

On the wear properties of Nylon6-SiC-Al₂O₃ based fused deposition modelling feed stock filament



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ABSTRACT

Fused deposition modelling (FDM) is one of the commonly used additive manufacturing (AM) technologies. In commercial FDM setup a plastic material based filament (usually of ABS) is unwound from a coil to produce functional/non-functional prototypes. The application domain of FDM is limited presently, because of selective material availability in commercial market as regards to the wear of functional prototypes is concerned. In order to enhance the application domain of commercial FDM setup, an effort has been made to develop new polymer composite (with hybrid reinforcement) wire of Nylon6-SiC-Al₂O₃ with enhanced wear resistant properties. In this research work experimental investigations has been carried out for optimizing wear properties of Nylon6-SiC-Al₂O₃ based feed stock filament of FDM. Further mathematical model for above mentioned property has been developed and counter verified as a case study.

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1. Introduction

During the last decade FDM has emerged as a widely used AM technology for various engineering applications [1]. In commercial FDM setup usually ABS material is used to prepare non-functional prototypes mainly intended for presentation purposes [2]. So many studies have been reported for FDM process parameter optimization, part and surface quality, dimensional features etc. [3–5]. But very less has been reported to increase the functional domain of FDM. Further the literature review reveals that some researchers have developed the filament wires as feed stock filament of FDM by taking a single reinforcement in polymer matrix to improve the mechanical properties (like: tensile strength, Young's modulus, percentage elongation etc.) [6,7]. But hitherto no work has been reported on the wear properties of FDM feedstock filament wire prepared by using

hybrid reinforcement. In the present work new polymer composite wire (with hybrid re-enforcement) of Nylon6-SiC-Al₂O₃ has been prepared to attain wear resistant property of functional/non-functional prototypes. Fig. 1 shows the schematic of FDM process.

The feed stock filament enters in form of wire through drive wheels into the liquefier assembly, covered with heating coils, thereby increasing the temperature of wire and changing the state of wire from solid to semi-liquid state. Further, this semi-liquid wire is expelled out from the nozzle of FDM machine and starts depositing in form of layers.

Melt flow index (MFI) is one of the standard tests that serve as basis of using feed stock filament wire in FDM machine. MFI defines the ease with which a thermoplastic polymer can flow. Fig. 2 shows the basic schematic of MFI tester. In the present study FDM setup of Stratsys USA (u-Print) has been used. In this commercial FDM setup ABS, P-430, feed stock filament wire having MFI 2.41 g/10 min is used [9]. In this research work, hybrid reinforced wire has been prepared having MFI near to MFI of OEM (original equipment manufacturer) wire. Pilot experimentation has been performed for finding MFI values by selecting different proportions of SiC and Al₂O₃ in Nylon6 matrix.

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The Nylon6 matrix and reinforcement is mechanically mixed and placed in an electric oven to remove any type of moisture present in the mixture. The mixture is then loaded into the barrel of MFI tester and is heated up to standard temperature of 230°C as per ASTM standard (D 1238-95). The mixture compositions were selected by short listing the MFI values near to 2.41 g/10 min and fed into screw extruder machine to draw the wires. These wires were made to run in FDM machine and standard specimens of length 30 mm and diameter 10 mm were prepared. These specimens were mounted on PIN ON DISK machine to perform wear testing. Fig. 3 shows basic schematic of PIN ON DISK machine. Specimens of standard dimensions were mounted in holder and made to slide on rotating E31 disk covered with emery paper to have appreciable amount of wear.

2. Experimentation

In this research work, an effort has been made to enhance the application domain of FDM by increasing the availability of the different wires with enhancement in wear properties. For this, a pilot experimentation was designed to perform a certain set of experiments. Table 1 shows the different compositions selected for experimentation.

The Nylon6 was used as binder material (E-35grade; particle size 4 mm; specific density-1.2 g/cm³) in a granular form having average particle size 4–5 mm. The reinforced material such as SiC (particle size 149 μm, 125 μm and 106 μm) and Al₂O₃ (particle size 149 μm, 125 μm and 106 μm) were used in the present study. The selected proportions of composite were mixed manually. After performing pilot experimentation (see Table 1), it was observed that MFI of different proportions of reinforcements in Nylon6 matrix materials were varying appreciably, when SiC and Al₂O₃ of different grain size were used.

