the various metal oxides that can be used for corrosion protection. Different processes for preparing microcrystalline coatings can be used to produce Nano-structured coatings by modifying the process parameters. Its inherent corrosion protection property might be improved in the Nano scale.

In the sol gel synthesis of alumina, since the property of the alumina thin film depends on the various coating process parameters such as molar concentration of the sol, dipping time and sintering temperature, a multivariable study could only furnish a thorough knowledge on the deposition of thin films and not a single variable-at-a-time study. In multivariate problems, a renowned statistical technique called design of experiments (DoE) is employed. Several methods such as full factorial design, response surface design and Taguchi methods are the commonly used DoE techniques. Among these techniques, Taguchi methodology is found effective in determining the influence of process parameters by conducting least number of experiments. Though there are many available references reporting the use of Taguchi method on various applications like milling ***[9]***, turning ***[9]***, and drilling ***[10]*** those relating to the sol gel technique and corrosion protection were limited.In this work, the effect of Nano-alumina film on the corrosion resistance of IS 2062 low carbon steel specimen was investigated experimentally. The coating was achieved using sol-gel dip coating method. The main objective of the present study was to identify a combination of coating process parameters which could yield higher corrosion resistance using the Taguchi methodology of DoE. The second objective was to investigate the influence of each coating process parameter on the corrosion resistance.

## 2. MATERIALS AND METHODS

### 2.1Taguchi Method

Taguchi method is a robust technique for identifying and optimizing those process parameters having a significant effect on the performance variation. The primary advantages of this method include:

a. Use of orthogonal arrays to organize the parameters affecting the processes by varying their levels.

b. Testing a limited collection of parameter combinations instead of checking all possible combinations like factorial design.

c. Determining factors which most affect the output response using minimum number of experiments, thus saving time and resources.

Taguchi defines a loss function which can be used to calculate the deviation of the experimental value from the desired value. Those factors which cause the deviation of product functional characteristics from their target values are called noise factors. These factors are uncontrollable and could be classified as external factors (e.g. human errors, temperatures) or manufacturing process distortions (e.g. variation of process parameters, product deterioration). The overall aim of Taguchi method is to identify the process parameters that are robust with respect to all noise factors.

Taguchi transforms the loss function into a signal-to-noise (S/N) ratio, η, whose analysis consist of three categories of quality characteristics, i.e. the lower the better, the higher the better, and the nominal the better. Irrespective of the category of performance characteristic, a larger S/N ratio implies better performance characteristic. The following equations were employed for calculating S/N ratio:

Larger the better (Maximize corrosion time)

$$\eta = -10\log[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i}] \quad (1)$$

Smaller the better (Minimize corrosion time)

$$\eta = -10\log[\frac{1}{n}\sum_{i=1}^{n}y_i] \quad (1.2)$$

Where, $\eta$ is the S/N ratio, $y_i$ is the observed response value for the $i^{th}$ repetition and n is the number of replications.

In this work, the corrosion resistance was measured based on the time taken for corrosion. Since, a higher value of time was desired, the equation for larger the better S/N ratio was used in present study. The mean S/N ratio for each level of process parameters were calculated using the above equations and the one having the highest S/N ratio was identified as the optimum level. To determine the influence of each process parameter on the output response, the difference between its maximum and the minimum S/N ratio was found out. The one with the highest difference is expected to have the highest influence on the response and vice versa.

The Analysis of Variance (ANOVA**) [11]** was carried out by dividing the total variability of S/N ratios into contributions by each process parameter and the error. The following equations were used to find the total sum of the squared deviations $(SS)_T$ and the sum of squared deviation due to each process parameter $(SS)_P$:

$$(SS)_T = \sum_{i=1}^{m} \eta_i^2 - m\eta_m{}^2 \qquad (1.3)$$

$$(SS)_P = \sum_{j=1}^{t} \frac{(s\eta_j)^2}{t} - \frac{1}{m}\left[\sum_{i=1}^{m} \eta_i\right]^2 \qquad (1.4)$$

where $m$ represents the number of experiments in an orthogonal array, $\eta_i$ the mean S/N ratio for the $i^{th}$ experiment, $\eta_m$ the total mean of S/N ratio, $j$ the level number of the process parameter $p$, $t$ the repetition of each level of the parameter $p$ and $s\eta_j$ the sum of the S/N ratio involving the parameter $p$ at level $j$.

