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Optimization of Coating Parameters on Coating Morphology of Basalt Short Fiber for Preparation of Al/Basalt Metal Matrix Composites Using Genetic Programming

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Abstract

An electroless method of coating copper on basalt fibers using copper sulphate solution is described. The effects of Sensitization time (A), Activation time (B) and Metallization time (C), used for electroless coating, were optimized to obtain a uniform and a continuous layer of copper coating on the fiber. These factors are crucial in the quality of metal matrix composites made with basalt fibers. The objective of the present paper is to investigate the effect of electroless coating parameters in electroless solution on coating morphology of basalt short fibers and optimization of the coating process parameters based on Genetic programming. A mathematical modeling was generated using Genetic programming and the results were validated using DISCIPULUSTM software. This work gains significance from the sense that, with a reasonably minimum number of experiments, a reliable model has been generated, validated and, further, the process has been optimized. Results show that the metallization time has the highest influence followed by activation and sensitization. Confirmation tests were carried out to verify the experimental results.

Keywords: *Basalt Fibers, Genetic Programming, Electroless Copper Coating.*

1. Introduction

In recent years, a major growth has been observed in the manufacture of Metal Matrix Composite (MMCs) reinforced with fibers. Due to its high specific strength, better wear and corrosion resistance and stability at high temperature, they are used in different sectors of industry and technology [1]. Basalt fibers are used as reinforcement of aluminium alloy because they improve their electrical and thermal conductivity properties by reducing their density and coefficient of thermal expansion [2]. Poor wettability is one of the major problems observed during the fabrication of fiber reinforced aluminium matrix composites. The interface between the matrix and the reinforcement is the critical region affected during fabrication. If this interface is not tailored properly, it can lead to degradation of the properties of the composites. Improper wettability and interfacial chemical reaction occurring between the dispersion surface and the matrix at the interface during synthesis can degrade the mechanical, electrical, thermal and chemical properties of the composite [3]. To solve these problems, many researchers have used special alloying coating metals like copper, silver, nickel, tantalum and cobalt and ceramics, like titanium boride and born carbide to coat fibers [4]. Copper and nickel coatings are commonly used by many researchers in the fabrication of metal matrix composites. In many MMCs, it is necessary to apply a thin coating on the reinforcements prior to their incorporation into the metal matrix. Given the major role of coatings, there are several techniques available for the deposition of thin coatings on long fibers and, to a much lesser extent, on short fiber and particulate reinforcement. Electroless Copper (EC) coating is highly preferable in the research community due to its simple, low-cost and easy-to-use process. It has also been successfully applied to prevent undesired interfacial reactions and promote the wettability through increasing the overall surface energy of the reinforcement [5].

2. Genetic Programming

Genetic Programming (GP) is a domain-independent problem-solving approach in which computer programs are evolved to solve, or approximately solve, problems [6]. GP is one of the most useful, general purpose problem-solving techniques available to developers. It has been used to solve a wide range of problems, such as symbolic regression, data mining, optimization, and emergent behavior in biological communities [7]. GP is a method to evolve computer programs. In artificial intelligence, GP is an evolutionary algorithm-based methodology inspired by biological evolution to find computer programs that

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perform a user-defined task. It is a specialization of genetic algorithms (GA) where each individual is a computer program. It is a machine-learning technique used to optimize a population of computer programs according to a fitness landscape determined by a program's ability to perform a given computational task [8; 9]. GP and a similar tool, Genetically Optimized Neural Network System (GONNS) are machine-learning techniques used to optimize a population of computer programs according to a fitness landscape determined by a program's ability to perform a given computational task [10; 11]. In order to get a uniform surface coating on the reinforcement, it is prudent to adopt analytical methods to determine optimal coating parameters by establishing the method for predicting the responses. For this purpose, GP is used, which gives a mathematical model relating the input variables and the output parameter. The model is validated using the experimental data collected and, further, it predicts the output for the given set of input variable.

