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Statistical Investigation on Effect of Electroless Coating Parameters on Coating Morphology of Short Basalt Fiber

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Abstract

The Objective of the present paper is to investigate the effect of electroless coating parameters, such as Sensitization time (A), Activation time (B) and Metallization time (C), on the coating morphology of the basalt short fiber and the optimization of the coating process parameters based on L_{27} Taguchi orthogonal design. Coated and non-coated basalt short fiber, typically used with 7075 Aluminium alloy as reinforcement, is studied. The effect of coating the short basalt fiber with copper has proved beneficial to interfacial bonding (wettability) between the reinforcement and the matrix. The interface between the matrix and the reinforcement plays a crucial role in determining the properties of metal matrix composites (MMCs). An L_{27} array was used to accommodate the three levels of factors as well as their interaction effects. From the Taguchi methodology, the optimal combinations for coating parameters were found to be $A_1B_3C_3$ (i.e., 5 min. sensitization time, 15 min. activation time and 3 min. for metallization time). In addition, the interaction between pH value and the coating time and that between the coating time and the temperature, influence the coating parameters significantly. Furthermore, a statistical analysis of variance reveals that the metallization time has the highest influence followed by the activation time and the sensitization time. Finally, confirmation tests were carried out to verify the experimental results, Scanning Electron Microscopic (SEM) & Energy Dispersive Spectroscope (EDS) studies were carried out on basalt fiber.

Keywords: *Electroless Coating, Basalt Fiber, Taguchi Technique, Genetic Programming.*

1. Introduction

The interface between the matrix and the reinforcement is crucial and can affect the fabrication process significantly. If this interface is not tailored properly, it can lead to the degradation of the properties of composites. One of the general approach to this problem is deposition of coating around the fiber [1]. The coating technique has been studied by many researchers for the fabrication of metal matrix composites (MMC) [1-4]. Coating of reinforcement is one of the successful techniques adopted to prevent interfacial reaction and enhance the wetting of reinforcement [5]. Electroless copper coating of the reinforcement, which is a simple, low-cost and an easy to use process, has been successfully applied to prevent undesirable interfacial reactions and promote the wettability through increased overall surface energy of the reinforcement [6-8]. An attempt is made in the present investigation to assess the influence of electroless coating parameters such as Sensitization time (A), Activation time (B) and Metallization time (C) on the coating morphology of basalt short fiber. There are several other coating parameters that can affect the coating process of basalt short fiber viz. pH value and temperature of the bath. In this study, Sensitization time (A), Activation time (B) and Metallization time (C) are considered for the optimization of coating. The main purpose of this work is to establish the influence of these coating parameters on the coating morphology of basalt short fiber and further to optimize the process based on L_{27} Taguchi orthogonal design [9-10]. This can be achieved through a series of experiments. However, such experiments will be expensive and often time-consuming. Design of Experiment (DOE) techniques, like the Taguchi method, can minimize the experimental runs to optimize the coating parameters. Furthermore, to identify which of the testing parameters have a significant influence over the performance of copper coating, Analysis of Variance (ANOVA) is also performed. Finally, verification and validation of the optimal condition obtained through orthogonal array design is carried out through a confirmation test and the improvement in the coating performance characteristics at the optimal condition is compared to the initial condition. Results thus obtained are also correlated with the ones obtained by genetic programming using DISCIPULUS^{TM} software.

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2. Design of Experiments (DOE)

Design of experiment is one of the important and powerful statistical techniques to study the effect of multiple variables simultaneously and it involves a series of steps which must follow a certain sequence for the experiments to yield an improved understanding of process performance. These experiments result in a certain number of combinations of factors and levels to be tested in order to achieve the required parametric variations. Taguchi approach relies on the assignment of factors in specific orthogonal arrays to determine those test combinations. Analysis of the experimental results uses a signal to noise ratio to aid in the determination of the best process design. The application of DOE concepts, like Taguchi technique factorial and surface response, has gained importance since these were helpful in providing information about the influence of various parameters in the hierarchal rank order. The combined effects of these parameters can be analyzed, and the correlation term can be found using these techniques.