The sum of squared deviation due to error $(SS)_e$ was calculated from the difference of the total sum of the squared deviations $(SS)_T$ and the sum of the sum of squared deviation due to each process parameter as shown below:

$$(SS)_e = (SS)_T - \sum_{p=1}^{3} (SS)_P \qquad (1.5)$$

From $(SS)_P$ and $(SS)_e$, the variance of process parameters ($V_P$) and error ($V_e$) were calculated as follows:

$$V_P = \frac{(SS)_P}{(df)_P} \qquad (1.6)$$

$$V_p = \frac{(SS)_e}{(df)_e} \qquad (1.7)$$

Where $(df)_P$ and $(df)_e$ are the respective degrees of freedom of the process parameter and the error given by (t – 1). The corrected sum of squares $(\hat{S})_P$ was calculated as per the following equation:

$$(\hat{S})_P = (SS)_P - (df)_p V_e \qquad (1.8)$$

Finally, the percentage contribution $\rho$ of each process parameter on the corrosion resistance properties of the deposit was found out using the following relation:

$$\rho = \frac{(\hat{S})_P}{(SS)_T} \qquad (1.9)$$

## 2.2Experimental Design

The first step in the design of experiments by Taguchi method is the selection of process parameters that likely influence the properties of Nano alumina film. The molar concentration of the alumina sol, dipping time and sintering temperature were the process parameters considered in this study. Subsequent to preliminary experimental trials in our laboratory, three levels of variation of each parameter were identified as shown in Table 1.

TABLE 1

Levels of Process Parameter

| Parameters | Unit | Level I | Level II | Level III |
|---|---|---|---|---|
| Molar Concentration | M | 0.01 | 0.02 | 0.03 |
| Dipping Time | min | 60 | 120 | 180 |
| Sintering Temperature | ºC | 300 | 400 | 500 |

The experiments were designed based on the L9 orthogonal array (9 tests, 3 variables, 3 levels) as shown in Table 2. Each row in this matrix represented one experimental run and the sequence of executing these runs was randomized.

TABLE1

L9 Orthogonal Array

| Run | Molar Conc (M) | Dipping Time (min) | Sintering Temperature (ºC) |
|---|---|---|---|
| 1 | 0.01 | 60 | 300 |
| 2 | 0.01 | 120 | 400 |
| 3 | 0.01 | 180 | 500 |
| 4 | 0.02 | 60 | 400 |
| 5 | 0.02 | 120 | 500 |
| 6 | 0.02 | 180 | 300 |
| 7 | 0.03 | 60 | 500 |
| 8 | 0.03 | 120 | 300 |
| 9 | 0.03 | 180 | 400 |

Since the design was orthogonal, the mean effect of each process parameter on the output response could be calculated by averaging the response corresponding to its each level. For example the mean effect of 0.01 M molar concentration on the coating can be found out by averaging the responses corresponding to run 1, 2 and 3 and so on.

## 2.3Deposition of Nano alumina film

Alumina film was deposited on IS 2062 low carbon steel plates of size 10 mm x 10 mm x 10 mm. The composition of the substrate is presented in Table 3.

TABLE 2

CHEMICAL COMPOSITION OF THE SUBSTRATE METAL

The substrates were polished with different grades of emery paper followed by cleaning with acetone

| Fe (%) | Mn (%) | Si (%) | P (%) | C (%) | S (%) | CE (%) |
|---|---|---|---|---|---|---|
| 95.99 | 1.50 | 0. 40 | 0.05 | 0.23 | 0.05 | 0.42 |

and distilled water. Fig.1. shows the flow chart for the preparation of the Nano alumina film. Aluminum Iso-propoxide (98% pure, Tokyo industries) and distilled water were mixed in a suitable molar ratio (0.01 M, 0.02 M and 0.03 M). While stirring, 1 M nitric acid was continuously added to the solution until its pH attained an approximate value of 4. The solution was then heated up to 80ºC under continuous stirring. The resultant transparent boehmite sol was allowed to cool down to room temperature. The substrates were dipped in the final sol for a given time (60 min, 120 min and 180 min). The sol-gel coated specimens were air dried at room temperature for 1 hr, followed by sintering at the required temperature (300 ºC ,400 ºC and 500 ºC ) for 1 hr, to ensure homogenous Nano-alumina coating ***[6,12,13,14,15]***.