Based upon Table 1 it was decided to carry forward the final experimentation with SiC and Al₂O₃ of 125 μm size. So, final wire drawing process on screw extruder machine was completed with the highlighted values in Table 1. The highlighted proportions of reinforcements in Nylon6 matrix in Table 1 have values of MFI near to OEM wire. So these proportions have been selected for preparation of wire on single screw extruder. Table 2 shows various input parameters which were kept fixed on screw extruder machine for preparing the wire filament of FDM.

Finally feed stock filament (in form of wires) prepared on single screw extruder were made to run on FDM machine and specimens in form of pins of length 30 mm and 10 mm diameters were prepared (see Fig. 4).

The wear test was carried out on pin on disk arrangement (DUCOM TL-20) as per ASTM G 99 standard under dry sliding

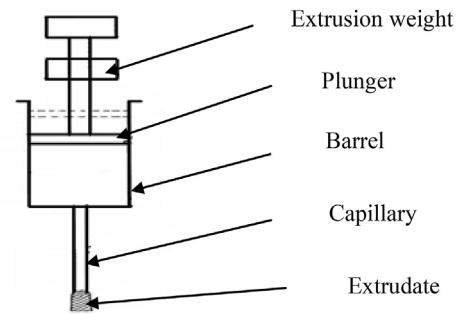


Fig. 2. Schematic of MFI tester [10].

conditions at room temperature. Many researchers have reported that polymers and polymers composites when sliding against steel counter face offered less wear due to the formation of thin protective film (Nagaraju et al., 2011) [12]. The counter sliding surface for pin was prepared by pasting silicon carbide (SiC) paper (600 grit size) on steel disc (EN-32; Hardness 65 HRC). Wear test was performed with sliding speed of 190 rpm, 80 mm track diameter and 5 N load for 10min test run. Wear value for standard ABS is 409 μm under same boundary condition used for this research work. The machine was equipped with data acquisition system. The value of wear obtained from wear testing is given in Table 3.

3. Results and discussion

Volume loss of material generally caused due to abrasion and adhesion phenomena and normally it starts with abrasion (Nagaraju et al., 2011) [12]. The pins based on composite material shows very less amount of wear as compared to the ABS pins. Hence it can be seen from the wear tracks and value obtained from the wear testing. The wear tracks were obtained to study wear phenomena and extent of wear. Fig. 5 and Fig. 6 shows the wear tracks obtained from wear testing of Nylon6-SiC-Al₂O₃ and ABS respectively.

Here it can be seen from comparison of Figs. 5 and 6 that extent of wear for ABS is much more than composite material based pin. These results are in line with the observations made by other investigators [1–15]. Further, SEM analysis was carried out to see the wear mechanism. Fig. 7a and Fig. 7b shows the SEM analysis for ABS and composite material.

As observed from Fig. 7b, the dominant factor for wear in composite material is abrasion and adhesion. When material slide

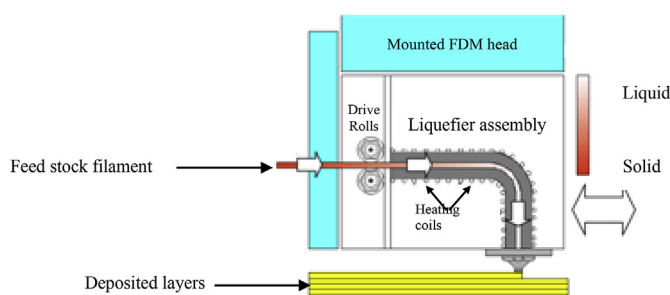


Fig. 1. Schematic of FDM process [8].

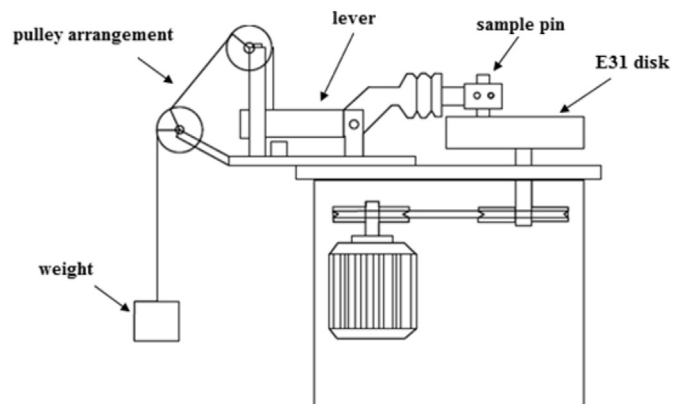


Fig. 3. Schematic of PIN ON DISK machine.

