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Statistical Analysis of Electroless Nickel Coating on Carbon Fiber

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Abstract

The objective of the paper is to investigate the effect of coating parameters such as time of sensitization, time of activation and time of metallization on the thickness of the coating being deposited on the carbon fiber and optimizing the coating parameters by making use of L_{27} Taguchi orthogonal design. Carbon and coated carbon fiber are used as reinforcement in Aluminum 7075 alloy. Coating the fiber with metallic element nickel has been helpful in acting as a barrier between the interface and preventing the undesirable chemical reaction which disturbs the mechanical properties of the metal matrix composites. An L is used to obtain three level of factors and their interfacial effects. From the Taguchi methodology, the most favorable parameter for coating was found. In addition, Analysis of Variance (ANOVA) indicated time of metallization has major influence on coating thickness followed by time of activation.

Keywords: Taguchi methodology, Carbon fiber, Electroless coating.

INTRODUCTION

The outcome of the fabricated compositemainly depends upon the interface between the two constituents present in the composite. Proper modification of the constituents is required or else it will lead to reaction of undesirableproduct with degraded mechanical property. One of the approaches of obtaining a defect free product is by depositing layer of metallic element on fiber. Various coating methodologies using various metallic elements has been studied by researchers [1-2]. Application of metallic layer on the surface of the fiber has helped in improving wettability and avoiding the chemical reaction. Electroless nickel coating of the fiber is an easy, cost effective and time dependent process of employing a coat around the fiber. A pursuit is made in the present experiment to know the effect of parameters of coating such as time of sensitization, time of activation and time of metallization on the coating morphology of the fiber. There are numerous parameter affecting the coating processsuch as temperature and percentage of hydrogen of the bath. In this study only time for sensitization, activation and metallization is considered for optimizing the coating process. The objective of this experiment is to study the interaction of the coating parameter on the morphology of the coat and to modify the process based on L_{27} Taguchi orthogonal design. This requires conduction of series of experiments. Such an approach is quite costly and time eater process. Firstly, one of the Design Of Experimental technique used here is Taguchi method which is useful in cutting off the experiments to be carried out and in tailoring the coating parameter [4]. Secondly, identification of the coating parameter which has the huge influence over the coating process is done by implementing Analysis Of Variance method.Finally, verification and computation of the optimal condition obtained through Taguchi orthogonal array design is carried out through a test and the modification in the coating performance characteristics at the optimal condition is compared to the initial condition.

DESIGN OF EXPERIMENT (DOE)

The technique is useful to study effect of multiple variables separately. The technique is effective to optimize the product and process design, to study effect of multiple variables on the performance and solve production related problems by conducting experiments. DOE helps to study the influence of individual parameter on the performance and to determine which parameter affects the most and which one has lesser influence. It also tells which parameter should have tighter tolerance and which one should be relaxed. It involves series of experimental steps and follows a proper sequence to yield better understanding of performance. It provides certain no of combination of factors and levels for the test to be carried to obtain required variations. Taguchi approach deals with assignment of factors in orthogonal array and obtains test combinations. Analysis of variance makes use of signal to noise ratio to obtain best process design. Combined effect of parameters can be analysed and correlating term can be obtained.

Taguchi Technique

It is effective method to deal with the responses obtained influenced by multiple parameters. It reduces no of experimental runs that are required to model response function. Advantage is to determine the possible interaction between parameters. It is devised for process optimizing and identification of combination of factor for a given response. It consists of 3 phases

- Planning phase
- Conduction phase

- Analysis phase

The planning phase is the most important phase of the experiment as it creates a standard orthogonal array to accommodate the effect of several factors on the target value and define the plan of experiments [10]. The experimental results are analysed using the analysis of means and variance to study the influence of the factors.

EXPERIMENTAL WORK

Materials and Method

Materials used were PAN based carbon fibre having diameter of 5.7µm.Electroless method of coating was carried out using nickel sulphate as the coating material andsodium hypo phosphate as a reducing agent. Table 1 shows properties of PAN based carbon fiber [2].