3. Experimental Procedure

3.1. Materials and Methods

Basalt is a natural material 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 a good thermal stability (under very low stress up to 1250°C, under common load only to 500°C) and is of a low cost compared to other fibers. The basalt short fiber, used as reinforcement in the present investigation, was purchased from a mineralogical research company. Reinforcements, used in this study, are in the form of continuous basalt fibers with a chemical composition as shown in Table 1. In the present investigation, the deposition of copper coating on basalt short fibers, by an electroless route, was optimized [12].

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. Coating 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 697°C for 10 min in air. Fibers have elastic modulus of 90 GPa, and a yield stress of 4500 MPa. The coating procedure consists 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 again activated through using stannous chloride ($SnCl_2$) and were sensitized for different times (5, 10 and 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 allow 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 [13].

Table 2. Chemical compositions for coating process

Stage and conditions	Concentration of chemicals
Sensitization	12 g/*l* $SnCl_2 \cdot 2 H_2O$
5min, 10min and 15 min at room temperature	40 m*l*/*l* HCl
Activation	0.2g/*l* $PdCl_2$
5min, 10min and 15 min at room temperature	2.5 m/*l* HCl
Metallization	10g/*l* $CuSO_4$ - 5 H_2O
Multiple conditions tested	45g/*l* EDTA
40^0C and 50^0C	20 g/*l* NaCOOH
pH 12 and pH 13	16 m*l*/*l* HCHO 36%
2 min - 20 min	NaOH for adjusting pH

Metallization is produced by the 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 required thickness were 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 copper coating process. For a non-conductive specimen, a 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 a 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 a 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 was done through JEOL JSM 6360 - A model with a magnification capacity of X500, X1000, X2000 and accelerating voltage of 20 kV with working distance of 10 mm. The first area of the image was chosen and focused then, through software EDS analysis, was done on the same image by either selecting spot analysis, line analysis, or area analysis. Morphology of the coated fiber is studied with SEM followed by EDS to evaluate elemental distribution [13].

3.4. Implementation of Genetic Programming

To implement the concept of Genetic Programming, DISCIPULUS^{TM} software was used. Data sets from the experiments were taken for the 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 [14]. DISCIPULUS^{TM} self configures itself to accept the last column, always as the expected output. Trial runs to find out the best parameters that generated optimal solution in the minimum possible time. Initially, the runs were performed with the default settings. One by one the parameters such as population size, crossover rate, DSS sub set size, and FPU registers used were varied to find optimum values [15]. The trials showed the results described below. Population size of 800 was optimum rather than the default setting of 500. A higher crossover rate {75% non-homologous and 25% homologous} was found to be optimum. A smaller DSS subset size {60} was more optimal than the default 100. The above factors favorably affected result generation. The process input and output parameters to be considered for genetic model are shown in Table 3. The actual experimental values of coating thickness in µm are tabulated in Table 4.

Table 3. Process input and output of coating parameter

Process input	Measured process output
Sensitization time (min)	Sensitization time (min)
Activation time (min)	Activation time (min)
Metallization time (min)	Metallization time (min)

3.5. Genetic Models

The mathematical model is a collection of statistical and mathematical techniques used for developing, improving and optimizing process variables; this is dedicated to the evaluation of relations existing between a group of controlled experimental factors and the observed results of one or more selected criteria. Genetic programming evolves a group of techniques used in empirical study of the relationship between a response and several input variables [16]. Selection of the function-set included plus, minus, multiply, divide, power and square. With the randomly selected group of experimental data, by varying fitness constants through numerous iterations, using Genetic Modeling System (GeMS) software, the Genetic Models were obtained. Mathematical models, obtained through genetic programming called Genetic Models, are given below:

Table 4. Coating thickness of short basalt fiber

Exp. No.	Time for sensitization (sec)	Time for activation (sec)	Time for metallization (sec)	Thickness coating (µm)
1	5	5	1	0.0465
2	5	5	2	0.0981
3	5	5	3	0.0984
4	5	10	1	0.08395
5	5	10	2	0.0536
6	5	10	3	0.0394
7	5	15	1	0.0376
8	5	15	2	0.0552
9	5	15	3	0.0240
10	10	5	1	0.0685
11	10	5	2	0.0157
12	10	5	3	0.0993
13	10	10	1	0.0722
14	10	10	2	0.0799
15	10	10	3	0.0617
16	10	15	1	0.0457
17	10	15	2	0.1068
18	10	15	3	0.04295
19	15	5	1	0.1048
20	15	5	2	0.0541
21	15	5	3	0.1112
22	15	10	1	0.10435
23	15	10	2	0.09075
24	15	10	3	0.061
25	15	15	1	0.035
26	15	15	2	0.0575
27	15	15	3	0.02

f0 =0.40457
f0 = [[0.40457 V2 - V1 V0]/ V1] *0.14971
f0 = [[V1 V0 - 0.40457 V2]/ 6.6796V1] + V2]2=A
f0 = A- f1
f0 = sin A
f0 =2 sin A
f0 = 4sin A
f0 = 4sin A/ V2
f0 = 4sin A/ V2 + V2
f0 = [4sin A+V22] / V2 =B
f0 = sin B
f0 = [2sin B/ V2] + 0.17607=C
f0 = sin C
f0 =2 sin C
f0 =-2 sin C
f0 =-0.209388 sin C
f0 =0.043843 sin2 C
f0 =0.08769 sin2 C
f0 = [(0.08769 sin2 C+0.065403) 2 * (-2 sin C)/ V2] + 0.0821141
f0 = [(0.08769 sin2 C+0.065403) 2 * (-2 sin C)*V2] + 0.0821141
Where
C= (2 sin B/ V2) + 0.17607
B= (4 sin A+ V22)/ V2
A= [[V1 V0 - 0.40457 V2]/ 6.6796V1] + V2]2
Specimen calculation:

Variable	Frequency	Average Impact
v[00] 0.73	0.29421	0.73464
v[01] 0.63	0.28339	0.74690
v[02] 1.00	0.57595	0.72940

Checking

V_0=5

V_1= 5

V_2=10

A= 112.941882

B=10.36836

C=0.21206

4. Results and Discussion

Experiments were carried out for collecting experimental data in a controlled manner. Such data were grouped separately at random for the genetic model (the model developed using GP) and further validating them. In the proposed concept the mathematical models for verifying the coating parameter are subject to adaptation. After many trials, with the help of validation and testing data, the fittest models were developed. Models generated for each output parameter (response) establish the relationship between the variable input parameters and the output parameter. The models generated for the output parameters were used to obtain the optimal set of coating parameters and also the order of their influence on the output parameter. In all the models above, Metallization time (C) is the dominant factor that influences the output parameter, followed by Sensitization time (A) and Activation time (B). Predicted values, using the models above, were compared with the actual values of training data set (Table 5) and the majority of the set of values are well within 10% variation and few sets have gone to an extent of 15% variation. This clearly shows that the experiments have been conducted for all sets of input variables in a controlled manner and the measurement errors are within the permissible limits. Secondly, the models developed through Genetic Programming are dependable since their fitness quality within the data chosen for training them is high. In addition, these models were validated using independent data sets and for these data sets also the comparison between predicted and actual values are well within the acceptable range.

4.1. Morphology of Electroless Cu Coating

Figure 1 (a) shows the SEM micrographs as received basalt fiber. Figure 1(b) and 1(c) show electroless copper coated basalt fibers with low and higher magnification [12]. 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 deposited on the fiber surface with good adhesion as indicated in Figures 1(b) and 1(c). From Figure 1 (c), it is observed that the deposition of a copper layer on the surface of basalt fiber with a thickness of 0.08207 μm as measured by SEM. A uniform and continuous coating of copper was given to a short basalt fiber by optimized value about 95% of the continuously coated fibers had a coating thickness range about 0.030-0.10 μm and above this showed isolated dendrite deposit of copper as shown in Figure 1 (d). Figure 2, (a) and (b), shows the EDS pattern of uncoated and copper coated basalt fiber, respectively. Micrographs clearly reveal 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 exhibits a glossy surface. It can also be seen via the micrographs that, after successful coating, the surface appears dim. The EDS results confirm the presence of Cu, indicating a successful activation, sensitization and metallization process.