Table 1
Observations of Pilot experimentation for MFI.

Nylon 6 (Proportion by weight %age)	SiC (Proportion by weight %age)			Al ₂ O ₃ (Proportion by weight %age)			MFI (g/10 min)
	Grain Size			Grain Size			
	149 μm	125 μm	106 μm	149 μm	125 μm	106 μm	
50	25	—	—	—	25	—	4.078
50	—	—	25	—	25	—	4.0775
50	25	—	—	25	—	—	4.33
60	20	—	—	20	—	—	8.97
55	11.25	11.25	—	11.25	11.25	—	7.08
65	8.75	8.75	—	8.75	8.75	—	7.42
70	5	5	5	5	5	5	4.88
75	4.17	4.17	4.17	4.17	4.17	4.17	6.54
50	—	5	—	—	45	—	1.55
50	—	10	—	—	40	—	2.42
50	—	20	—	—	30	—	2.72
50	—	30	—	—	20	—	2.9
40	—	30	—	—	30	—	1.14
45	—	25	—	—	30	—	1.84
45	—	30	—	—	25	—	2.36
50	—	25	—	—	25	—	2.76
55	—	25	—	—	20	—	3.149
60	—	20	—	—	20	—	4.613
70	—	15	—	—	15	—	7.501
50	—	15	—	—	35	—	2.6

against the emery paper, which was pasted on the rotating disk, material start delaminating from the pin and grooves were formed on the surface and the wear was majorly reduced due to presence of abrasive particle in composite material marked in dark black circles.

After calculating the wear for the composite material, 'Design expert' software was used to model the wear results for the different pins. The 'Mixture' module of response surface methodology tool was used to model the results. This mathematical tool basically helps to develop an empirical relation between different results of different composition. The values of wear as per Table 3 were put in to this tool and analyzed. Table 4 shows ANOVA for mixture quadratic model.

The Model F-value of 333.22 implies that the model is significant. There is only a 0.30% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case, Linear Mixture Components, AC, BC are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. The values of R-values along with mean and standard deviation values are given in Table 5.

"Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Theratio of 47.743 indicates an adequate signal. This model can be used to navigate the design space. The values of different model components and with the values of their standard errors are listed as in following Table 6.

After complete analysis by Design expert software, following equations in terms of actual components were given as following:

Final equation in terms of actual components:

$$\begin{aligned} \text{Wear} = & -6338.60000 * A + 653.40000 * B - 24238.60000 * C \\ & + 6000.00000 * A * B + 58960.00000 * A * C \\ & + 18400.00000 * B * C \end{aligned} \quad (1)$$

where A,B,C are the notation given to Nylon6, SiC, Al₂O₃ respectively. This empirical relation helps to model the wear for any tailor made application. To find the desired value of wear, composition of A,B,C may be varied accordingly.

After development of wear model feasibility of this model was counter checked.

Corollary:

A (Nylon -6) = 0.50

B (SiC) = 0.10

C (Al₂O₃) = 0.40



Fig. 4. Pins prepared on FDM.

Table 2
Fixed input parameters of single screw extruder

Die head temperature (in °C)	Barrel Temperature (in °C)	Screw speed (in rpm)	Take up speed (in rpm)
180	200	15	20

Table 3
 Observation of wear at selected proportions of reinforcements. (Note: the experimental conditions at S. No. 1–9 have been selected from highlighted values of Table 1 with MFI value near to 2.41 g/10 min).

S. No.	Nylon 6 (in % by weight) A	SiC 125 μm (in % by weight) B	Al ₂ O ₃ 125 μm (in % by weight) C	Wear (μm)
1.	50	10	40	28
2.	50	20	30	242
3.	50	25	25	199
4.	50	30	20	81
5.	45	25	30	54
6.	45	30	25	107
7.	55	25	20	60
8.	60	20	20	51
9.	50	15	35	50
10.	^a ABS	0	0	409

^a Signifies properties of standards ABS material taken for reference with MFI of 2.41 g/10 min.