2.1. Taguchi Technique

The Taguchi method, which is effective to deal with responses, was influenced by multi-variables. This method drastically reduces the number of experiments that are required to model the response function compared with the full factorial design of experiments. The major advantage of this technique is to find out the possible interaction between the parameters. The Taguchi technique is devised for the process optimization and the identification of the optimal combination of the factors for a given response. This technique is divided into three main phases, which encompass all the experimentation approaches. The three phases are:

1. The planning phase
2. The conduction phase
3. The analysis phase.

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

3. Experimental Work

3.1. Materials and Methods

Basalt is a natural material that is found in volcanic rocks. It has a melting temperature of 1300-1700°C, which is crushed and spun into fine continuous fibers. These are made with thermosetting resins, such as epoxy and (phenolic) polyesters using techniques like prepregs, laying out, winding, direct pressure autoclaving, and vacuum moulding etc. Continuous basalt fibres (CBF) has good thermal, electrical and sound insulating properties, good resistance to acids and solvents, and good thermal stability (under very low stress up to 1250°C, under common load only to 500°C) and has low cost compared to other fibers. Reinforcements used in this study are in the form of continuous basalt fibers with chemical composition as shown in Table 1. In the present investigation, the deposition of copper coating on basalt short fibers by an electroless route has been optimized.

Table 1. Chemical composition of short basalt fiber

Element	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	MnO
%	69.51	14.18	3.92	2.41	5.62	2.74	1.01	0.55	0.04

3.2. Pre-Procedure

The continuous basalt fibers of average diameter 6 µm were chopped down to short fibers of about 1 to 2 mm length. The complete process of coating starts with the treatment of fibers in a muffle furnace for 10 min. at 500°C to eliminate the pyrolytic coatings around as received fibers. The electroless process, used to deposit the copper coatings on the basalt fiber, relies on a sequence of sensitizing, activation and metallization, with important cleaning, rinsing, washing and drying stages also being included.

3.3. Experimental Procedure

The short basalt fiber was cleaned in distilled water and dried at 90°C. The sizing and finishing treatment from the surface of the fibers, prior to coating, were removed by heating them to about 970 K for 10 min. in air. Fibers have elastic modulus of 90 GPa, and a yield stress of 4500 MPa. The coating procedure consist of three well-defined stages, namely sensitization, activation and metallization. The heat-cleaned fibers are first treated with glacial acetic acid to activate the surface, and then activated again using stannous chloride ($SnCl_2$); they were sensitized for different times (5, 10 & 15 min.) under continuous stirring. Fibers are then filtered and cleaned with distilled water. In order to have catalytic surfaces, the sensitized fibers were exposed to an aqueous solution containing 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 copper. The complete process of metallization starts with the treatment of fibers in an open oven for 10 min. at 500°C to eliminate the pyrolytic coatings around as received fibers.