The presence of alumina in the coated surface was confirmed by subjecting it to X-Ray Diffraction (XRD) analysis using a computer controlled X-Ray Diffract meter (Make RigakuUltima III and Model) under Cu-K$\alpha$ radiation ($\lambda$ = 1.54 A$^0$).

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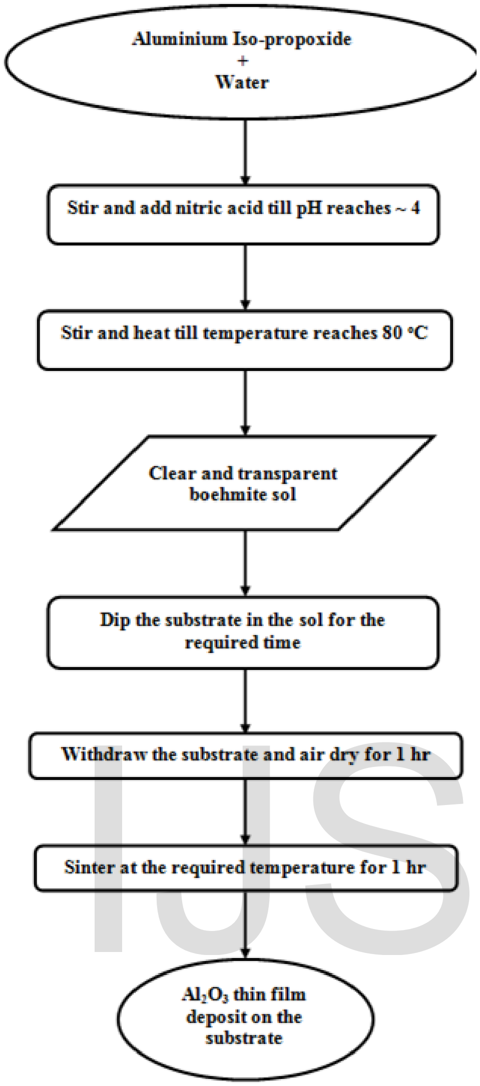


Fig 1: Flow chart showing the preparation of Nano alumina film from aluminum isopropoxide

## 2.4Corrosion Testing

The corrosion testing was carried out using the Neutral Salt Spray (NSS) technique as per the ISO 9227 standard **[16].**Salt spray tests are generally suitable as corrosion protection tests for rapid analysis for discontinuities, pores and damage in organic and inorganic coatings. In addition, for quality control purposes, comparison can be made between specimens coated with the same coating. As comparative tests, however, salt spray tests are only suitable if the coatings are sufficiently similar in nature. The device for spraying the salt solution comprises a supply of clean air, of controlled pressure and humidity, a reservoir to contain the solution to be sprayed, and one or more atomizers. The compressed air supplied to the atomizers shall be passed through a filter to remove all traces of oil or solid matter, and the atomizing pressure shall be at an overpressure of 70 kPa 4) to 170 kPa. In order to prevent evaporation of water from the sprayed droplets, the air shall be humidified before entering the atomizer, by passage through a saturation tower containing hot distilled water or deionized water at a temperature 10 °C above that of the cabinet. The appropriate temperature depends on the pressure used and on the type of atomizer nozzle and shall be adjusted so that the rate of collection of spray in the cabinet, and the concentration of the collected spray, are kept within the specified limits .The level of the water shall be maintained automatically to ensure adequate humidification. The following figure shows the salt spray testing apparatus.




Fig 2: Salt spray testing equipment

## 3. RESULTS AND DISCUSSION

Fig. 3. Shows the X-Ray Diffraction pattern of the coated specimen. The peaks obtained at 2θ values of $35.50^{\circ}$, $43.08^{\circ}$, $57.08^{\circ}$ and $62.55^{\circ}$ along (1 0 4), (1 1 3), (1 1 6) and (0 1 8) planes (JCPDS 85-1337) confirmed the presence of alumina.