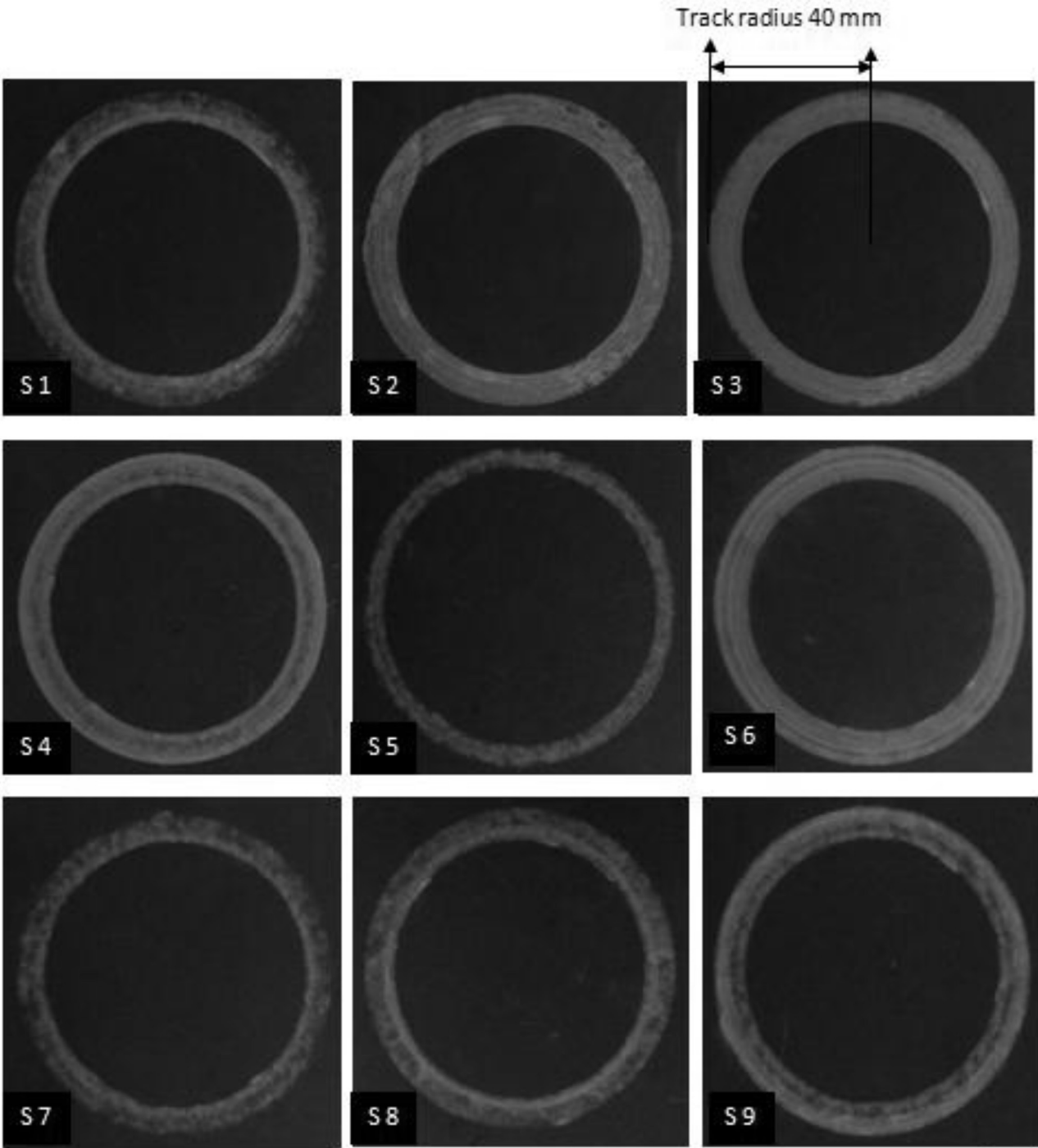


Fig. 5. Wear track images from specimen testing.