Metallization is produced by immersion of activated fibers into a solution containing $CuSO_4.5H_2O$ as metal ion sources also held under agitation. Different metallization conditions have been tried with timings, as indicated in Table 2, for the three processes, and the required thickness has been achieved. The reactive volume used assures that the concentration of the diluted copper can be considered constant during the deposition. The coatings obtained at different metallization times were then studied by SEM and the thickness of the copper layer was determined in transversal cross section. The specimen was mounted on a metal stub on top of which a double sided carbon tape was used and the sample was stuck on a carbon tape. Later the entire stub was placed in the coating machine for the copper coating process. For a non-conductive specimen, metal coating was usually applied to give the specimen electrical conductivity. This decreases the specimen's capacity to acquire an electrostatic charge and increases the yield of secondary electrons. The important thing to remember while applying coating on short basalt fiber is to make sure that the coating on basalt short fiber must be as thin as possible so that the specimen surface morphology is not completely covered by coating. The resultant images reveal remarkable structural resolution down to a few nanometers with great accuracy, because the film provides a continuous coating over all the sample contours. The mould or stub was kept in the vacuum chamber and SEM imaging and EDS were done through JEOL JSM 6360 - A model with a magnification capacity of X500, X1000, X2000 and accelerating voltage of 20 kV with a working distance of 10 mm. The first area of image was chosen and focused then through software EDS analysis was done on the same image by either selecting spot analysis or line analysis or area analysis. The morphology of the coated fiber is studied with SEM followed by EDS to evaluate elemental distribution. Experiments were conducted based the outcome of Taguchi's technique. A L_{27} orthogonal array was selected for the analysis of the data as given in Table 3. Investigation to find the effect of time for sensitization, time for activation & time for metallization was carried out using Taguchi S/N ratio and regression equations for each response were developed. Objective of the model was chosen as 'Nominal the best type' [12] characteristics to analyze the effect of coating parameter.

3.4. Plan of Experiment

Conventional experimental design methods are too complex and expensive. A large number of experiments have to be carried out to study the process. Taguchi method uses an orthogonal array to study the entire process with only a small number of experiments. Moreover, traditional experimentation involves one factor at a time, wherein one variable is changed while the rest are held constant. The major disadvantage of this method is that it fails to consider any possible interactions between the parameters. An interaction is the failure of one factor not to produce the same effect on the response at different levels of a second factor varying. It is also not possible to study all the factors involved in the process and to determine their main effects (i.e., the individual effects) in a single experiment. Taguchi technique overcomes all these drawbacks and hence used for optimizing coating parameters and identifying the optimal combination of factors for the desired responses [15]

The steps involved are:

1. Identification of the response functions and the process parameters.
2. Determination of the number of levels for the process parameters and possible interaction between them.
3. Selection of the appropriate orthogonal array.
4. Selection of the optimum level of process parameters through Analysis of Variance [ANOVA].
5. Performing a confirmation experiment to verify the optimal process parameters.

The experiments were conducted as per the standard orthogonal array. The selection of the orthogonal array was based on the condition that the degrees of freedom for the orthogonal array should be greater than or equal to the sum of those coating parameters. In the present investigation, an L_{27} orthogonal array was chosen, which has 27 rows and 13 columns, as shown in the Table 3.

Table 2. Coating parameters with their values at three levels

Duration (minutes)			
Level	**Sensitization**	**Activation**	**Metallization**
1	5	5	1
2	10	10	2
3	15	15	3

The input parameters chosen for the experiments are Sensitization time (A) in minutes, Activation time (B) in minutes and Metallization time (C) in minutes. Table 2 indicates the factors and their levels. Trail runs were carried out by varying one of the coating parameters while keeping the rest of them at constant values. The working range was decided upon by measuring the achievable amount of deposition of copper coating on basalt short fiber. The range and the number of levels of the design parameters are given in Table 2. The fiber coating experiment was performed as per the condition dictated by design matrix, developed through Taguchi technique. The experiments consist of 27 tests (each row in the L_{27} orthogonal array) and the columns were assigned with parameters. The first column in Table 3 was assigned to time for sensitization (A), the second column for activation (B) and the fifth for metallization (C) and the remaining columns were assigned to their interactions. The response to be studied was the coating thickness on the fiber with the objective as "Nominal the best". The experiments were conducted as per the orthogonal array with level of parameters given in each array row. The coating test results were subjected to the analysis of variance.