Table 1. Properties of PAN based carbon fibers

Property	Commercial, standard modulus
Tensile modulus	228 (GPa)
Tensile strength	3800 (MPa)
Elongation at break	1.6%
Electrical resistivity	1650 (µΩcm)
Thermal conductivity	20 (W/mK)
Density	1.8 (g/cc)
Carbon content	95%
Filament diameter	6-8(µm)

Pre-Procedure

The continuous carbon fibres of average diameter 5.7 µm were chopped down to short fibers of about 1 to 2 mm length using printing press cutting machine. The process begins with the treatment of fibres in a muffle furnace for 10 min. at 450°C to remove the pyrolytic coatings around fibres[5-6]. The Electroless process of depositing the nickel coatings on the carbon fibre, follows sequence of sensitizing, activation and metallization, with cleaning, rinsing, washing and drying stages carried out in between the process.

Experimental Procedure

The short carbon fiber was cleaned in distilled water and dried at 85°C. The sizing and finishing treatment of the surface of the fibers, prior to coating, was done by heating them to about 450°C for 10 min. in air. The coating procedure consist of three stages, namely sensitization, activation and metallization [7]. The pre heated fibers were first treated with acetic acid to clean the surface, and then activated using stannous chloride ($SnCl_2$). They were sensitized for different times (5, 10 & 15 min.)

under continuous stirring. Fibers were filtered and cleaned with distilled water. In order to have catalytic surfaces, the sensitized fibers were exposed to an aqueous solution of palladium chloride ($PdCl_2$) and HCl under ultrasonic agitation. This process, called activation, produces the formation of Pd sites on the fiber surface, which allows the subsequent metallization with nickel.

Metallization starts with the treatment of fibers in an open oven for 10 min. at 500°C to eliminate the pyrolytic coatings around fiber. Metallization is produced by immersion of activated fibers into a solution containing $NiSO_4$. $5H_2O$ as metal ion sources also held under agitation. The coatings obtained at different metallization times were then studied by SEM and the thickness of the nickel layer was determined in transversal cross section. The surfacecharacteristics of uncoated fibers and nickel coated ones using scanning electron microscope. These fibres were observed with EVO MA18 with Oxford EDS having a resolution of 100KX. The depth of focus was at a magnification of 1000x, aperture size of 100 microns and working distance of 10 mm. The morphology of original carbon fiber and coated carbon fiber was observed using a field emission scanning electron microscope (SEM) measurement were made with a EVO MA18 with Oxford EDS(X-act l with a magnification capacity of 500×, 1000×, 2000× and accelerating voltage of 20 KV with working distance (WD) 10 mm. Microscopes were equipped with analytical facilities (energy dispersive X-ray spectroscopy-EDS). The attached EDS detector helps in quantifying the elements present in the sample. Taguchi S/N ratio was used to find the effect of time for sensitization, time for activation & time for metallization on the fiber. 'Nominal the best type' characteristics was selected to analyze the effect of coating parameter.

Plan of the experiment

Traditional experimental design are costly and time consuming. Process requires large amount of tests to be carried out. Taguchi methodology makes use of an orthogonal array to study the entire process and with less amount of experimental runs. Traditional process studies one factor at a time, by changing one parameter while keeping rest of the parameter constant. Therefore it fails to consider interaction between the parameters. This traditional methods fails to study all the factors in the process and to determine their individual effects in single experiment. Taguchi technique is used instead of old method which is helpful in optimizing coating parameters and identifying the optimal combination of factors for the desired responses [10].

Steps involved in the process:

- Identifying response functions and the process parameters
- Determination of the number of levels for the process parameters and possible interaction between them.
- Selection of the appropriate orthogonal array.
- Selection of the optimum level of process parameters by Analysis of Variance [ANOVA].
- Performing a confirmation test to verify the optimal process parameters.

Standard orthogonal array was used to conduct the experiment. In the present investigation, an L_{27} orthogonal array was chosen, which has 27 rows and 13 columns.

The input parameters are Sensitization time in minutes, Activation time in minutes and Metallization time in minutes. Table 2 indicates the factors and their levels. Trails were conducted by varying one of the parameter and keeping the other parameters constant. The experiment consisted of 27 tests (each row in L_{27} orthogonal array) column indicates the parameters[10].