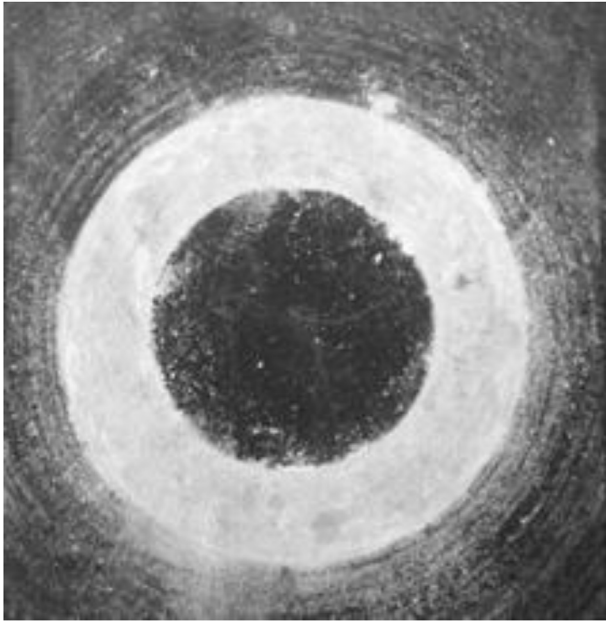


Fig. 6. Wear track for ABS pin.

Table 4

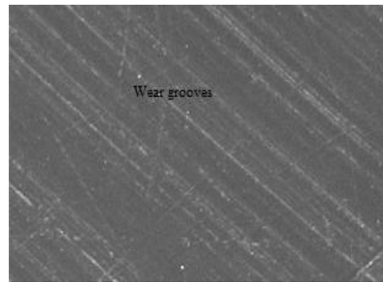
ANOVA for Mixture Quadratic Model (Analysis of variance table [Partial sum of squares]).

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F
Model	42652.30	5	8530.46	333.22	0.0030
Linear Mixture	–12007.67	2	–6003.83	–234.52	1.0000
AB	103.62	1	103.62	4.05	0.1819
AC	24932.35	1	24932.35	973.92	0.0010
BC	29624.00	1	29624.00	1157.19	0.0009
Residual	51.20	2	25.60		
Cor Total	42703.50	7			

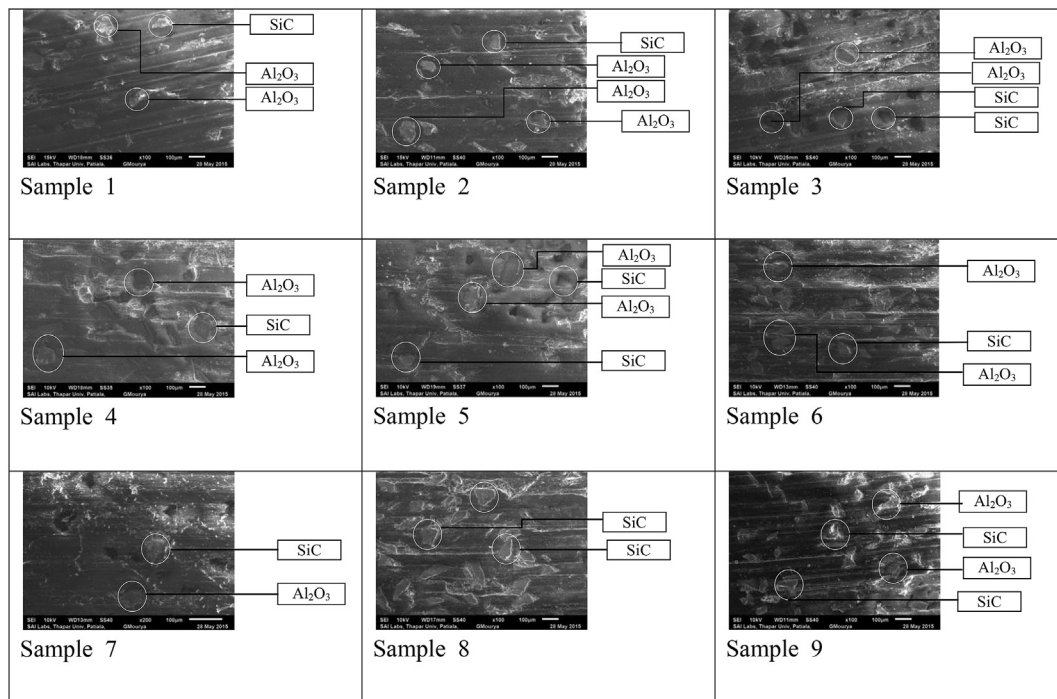
Table 5

Statistical analysis for wear model.