Table 3. Orthogonal Array L_{27} (3^{13}) of Taguchi

$L_{27}(3^{13})$ Test	1	2	3	4	5	6	7	8	9	10	11	12	13
1	1	1	1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	1	2	2	2	2	2	2	2	2	2
3	1	1	1	1	3	3	3	3	3	3	3	3	3
4	1	2	2	2	1	1	1	2	2	2	3	3	3
5	1	2	2	2	2	2	2	3	3	3	1	1	1
6	1	2	2	2	3	3	3	1	1	1	2	2	2
7	1	3	3	3	1	1	1	3	3	3	2	2	2
8	1	3	3	3	2	2	2	1	1	1	3	3	3
9	1	3	3	3	3	3	3	2	2	2	1	1	1
10	2	1	2	3	1	2	3	1	2	3	1	2	3
11	2	1	2	3	2	3	1	2	3	1	2	3	1
12	2	1	2	3	3	1	2	3	1	2	3	1	2
13	2	2	3	1	1	2	3	2	3	1	3	1	2
14	2	2	3	1	2	3	1	3	1	2	1	2	3
15	2	2	3	1	3	1	2	1	2	3	2	3	1
16	2	3	1	2	1	2	3	3	2	1	2	3	1
17	2	3	1	2	2	3	1	1	2	3	3	1	2
18	2	3	1	2	3	1	2	2	3	1	1	2	3
19	3	1	3	2	1	3	2	1	3	2	1	3	2
20	3	1	3	2	2	1	3	2	1	3	2	1	3
21	3	1	3	2	3	2	1	3	2	1	3	2	1
22	3	2	1	3	1	3	2	2	1	3	3	2	1
23	3	2	1	3	2	1	3	3	2	1	1	3	2
24	3	2	1	3	3	2	1	1	3	2	2	1	3
25	3	3	2	1	1	3	2	3	2	1	2	1	3
26	3	3	2	1	2	1	3	1	3	2	3	2	1
27	3	3	2	1	3	2	1	2	1	3	1	3	2

4. Results and Discussion

4.1. Analysis of Experimental Results

After being collected, the data are to be analyzed by means of calculating S/N ratio (S/N: Signal/Noise) [16]. The signal to noise ratio in this case is a quality indicator by which the effect of changing a particular coating parameter for deposition of copper on the short basalt fiber is evaluated. In general, a better signal is obtained when the noise is smaller, so that a larger S/N ratio yields better final results. This means that the divergence of the final results becomes smaller. Signal to noise ratios of each experimental run is calculated based on the following equation, and the values are

$S/N\ (\eta) = 10\log_{10}(\mu^2/\sigma^2)$; Where $\mu = 1/n\Sigma y_i$,

$\sigma^2 = 1/(n-1)\ \Sigma(y_i - \mu)^2$

Where $y_1, y_2,$ ----------- y_n are the response of the coating thickness and 'n' is the number of observation.

The experimental results are analyzed, to see the main effects of interaction between level 1, level 2 and level 3 of the variables on coating parameters in the experiments. In the present work, three runs are performed 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; the above mentioned approach has been adopted. However, in situations where there is a feasibility to perform multiple runs for each combination of parameters provided by the design matrix, the Taguchi analysis can be performed by using S/N ratio analysis. In the present analysis, considering the constraint (coating thickness and time) mentioned above, Taguchi analysis is performed based on "average of results" methodology, which is shown in Table 4. Based on the analysis in Table 4, using S/N ration, the optimum combination would be $[A_1B_3C_3]$

4.2. Analysis of Variance

The use of ANOVA is to analyze the influence of coating parameters like (A) time for sensitization, (B) time of activation, and (C) time for metallization. This analysis was carried out for a level of significance of 5% that is the level of confidence 95%. Table 5 shows the results of ANOVA analysis. 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. The last column in Table 5 shows the percentage contribution of each factor on the total variation indicating their degree of influence on the result. The interaction between the above factors does not have a significant variation on the coating thickness on the fiber. It can be observed from the ANOVA table that the time for metallization (p=0.97%) is comparatively less than the time for activation (p=25.94%) and time for sensitization (p=3.25%), in spite of which there is no appreciable decrease in coating thickness. However, the interaction between time for sensitization and time of activation (p=18%), time of activation and time for metallization (p=45.03 %) shows the highest when compared to time of activation and time for metallization (p=4.44%). The pooled error associated in ANOVA table was approximately about 2.37%. This approach gives the variation of means and variance to absolute values considered in the experiment and not the unit value of the variable.