Table 2. Coating parameters and their values at three levels

Level	Sensitization (min)	Activation (min)	Metallization (min)
1	5	5	1
2	10	10	2
3	15	15	3

RESULT AND DISCUSSION

Statistical Analysis

The parameters effecting the coating thickness was subjected to statistical analysis with response table for means by making using of Minitab 16 software tool. The experimental results of L_{27} orthogonal array is shown in the table 3. The experimental results were analysed to study the effect of interaction between level 1, level 2 and level 3 of the coating parameters in the experiment. In the present work, three runs were carried out for each of the nine experiments. The main objective of the present work is to optimize Sensitization time, Activation time and Metallization time to achieve the desired coating thickness. Taguchi analysis is performed based on "nominal the best" characteristic to analyse the effect of coating parameter on thickness.

Table 3. Experimental results showing mean thickness of coating

Exp No	Time for Sensitization (TS) min	Time of Activation (TA)min	Time for Metallization (TM)min	Mean thickness of the coat(μm)
1	5	5	1	0.03807
2	5	5	2	0.03514
3	5	5	3	0.03740
4	5	10	1	0.03085
5	5	10	2	0.04190
6	5	10	3	0.04870
7	5	15	1	0.03985
8	5	15	2	0.04210

9	5	15	3	0.04936
10	10	5	1	0.03696
11	10	5	2	0.03790
12	10	5	3	0.04620
13	10	10	1	0.04189
14	10	10	2	0.04080
15	10	10	3	0.04680
16	10	15	1	0.04180
17	10	15	2	0.04700
18	10	15	3	0.04990
19	15	5	1	0.03640
20	15	5	2	0.04560
21	15	5	3	0.04890
22	15	10	1	0.04470
23	15	10	2	0.04890
24	15	10	3	0.05540
25	15	15	1	0.04910
26	15	15	2	0.04990
27	15	15	3	0.06200

ANOVA Analysis

ANOVA was used to investigate which design parameter significantly affected the quality characteristic. Here, ANOVA is used to analyze the influence of coating parameters like (TS) time for sensitization, (TA) time of activation, and (TM) time for metallization. The ANOVA of thickness of the coating were carried out using the adjusted sum of square for tests. It is a group of statistical models used to examine the differences among group means. Table 4 shows the results of ANOVA analysis [13]. One can observe from the ANOVA analysis that the time for sensitization, time for activation and time for metallization have the influence on coating thickness on fiber.

Usually, the P value is the term that indicates whether the parameter has significant effect or not on the process. If the value of $P > 0.05$,then that particular parameter has no significant effect and it can be neglected. The parameter is significant only if the value < 0.05. At same time value F indicates which parameter is dominate compared to other parameter. From the table below, all the factors have their values below 0.05, hence all have some effect on the coating thickness and time for metallization($F=23.99$) has dominate effect on the coating thickness.

Table 4. ANOVA Results

Source	DF	Seq SS	Adj SS	Adj MS	F	P
T S	2	0.0003109	0.0003109	0.0001555	17.66	0.00
T A	2	0.0002603	0.0002603	0.0001301	14.78	0.00
T M	2	0.0004224	0.0004224	0.0002112	23.99	0.00
Error	20	0.0001761	0.0001761	0.0000088		
Total	26	0.0011697				

Control factor study

The response table for means of thickness of the coat deposited on the fiber is shown in table 5. It helps to analyze the effect of control factors based on delta statistics value. The highest average value minus lowest average value of the individual factor gives the delta value and the delta ranks are assigned based on these values, rank 1 is achieved for highest delta value, second highest denotes rank 2 and so on. This analysis helps to extract useful data about the process and the parameter having highest delta value will be the most influential parameter for the thickness to get deposited.