TABLE 3**OPERATING CONDITION**

| Sl no. | Parameter | Actual |
|---|---|---|
| 1 | Salt concentration | 5% by weight |
| 2 | Chamber temperature | 35-38 ºC |
| 3 | Volume fog solution (avg of 4hrs) | 1.8 ml/hr |
| 4 | Test duration | 2 hrs |

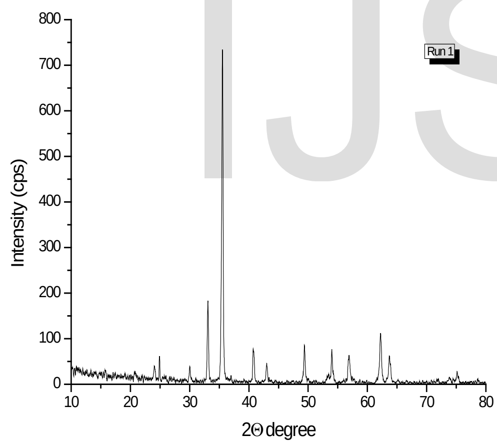


Fig 3:X-Ray Diffraction pattern of the coated specimen

The corrosion time measured for the uncoated specimen was 1 hr. Table 5 shows the corrosion time measured for the coated specimens corresponding to all experimental runs.

TABLE 4

Measured Corrosion Time for Each Experimental Run

| Run | Molar concentration | Dipping time (min) | Temperature (ºc) | Corrosion time (hrs) |
|---|---|---|---|---|
| 1 | 0.01 | 60 | 300 | 3 |
| 2 | 0.01 | 120 | 400 | 2 |
| 3 | 0.01 | 180 | 500 | 6 |
| 4 | 0.02 | 60 | 400 | 1.5 |
| 5 | 0.02 | 120 | 500 | 7 |
| 6 | 0.02 | 180 | 300 | 8 |
| 7 | 0.03 | 60 | 500 | 1.25 |
| 8 | 0.03 | 120 | 300 | 4 |
| 9 | 0.03 | 180 | 400 | 7.5 |

A close examination of Table 5, interestingly reveals variations in corrosion time with variations in the process parameters. Results indicated that nano-alumina coated specimens had better corrosion resistance than the uncoated specimen. The highest corrosion time and thus the highest corrosion resistance was noted for run 6 corresponding to a molar concentration of 0.02 M, dipping time of 180 min and sintering temperature of 300ºC, followed by runs 9 and 5. Fig. 4 & 5 shows the coated specimen corresponding to run 6 before and after corrosion.

Fig 3: Coated specimen before rust formation

**Fig4:** Coated specimen after rust formation

The above analysis confirmed that the coating process parameters are vital for enhanced corrosion resistance. A detailed discussion on the effect of each coating process parameter on the corrosion time is discussed in section 3.1.

## 3.1.Effect of process parameters

The main effects of each process parameter on the corrosion resistance are illustrated in Figs.6-8.

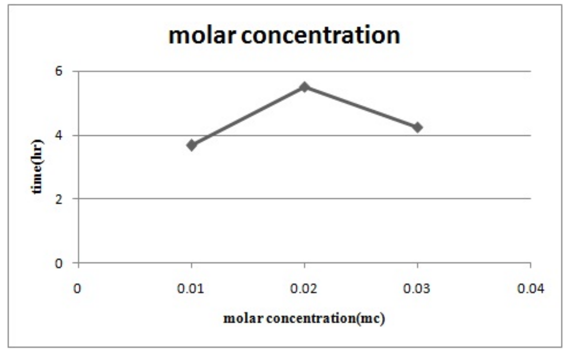


Fig5**:**Effect of molar concentration of the alumina sol on the corrosion time

The effect of molar concentration of the sol on the corrosion time is shown in Fig.6. It is seen that the corrosion time increased with an increase in molar concentration up to a median level (level II) and decreased thereafter.

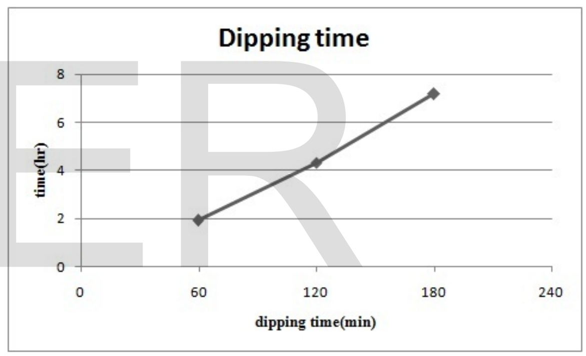


Fig6**:**Effect of dipping time on the corrosion time

The effect of dipping time on the corrosion time shown in Fig. 7 revealed that the corrosion time increased with the dipping time