Table 4. S/N ratios for coating thickness for different coating parameters

Exp. No.	Time for sensitization (min)	Time for activation (min)	Time for metallization (min)	Mean thickness coating (μm)	S/N ratio (dB)
1	5	5	1	0.0465	-6.256
2	5	5	2	0.0981	0.218
3	5	5	3	0.0984	0.245
4	5	10	1	0.0840	-1.135
5	5	10	2	0.0536	-5.032
6	5	10	3	0.0394	-7.705
7	5	15	1	0.0376	-12.011
8	5	15	2	0.0552	-4.776
9	5	15	3	0.0157	-18.111
10	10	5	1	0.0685	-2.901
11	10	5	2	0.0240	-15.697
12	10	5	3	0.0993	0.324
13	10	10	1	0.0722	-2.444
14	10	10	2	0.0799	-1.564
15	10	10	3	0.0617	-3.809
16	10	15	1	0.0457	-6.417
17	10	15	2	0.1068	0.957
18	10	15	3	0.0430	-6.956
19	15	5	1	0.1048	0.792
20	15	5	2	0.0541	-4.951
21	15	5	3	0.1112	1.307
22	15	10	1	0.1044	0.755
23	15	10	2	0.0908	-0.458
24	15	10	3	0.0610	-3.908
25	15	15	1	0.0350	-8.734
26	15	15	2	0.0575	-4.422
27	15	15	3	0.0200	-13.594

Table 5. ANOVA Results

Source of Variances	SS	DOF	Variance	F_{cal}	F_{th}	p %
A	0.000583	2	0.000292	05.49	3.39	03.25
B	0.004649	2	0.002324	43.75	3.39	25.94
C	0.000174	2	0.000086	01.63	3.39	00.97
A x B	0.003226	4	0.001613	15.17	3.39	18.00
A x C	0.008071	4	0.004035	37.98	3.39	45.03
B x C	0.000795	4	0.000398	03.74	3.39	04.44
Error	0.000425	8	0.000213		3.39	02.37
Total	0.017923	26	0.008961			100.00

4.3. Morphology of Electroless Cu Coating

Figure 1 (a) shows the SEM micrographs as received basalt fiber and Figure 1(b) and 1(c) show electroless copper coated basalt fibers with low and higher magnification [17]. As shown in Figure 1 (a), basalt fibers have smooth surface striations along the fiber axis. In spite of the different surface morphology, the electroless copper films have been be deposited on the fiber surface with good adhesion as indicated in Figures 1(b) and 1(c). From Figure 1 (c), we observe the deposition of copper layer on the surface of basalt fiber with a thickness of 40 nanometres as measured by SEM. A uniform and continuous coating of copper was given to short basalt fiber by optimized value about 95% of the continuously coated fibers had a coating thickness range about 20-50 nanometres and above this showed isolated dendrite deposit of copper. Figure 2, (a) and (b), shows the EDS pattern of uncoated and copper coated basalt fiber, respectively. Micrographs reveal clearly the deposition of copper on the short basalt fiber. Superior aggregations among the basalt fiber were observed due to the high chemical activity of copper atoms. The original basalt fiber exhibit a glossy surface. It can also be seen via the the micrographs that, after successful coating, the surface appears dim. The EDS results confirm the presence of Cu, indicating a successful activation, sensitization and metallisation process.

4.4. Orthogonal Array of Taguchi for Coating Thickness

The experimental studies were conducted according to the L_{27} orthogonal mixed level array. The coating thickness was obtained as a result of the electroless coating experiments and the corresponding S/N ratios values are tabulated in the Table 4.