Table 5. Response table for means

Level	TS	TA	TM
1	0.04037	0.04029	0.03996
2	0.04325	0.04402	0.04283
3	0.04857	0.04789	0.04941
Delta	0.00819	0.00760	0.00945
Rank	2	3	1

It is observed from table 5, the delta characteristic value for time for metallization is greater compared other parameters. Hence, the most influential parameter having significant effect on thickness of coating on the fiber is time for metallization (TM) followed by time for sensitization (TS). The main effects plot of the process parameter is shown in the figure 1. If the graph shows decrease trend pointing towards the horizontal axis then that particular parameter has no significant effect on the process. On the other hand if the parameter line shows the increasing trend away the horizontal X-axis then it has significant effect on the process. It is observed from the plots that all the parameter shows increasing trend moving away from horizontal x-axis. The graph of time for metallization and mean coating thickness shows higher trend indicating a mean coating value of 0.0494, hence it is more significant than other parameter.

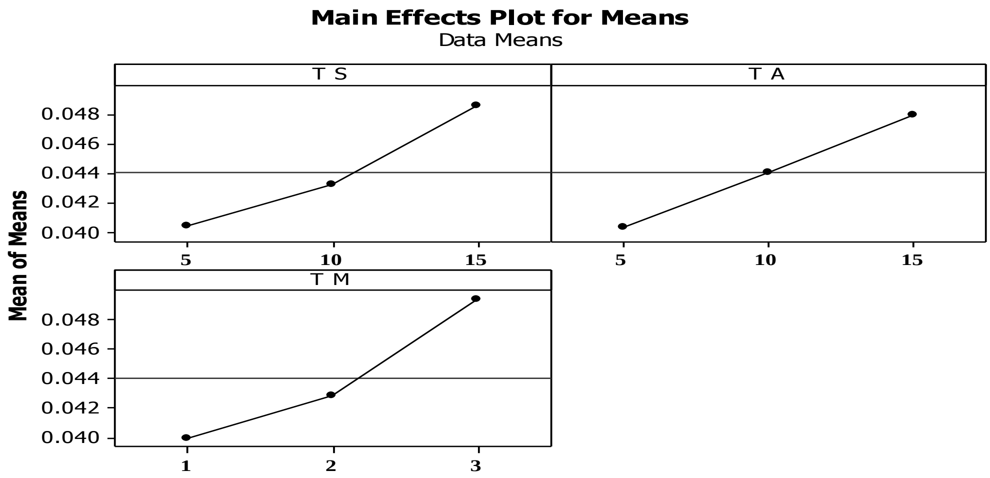

Figure1. Mean effect plot for means for thickness of coating on fiber

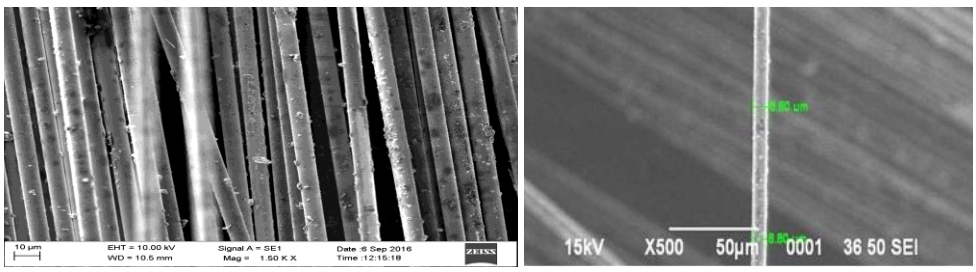

Figure (2) a & b. Nickel coated carbon fibre

Figure 2 a & b are the scanning electron microscopic images which evidence the thin electroless nickel coating on carbon fibres.

Conclusion

From the results obtained from the analysis of the coating parameters on the thickness of the coat using Electroless nickel coating method, following conclusions can be drawn:

- Coating of the carbon fibre by Electroless nickel coating helps to deposit the coat uniformly and continuously on the fibre.
- Taguchi L_{27} orthogonal array design is suitably applied to optimize and to study interaction with the three coating parameters such as time for sensitization(TS), time for activation(TA) and time for metallization(TM) to achieve the required coating thickness of Ni.
- From ANOVA analysis, it is observed that all the parameters have significant effect on the process, with time for metallization (F=23.99) having dominate effect on the thickness of the coat.

- Control factor study revealed, the most influential parameter having significant effect on thickness of coating on the fibre is time for metallization (TM) followed by time for sensitization (TS).

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