Table 5. Results of experimental & GP value

Exp. No.	Target Output	GP Output
1	0.09075	0.09094
2	0.0552	0.04679
3	0.0457	0.04972
4	0.0541	0.04989
5	0.0465	0.03625
6	0.04295	0.04440
7	0.10435	0.10183
8	0.0617	0.05787
9	0.1048	0.10528
10	0.0981	0.07737
11	0.0376	0.04920
12	0.061	0.06498
13	0.0993	0.08804
14	0.0575	0.04487
15	0.04424	0.04544
16	0.0984	0.08596
17	0.035	0.05115
18	0.08395	0.07459
19	0.0799	0.07454
20	0.1112	0.10222
21	0.0722	0.08192
22	0.0536	0.06834
23	0.0685	0.07238
24	0.0394	0.05499

4.2. GP Results

GP results can be used as conformity test to validate the experimental values. For an easy understanding and an explicit depiction, plots were made for each output parameter. Plots made show the trend of the prediction capability of the model in both the training set and validation set of data; further the plots exhibit the trend of variables interaction in the process. This work resulted in developing a model for the coating parameters in a scientific manner with a reliability of 98%. This work gains significance in the sense with a reasonably minimum number of experiments (Figure 3), a reliable model has been generated, validated and, further, the process has been optimized