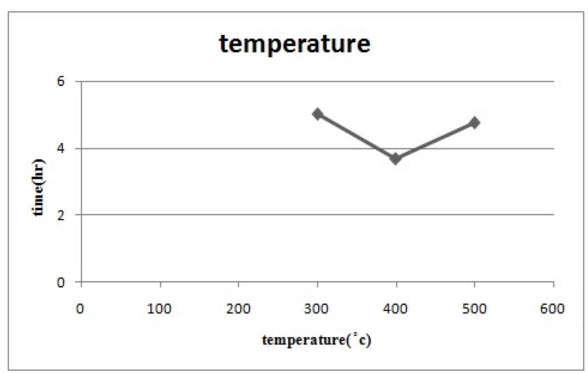


Fig7: Effect of sintering temperature on the corrosion time

It is seen from Fig. 8. that the corrosion time decreased with an increase in the sintering temperature upto a medium value (400°C) and then increased.

## 3.2. Statistical analysis

Statistical analysis of the corrosion time was carried out in order to find out the optimum parameter settings and analyze the contribution of each factor to the output response. This was accomplished by calculating the signal to noise (S/N) ratio followed by analysis of variance (ANOVA).**[15]**

The S/N ratio calculated for the relative emission rate and the mean S/N ratio at three levels of process parameters are presented in Table 6.

TABLE 5

S/N Ratios for Corrosion Time

| Factor | Level I | Level II | Level III | Max-Min |
|---|---|---|---|---|
| **M** | 15.05 | 15.11 | 14.09 | 1.02 |
| **D** | 11.57 | 14.85 | 17.83 | 6.26 |
| **S** | 16.21 | 13.79 | 14.25 | 2.42 |

Regardless of the type of performance characteristics desired, i.e. higher the better, lower the better or nominal the better, a large S/N ratio implied better performance characteristics. Thus, the highest S/N ratio yields the most optimal level of the process parameters. Hence it was inferred from the table that a medium molar concentration, a higher dipping time and a lower sintering temperature were favorable in increasing the corrosion time. The difference between the maximum and minimum S/N ratio values for each process parameter was noted. The highest value was for dipping time implying its highest influence on corrosion time and the lowest value was for the molar concentration of the sol showing that it is least influential.

The ANOVA result for the output response is presented in Table 7 from where contribution of each process parameter on the corrosion time can be observed.

TABLE 6

RESULTS OF ANOVA

| | Source | Df | SS | V | Ŝ | % Contribution |
|---|---|---|---|---|---|---|
| Corrosion time | M | 2 | 0.2184 | 0.1092 | 0.2071 | 2.60 |
| | D | 2 | 6.5362 | 3.2681 | 6.52 | 82.9 |
| | S | 2 | 1.1010 | 0.5505 | 1.089 | 13.8 |
| | Error | 2 | 0.0112 | 0.0056 | | |
| | Total | 6 | 7.867 | | | |

From this we observe that the dipping time is the parameter which has the maximum contribution to corrosion resistance out of the three parameters-dipping time, molar concentration and sintering temperature. It has about 82.9% contribution towards corrosion resistance. It proves that as dipping time increases, corrosion resistance of coated specimen also increases.

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enduring support which is inevitable for success of our future.

## 5. CONCLUSIONS

This paper presented the implementation of Taguchi method in the selection of process parameter combinations for achieving higher corrosion resistance for a sol-gel derived nano alumina deposit. The presence of alumina in the coated specimen was experimentally identified through XRD analysis. The following conclusions were drawn based on the experimental results of this study:

a. Coated specimens exhibited around 80% increase in the corrosion time compared to the uncoated one.
b. A combination of average value of molar concentration (0.02 M) and lower sintering temperature (300$^{o}$C) and a higher value of dipping time (180 min) were favorable in increasing corrosion resistance.
c. Dipping time of the alumina sol contributed 82.9% towards increasing the corrosion resistance while sintering temperature and molar concentration contributed only 13.8 % and 2.60 % respectively.
d. The confirmation test results at optimal conditions were in good agreement with the prediction of the Taguchi design approach.

The methodology implemented in this study can be readily extended in the future to other coating techniques and for various applications.

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