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Effect of single particle size, double particle size and triple particle size Al₂O₃ in Nylon-6 matrix on mechanical properties of feed stock filament for FDM



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Rupinder Singh ^a, Piyush Bedi ^a, Fernando Fraternali ^{b, *}, I.P.S. Ahuja ^c

^a Department of Production Engineering, Guru Nanak Dev Engineering College, Ludhiana, India

^b Department of Civil Engineering, University of Salerno, Italy

^c Department of Mechanical Engineering, Punjabi University, Patiala, India

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ABSTRACT

Fused Deposition modelling (FDM) is one of the additive manufacturing (AM) technologies used extensively for modelling and prototyping applications. In commercial FDM setup, filament wire is uncoiled from wire spools and plastic based material is supplied to produce the part. The application area of FDM is limited presently due to selective material availability in market. Some researchers have high-lighted the use of reinforced composite wires as FDM filament. But hitherto no work has been reported on the effect of Single particle size (SPS), Dual particle size (DPS), Triple particle size (TPS) of Al₂O₃ (as reinforcement) in Nylon-6 matrix to be used as feed stock filament for FDM. In this paper, effect of SPS, DPS, and TPS of Al₂O₃ (as reinforcement) in Nylon-6 matrix on mechanical properties (like: percent age elongation, tensile strength, yield strength, Young's modulus) has been studied. Further, empirical relations have been developed for above mentioned properties and a surface characteristic of developed wires has been observed with SEM image.

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1. Introduction

In the present competitive environment two major processes are being used to produce prototypes, namely machining and additive manufacturing (AM). Machining is generally more accurate and precise, but it is difficult to produce objects with complicated features/intricate dimensions. In contrast AM can produce objects with complicated features. In recent years, AM processes have been found to be capable of producing industrial products with controllable porosity [1], which allows materials to be used more efficiently [2]. Reducing the time to produce prototypes is a key to speeding up the development of new products. Today's commercially available AM systems work with different techniques by using paper, polymers and waxes etc. as process consumables [3]. FDM is one of the AM technique in which plastic/polymer based material usually acrylonitrile butadiene styrene (ABS) is used for preparation of prototypes [4–6].

The FDM system (used in the present study), developed by

Stratasys Inc. USA (one of the commercial manufacturer), currently fabricate parts of elastomers, ABS and investment casting wax using the layer by layer deposition of extruded material through a nozzle using feedstock filaments from a spool [6]. Several studies have been reported to improve the part accuracy, surface finish, strength, etc. by proper adjustment of process parameters [7,8]. Since mechanical properties are important for the functional parts, it is absolutely essential to study influence of various process parameters on mechanical properties so that improvement can be made through selection of best settings [9]. Fig. 1 shows the basic schematic of FDM setup.

The feed stock filament is uncoiled from the spool and enters into heated liquefier assembly through feed wheels [11]. The wire changes its state from solid to semi liquid state and is made to eject through nozzle onto base of machine. The ejected material is made to deposit in the form of fine layers [12].

Melt flow index (MFI) is widely accepted as one of the crucial rheological property [13–17] that determines the basis of running in-house developed FDM filament in the machine. MFI is generally expressed in terms of weight (in g) of polymer which will flow per 10min of time period (i.e. g/10min.). It is to be worth noted that the present study has been performed by taking MFI as a crucial base



^{*} Corresponding author. E-mail address: f.fraternali@unisa.it (F. Fraternali).



Fig. 1. Schematic of FDM [10].

property among other rheological properties as because it is very convenient and cost effective method used in field applications. The present study has been performed on commercial FDM setup (Stratsys, USA, u- Print model) on which filament wire of ABS P430 material (having MFI 2.41 g/10min.) is used. So, the present work is focused on developing alternate feed stock filament wire with MFI comparable to 2.41 g/10min. by reinforcing various combinations of sizes (SPS, DPS, TPS) of Al₂O₃ in Nylon-6 matrix and thereby observing their effect on mechanical properties of developed wire. It should be noted that the SPS represents single particle size (of 100 µm), DPS represents two particle sizes in equal proportion by weight (of 100 μ m and 120 μ m) and TPS represents three particle sizes in equal proportion by weight (of 100 µm, 120 µm and 150 µm). It has been observed that in reported literature, researchers have studied only the effect of SPS and very less exploration is done in the field of study of mechanical properties considering DPS and TPS. The above stated sizes of Al₂O₃ are taken, considering the diameter of nozzle head of FDM setup used, as if the size of particles more than 150 µm can choke the nozzle. Fig. 2 shows a basic schematic of MFI tester.