A graph of main factors plotted against the factor level obtained by calculating the coating thickness of the specimens are as shown in fig 3(a) & 3(b). It is observed from the graph that the coated thickness tends to increase in factor A (time for sensitization), a decrease in factors B (time for activation) and C (time of metallization). The levels that have the highest value of the S/N ratio and mean of mean thickness are the best factor levels. The optimum value is observed for combination of $A_1B_3C_3$ equation obtained by the S/N ratios.

4.5. Confirmation Test

A parallel study using GP was taken up to correlate the Taguchi based approach using DISCIPULUS^{TM} software. To implement the concept of Genetic Programming, DISCIPULUS^{TM} software was used. Data sets from the experiments were taken for analysis. The data samples were randomized manually using Microsoft Excel software. The randomized data sets were fed into the software by initially splitting them into three sets viz., training, validation and applied testing [18].

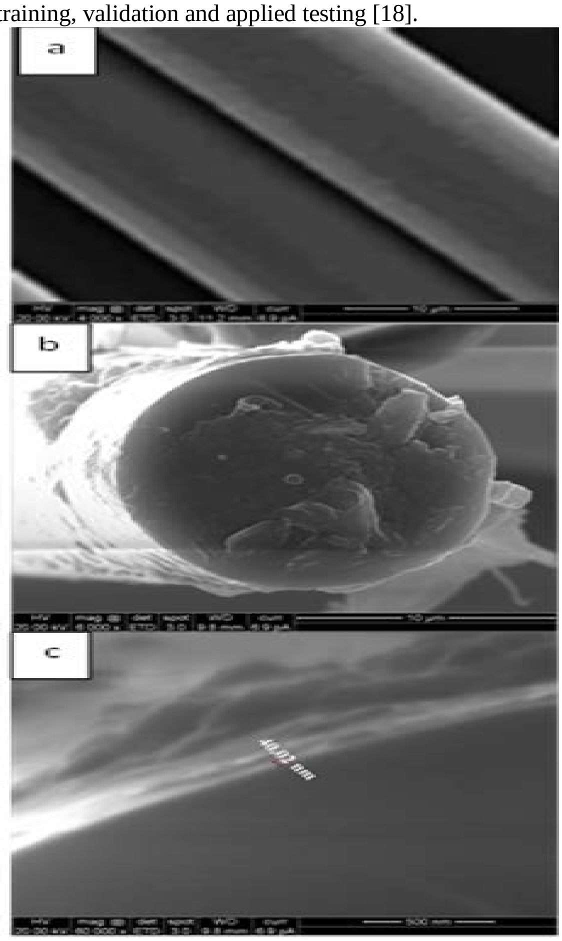

Figure 1. SEM micrographs

a) Uncoated basalt fiber

b) Lower magnification copper coated basalt fiber

c) Higher magnification copper coated basalt fiber [17]

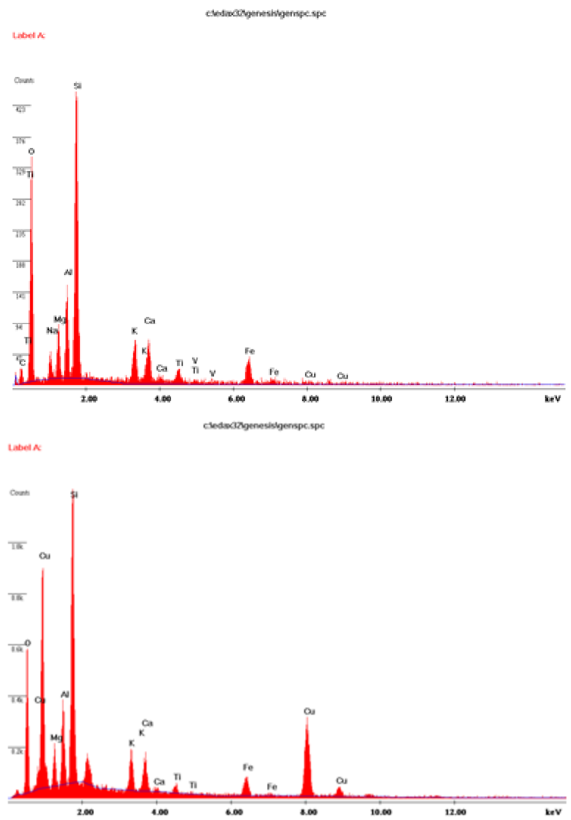

Figure 2. EDS image pattern

a) Uncoated basalt fiber

b) Copper coated basalt fiber [17]