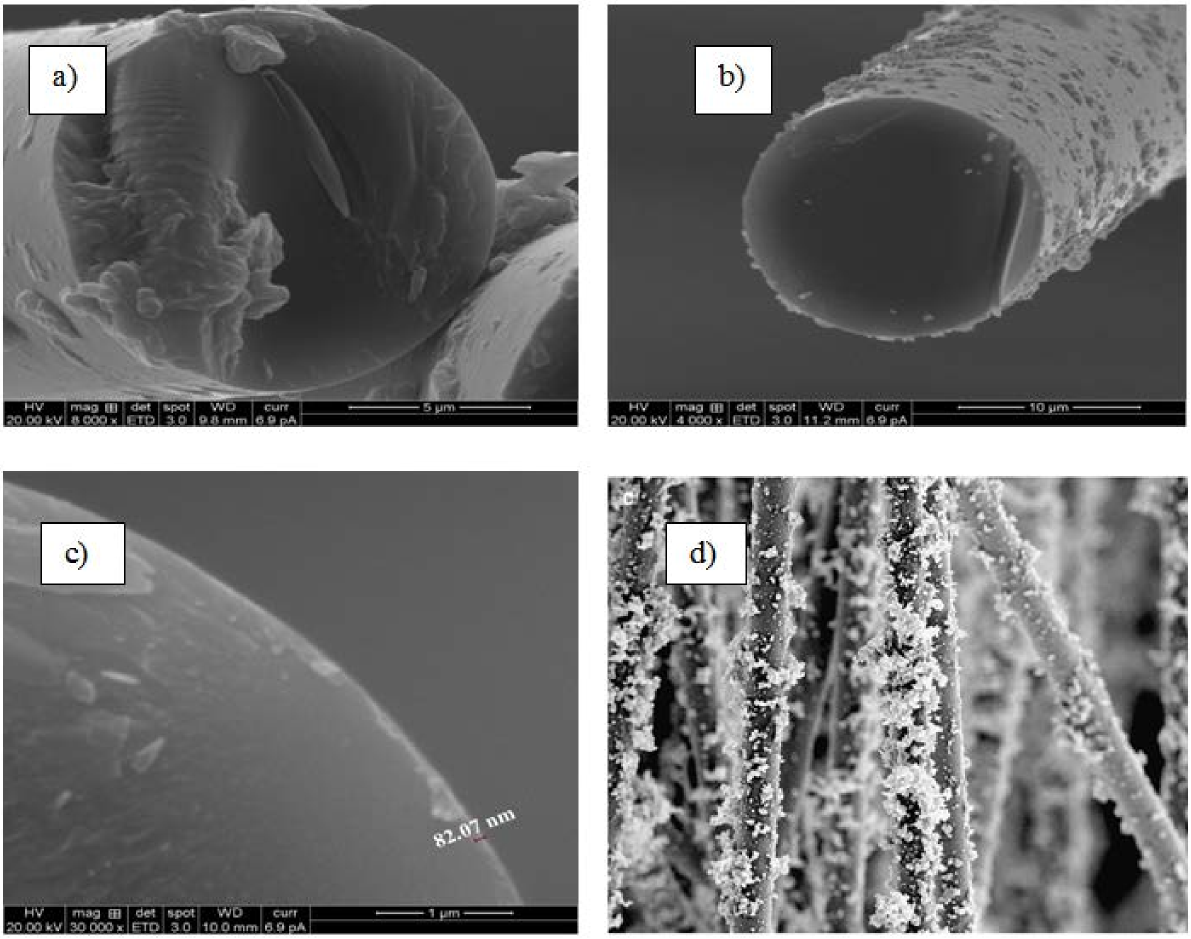

Figure 1. SEM micrographs (a) Uncoated basalt fiber, (b) Lower magnification copper coated basalt fiber, (c) Higher magnification copper coated basalt fiber, and (d) Isolated dendrite deposit of copper

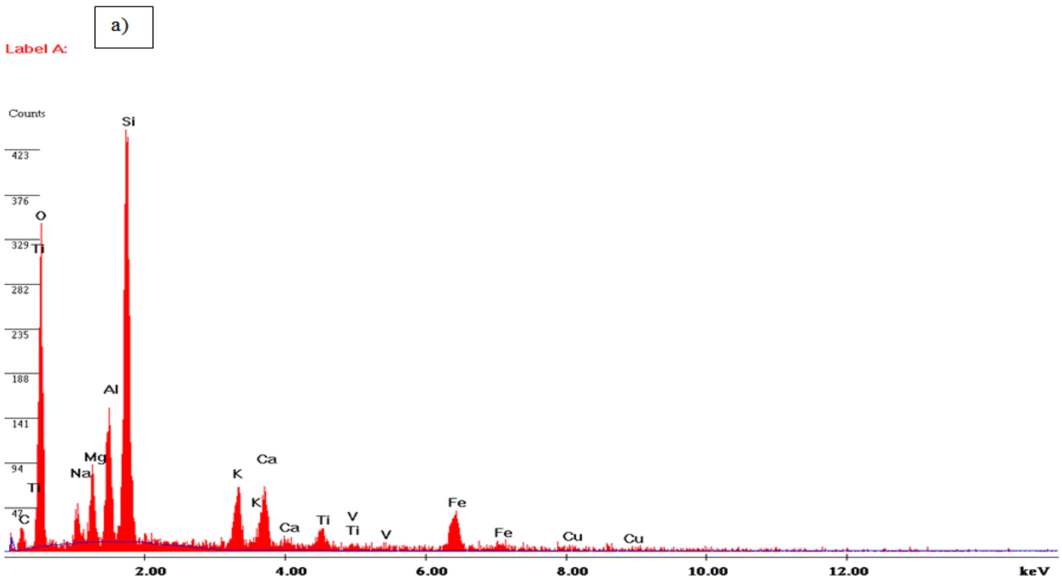

Figure 2. EDS image pattern (a) Uncoated basalt fiber.