Various combinations of Nylon-6 granules along with reinforcements of Al_2O_3 as per pilot experimentation were mechanically mixed and placed in electric oven to eliminate any type of moisture present. It is to be noted that Nylon-6 has been taken as matrix material, as Nylon 6 has properties similar to that of commercial ABS material but the cost of Nylon-6 is very low than that of ABS. At high temperatures (especially during investment casting applications at de-waxing/de-plasticising stage), ABS produces ultrafine particles which have bas effects on human heath as well as on environment. Furthermore, Al₂O₃ was used as reinforcement as it is considered to be a good for draw ability properties. It is to be noted that various combinations/proportions of the nylon matrix and reinforcements are considered so that MFI of the total mixture may come nearer to that of commercial ABS material, i.e., 2.41 g/ 10min. The parent material/Nylon-6 granules and reinforcement is mechanically mixed and placed in an electric oven to eliminate any type of moisture present in mixture. The mixture is then put into the pre-heated barrel of MFI tester. The weight as per the ASTM standard (D 1238-95) is put on the piston to expel the molten material from barrel and thereby made to exit out of die opening as extrudate and weighed to find MFI in terms of gm/10 min. The mixture compositions were selected by selecting the MFI values near to 2.41 g/10min and fed into single screw extruder machine to draw the wires. Manufactured wires were tested using two column Universal testing machine (UTM) and mechanical parameters like Percent age elongation, Young's Modulus, Yield stress etc. were determined. Fig. 3 shows 3D view of UTM machine being used.

2. Experimentation



Fig. 2. Schematic of MFI tester [14].

Present work is focused on enhancement of application area of FDM machine by developing filament wire, which has tailor made properties. For this experimentation work, set of pilot experiments (test runs) has been designed. Annexure 1 shows MFI values of



Fig. 3. 3D view of UTM and its parts.

different set of compositions/proportions for pilot experimentation.

The highlighted compositions/proportions in 51×1 were judicially selected for final experimentation as these compositions were minimum MFI value in their respective categories which were also close to 2.41 g/10min (keeping in view MFI of P430, OEM filament wire). The selected average MFI values are given in Table 1. It should be noted that in order to reduce the experimental error three runs were performed.

These selected compositions/proportions were fed into single screw extruder machine to manufacture the filament wire. Fig. 4 shows schematic of screw extruder machine used.

Various input parameters which were kept fixed on screw extruder machine for manufacturing the filament wire were:

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

The variable input parameter in this study is only composition/ proportion of reinforcement. The compositions/proportions of all grades of Al₂O₃ (100, 120, 150 grades) were varied. The output parameters considered in the study were:

- Percentage elongation
- Tensile strength (kg/sq.mm)
- Yield strength (kg/sq.mm)
- Young's modulus (kg/sq.mm)

Table 2 summarizes average of three runs for various mechanical properties (like: Percentage elongation, Tensile strength, yield strength and Young's modulus) of selected 10 compositions (as per Table 2) as tested on UTM. The tensile properties of feed stock filament wires were tested as per ASTM-638 standard. The mechanical properties tested are rate dependent and are tested at strain rate of 50 mm/min.

After determining mechanical properties, spools of wire were installed onto FDM machine and pins of defined diameter (10 mm) were prepared. The pins were made at solid density option and at zero degree orientation as per commercial software of Stratasys Inc. USA (one of the commercial manufacturer). Fig. 5 and Fig. 6 show 3D view of Stratasys FDM machine and prepared pins on FDM respectively.

3. Results and discussions

Table 1

From Table 2, maximum values for each mechanical property was taken for each type of particle size (i.e, from SPS, DPS and TPS). Table 3 summarizes the maximum values of different mechanical properties with variation in composition of different particle sizes. It can be clearly identified that SPS gives the best percentage

MFI of selected compositions (average of three experimental runs).

elongation while DPS proved to be the best for other mechanical properties (tensile strength, yield strength, young's modulus, etc.)

The plots of different mechanical properties with change in particle sizes are given as in Figss. 7-10.

From Fig. 7, it can be observed that percentage elongation is decreasing as particle size is changing from SPS to TPS. This trend is quite obvious because as no. of particles are increasing; strength of material is also increasing due to which percentage elongation obviously will be decreased.