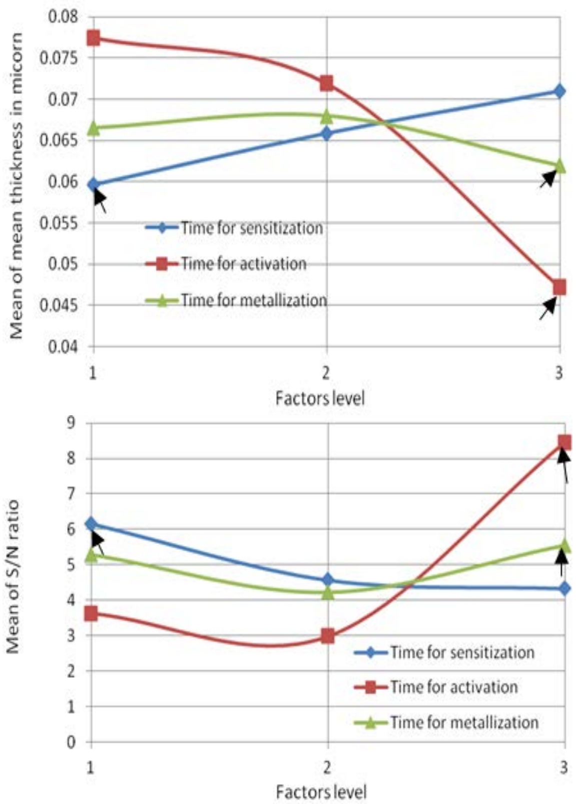

Figure 3. Relative effect of main factors on coating

a) Mean of mean thickness

b) S/N ratio.

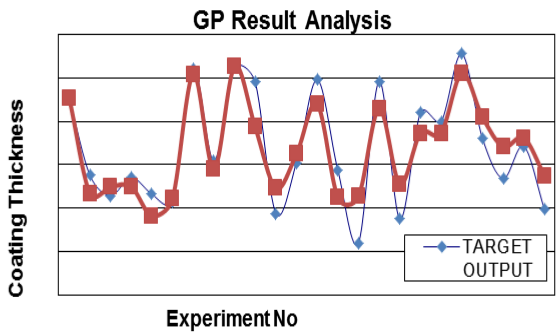

Figure 4. GP Result Analysis

5. Conclusions

From the analysis on the results of coating parameter on coating morphology of short basalt fiber by electroless method, the following conclusions can be drawn:

1. Coating of copper on short basalt fiber has been carried out successfully by electroless method.
2. Coating with optimized values resulted in about 95% of the continuously coated fibers with a coating thickness range of 20-50 nanometres and isolated dendrite deposit of copper in the remaining region.
3. Taguchi orthogonal array design is suitably applied to optimize the three coating parameters such as time for sensitization(A), time for activation(B) and time for metallization(C) to achieve the required coating thickness of Cu.
4. The optimum testing condition $[A_1B_3C_3]$ obtained from the analysis yields optimum coating thickness compared to other parametric combinations.
5. ANOVA shows that the time for metallization (p=0.97%) is comparatively less than the time for activation (*p*=25.94%) and time for sensitization (p=3.25%), in spite of which there no appreciable decrease in coating thickness.
6. The SEM micrographs and EDS results indicate that the optimized parameters resulted in consistent coating morphology and thickness.
7. The results obtained from genetic programming were compared with the experimental values obtained by Taguchi technique. The results obtained from genetic programming are in general agreement with the experimental results with minimal variations.

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