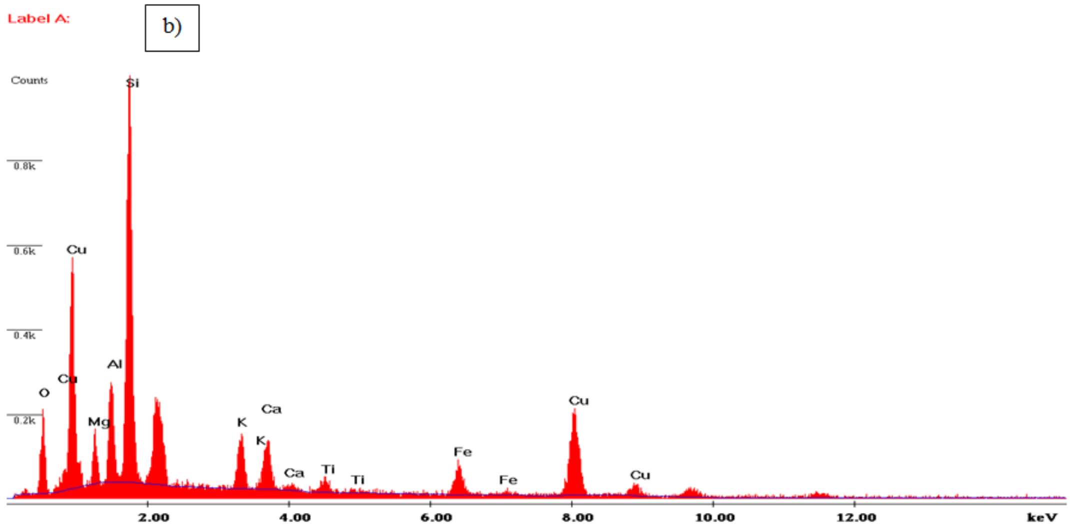

Figure 2. EDS image pattern (b) Copper coated basalt fiber

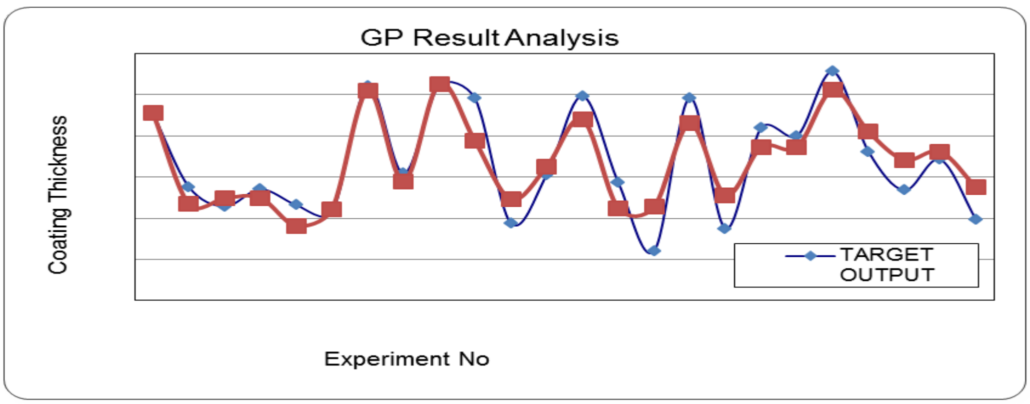

Figure 3. GP result analysis

5. Conclusion

The genetic models developed for optimizing a coating parameter (i.e., Sensitization time, Activation time and Metallization time) in electroless solution bath have a high order of fitness quality within the training data sets and also have a good comparison for the validation data sets. Hence, the models are found to be dependable and can be used for all practical purposes in the surface coating of fibers for choosing the set of coating parameters in order to obtain a thin and uniform copper coating on the fiber. Considering the experimental test results and also those of the analytical model results input parameters of V_0=15 min V_1=15 min and V_3=3 min is considered as an optimum set of parameter. It is also noted that this set of parameters has a good comparison of predicted values with that of experimental values. Genetic programming is considered to be time-consuming since the number of the taken iterations is fairly large; however, it is worth adopting this method, considering the prediction-accuracy of the model developed both in the case of training and validation purposes. Capability of the genetic models for predicting the responses of a process is extremely good since they have a higher accuracy compared to the performance of other analytical methods.

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