From Fig. 8, it can be seen that tensile strength first increases and attain its maximum value for DPS and then it again decreases and achieve its minimum value for TPS. The tensile strength is maximum for DPS may be because of uniform mixing of two sizes of particles. Further, it was decreased for TPS because of exceeded limit of volume of base polymer to accumulate more particles for TPS.

From Fig. 9, it can be seen that yield strength first increases and attain its maximum value for DPS and then it again decreases for TPS. The yield strength is maximum for DPS because of mixing of two sizes of particles. Further, it was decreased for TPS because of exceeded limit of volume of base polymer to accommodate more particles for TPS.

From Fig. 10, it can be seen that Young's modulus first increases and attain its maximum value for DPS and then it again decreases and achieve its minimum value for TPS. The Young's modulus is maximum for DPS because of mixing of two sizes of particles. Further, it was decreased for TPS because of exceeded limit of volume of base polymer to accommodate more particles for TPS.

Further SEM analysis of all pins were carried out which ensures the uniform presence of every single type of particle used in the subsequent mixture thereby ensuring the cohesion between the printed layers. Figs. 11–13 shows the SEM analysis of sample pins for SPS, DPS and TPS.

It can be clearly observed from above SEM images that DPS particles are very well diffused into the base polymer matrix as compared to SPS and TPS due to which DPS filament is exhibiting better mechanical properties than SPS and TPS.

Further, data from Table 2 was put into the Design Expert software. Design—Expert is a statistical software package from Stat-Ease Inc. which is specifically designed to perform design of experiments (DOE). It offers comparative tests, screening, characterization, optimization, robust parameter design, mixture designs and combined designs. It provides test matrices for screening up to 50 factors. Statistical significance of these factors is established with analysis of variance (ANOVA). Graphical tools help to identify the impact of each factor on the desired outcomes and reveal abnormalities in the data. Based on Table 2, and by applying Mixture Design (User defined model as 4 components and 4 responses) ANOVA for mixture quadratic model was conducted (See Table 4).

The Model F-value of 8.47 implies that the model is significant. There is only a 1.41% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate

	Nylon 6 (in wt%)	Al ₂ O ₃ 100 grade (in wt%)	Al ₂ O ₃ 120 grade (in wt%)	Al ₂ O ₃ 150 grade (in wt%)	MFI (g/10min.)
SPS	50	0	50	0	5.23
	60	0	40	0	6.61
DPS	50	25	25	0	4.29
	60	20	20	0	4.72
	50	25	0	25	2.82
	60	20	0	20	4.15
	50	0	25	25	3.25
	60	0	20	20	5.82
TPS	50	16.66	16.67	16.67	4.12
	60	13.34	13.33	13.33	4.80



Fig. 4. Schematic of single screw extruder machine [15].

 Table 2

 Mechanical properties of different prepared filament wires (average values of three repetitions).

	Nylon 6 (in wt%)	Al ₂ O ₃ 100 grade (in wt%)	Al ₂ O ₃ 120 grade (in wt%)	Al ₂ O ₃ 150 grade (in wt%)	Percentage elongation	Tensile strength (kg/ sq mm)	Yield strength (kg/sq mm)	Young's modulus (kg/ sq mm)
SPS	50	0	50	0	16.63	2.68	0.33	69.82
	60	0	40	0	10.9	3.99	0.96	55.10
DPS	50	25	25	0	6.88	3.25	0.74	88.90
	60	20	20	0	5.45	3.38	0.31	70.22
	50	25	0	25	5.35	2.63	1.65	47.12
	60	20	0	20	3.55	3.71	0.99	36.70
	50	0	25	25	15.6	4.54	1.14	28.10
	60	0	20	20	7.2	4.51	3.18	21.68
TPS	50	16.66	16.67	16.67	8.83	3.12	1.82	55.71
	60	13.34	13.33	13.33	4.7	2.35	1.78	43.14
Pure	(Nylon-6)	-	-	-	2-150	4-33	-	112-1630
ABS	(P-430)	-	-	-	3	3.77	-	232

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 R-values along with mean and standard deviation values are given

in Table 5.

After excluding a one outlier value from the model in design expert, Table 5 was obtained showing various R-squared values. R squared value is a statistical measure of how close the data are to the fitted regression line. It is also known as the coefficient of determination. As per Table 5, it is coming out to be 0.9824 which is satisfactory value and signifies that the statistical model developed yield a good fit.