Std. Dev.	5.06
Mean	118.75
C.V.	4.26
PRESS	N/A
R-Squared	0.9988
Adj R-Squared	0.9958
Pred R-Squared	N/A
Adeq Precision	47.743



a.SEM image for ABS



b.SEM image for composite material

Fig. 7. a SEM image for ABS. b SEM image for composite material.

Table 6
Standard error for components.

Component	Coefficient Estimate	DF	Standard Error	95% CI	
				Low	High
A-A	−177.00	1	105.91	−632.68	278.68
B-B	68.00	1	11.63	17.95	118.05
C-C	−657.00	1	30.06	−786.32	−527.68
AB	375.00	1	186.39	−426.99	1176.99
AC	3685.00	1	118.08	3176.94	4193.06
BC	1150.00	1	33.81	1004.54	1295.46

highly wear resistant. The wear model was developed and cross checked for its accuracy. The wire as feed stock filament of FDM with the tailor made wear properties can be easily predicted by the proposed empirical model. The application of the analysed FDM material for the additive manufacturing of a large variety of innovative materials and structures [16–38] will form the subject of future work.

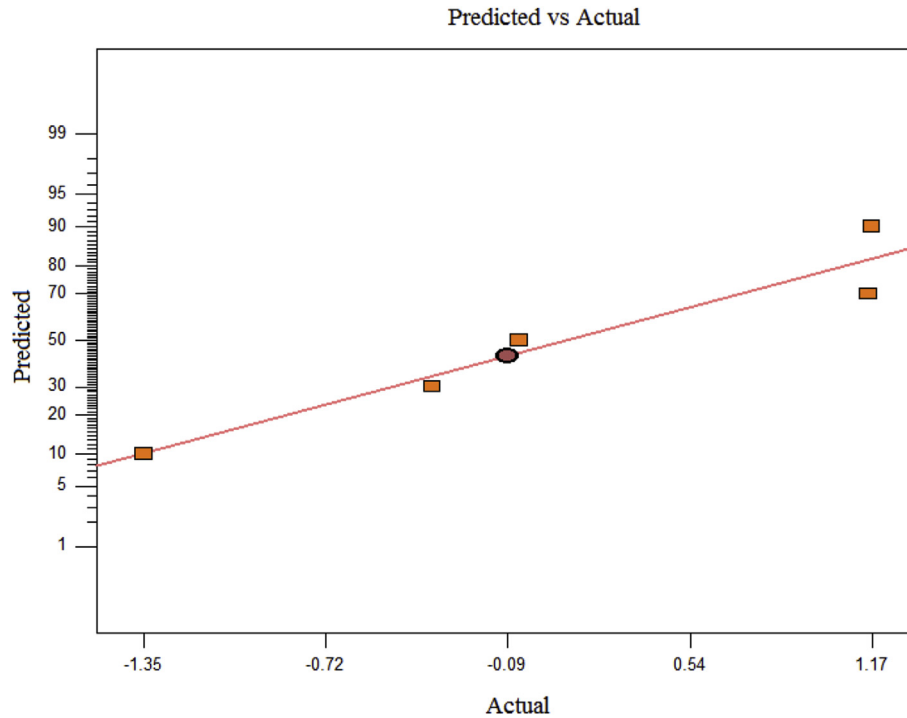


Fig. 8. Graph between actual and predicted values.

Here the value (0.10) means 50% by weight and similarly for others. By putting these values in Eq. (1)

$$\begin{aligned}
 & -6338.60000 * .50 + 653.40000 * .10 - 24238.60000 * .40 \\
 & + 6000.00000 * .50 * .10 + 58960.00000 * .5 * .40 \\
 & + 18400.00000 * .10 * .40
 \end{aligned}$$

The value from equation comes out to be **28.6 μm** and actual value based upon experimental observation is **28 μm** . Fig. 8 shows the graph between actual and predicted values for final model validation.

4. Conclusions

The outcomes for the present work show feasibility of development of FDM wire from alternative material. As ABS wire is having limited wear properties so alternative material can be used to have tailor made properties. In this case a wire from alternative material has been successfully developed and pins were successfully prepared. The value for wear and MFI has been established. A Nylon-6 based feed stock filament was successfully developed with hybrid reinforcement of SiC and Al₂O₃. The prototype/pins of composite material have been developed with high resistance to wear. The wear track obtained in this study shows that material is

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