The adjusted R-squared compares the explanatory power of regression models that contain different numbers of predictors. It increases only if the new term improves the model more than



Fig. 5. Stratasys FDM machine.



Fig. 6. Manufactured pins on FDM.

Table 3

Variation in mechanical properties with change in type of particle sizes.

Particle type	Nylon 6 (in wt%age)	Al ₂ O ₃ -100 grade (150 micron) (in wt%age)	Al ₂ O ₃ -120 grade (120 micron) (in wt%age)	Al ₂ O ₃ — 150 grade (100 micron) (in wt%age)	
					Percentage elongation
SPS	50	0	50	0	16.63
DPS	50	0	25	25	15.6
TPS	50	16.66	16.67	16.67	8.83
					Tensile strength (in kg/sq.mm)
SPS	60	0	40	0	3.99
DPS	50	0	25	25	4.54
TPS	50	16.66	16.67	16.67	3.12
					Yield strength (in kg/sq.mm)
SPS	60	0	40	0	0.96
DPS	60	0	20	20	3.18
TPS	50	16.66	16.67	16.67	1.82
					Young's Modulus (in kg/sq.mm)
SPS	50	0	50	0	69.82
DPS	50	25	25	0	88.90
TPS	50	16.66	16.67	16.67	55.71







Fig. 8. Plot for tensile strength with change in particle size.

would be expected by chance. It decreases when a predictor improves the model by less than expected by chance. The adjusted R-squared can be negative, but it's usually not. It is always lower than the R-squared. In the present study, in Table 4, it is coming out to be 0.9718 which is lesser than that of R-squared value.



Fig. 9. Plot for yield strength with change in particle size.



Fig. 10. Plot for Young's modulus with change in particle size.

The predicted R-squared indicates how well a regression model predicts responses for new observations. If predicted R-squared that is much lower than the regular R-squared, there are chances that the model contains too many terms. In the present study, as



Fig. 11. SEM image of Pin 1 (SPS) at 100×.



Fig. 12. SEM image of Pin 7 (DPS) at $100 \times$.



Fig. 13. SEM image of Pin 9 (TPS) at $100 \times$.

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 Linear mixture	167.55 167.55	3 3	55.85 55.85	8.47 8.47	0.0141 0.0141
Residual Cor total	39.56 207.11	6 9	6.59		

ble	5
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austical allalysis.	
Std. dev.	0.79
Mean	8.77
R-squared	0.9824
Adj R-squared	0.9718
Pred R-squared	0.9381
Adeq precision	24.910

per Table 5, it is coming out to be 0.9381 which is in reasonable agreement with the adjusted R-squared value.

"Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 24.910 indicates an adequate signal. This model can be used to navigate the design space.

After complete analysis by design expert, model equations in terms of actual component is given as under:

Final Equation in Terms of Actual Components:

For Percentage elongation = | - 14.29722 A + 17.76623 B + 46.71259 C + 34.04853 D|

where, A is composition of Nylon 6

B is composition of Al₂O₃ (Grade 100)

C is composition of Al_2O_3 (Grade 120)

D is composition of Al₂O₃ (Grade 150)

After development of percentage elongation model, feasibility of this model was counter checked.

COROLLARY. : For Eg., for last composition in Table 5 in which

 $\begin{array}{l} A(Nylon-6)=0.6\\ B(Al_2O_3\ grade\ 100)=0.1334\\ C(Al_2O_3\ grade\ 120)=0.1333\\ D(Al_2O_3\ grade\ 150)=0.1333\\ Percentage\ elongation=-14.29722(0.6)+17.76623(0.1334)\\ +46.71259(0.1333)+34.04853(0.1333)=\textbf{4.55}(\textbf{approx.}),\\ \textbf{which is approximately near to observed value of\ 4.7 \end{array}$

for same composition

Similarly, following the same procedure as above, the empirical relations for other mechanical properties were developed.

For Tensile Strength

The prepared model was analysed for tensile strength and linear model was proposed for tensile strength, the equation of which is developed as under:

$$\label{eq:constraint} \begin{split} \textbf{Tensile strength} &= |0.60983 \text{ A} + 8.24141 \text{ B} + 5.33765 \text{ C} \\ &+ 1.95317 \text{ D}| \end{split}$$

where, A is composition of Nylon 6 B is composition of Al₂O₃ (Grade 100) C is composition of Al₂O₃ (Grade 120) D is composition of Al₂O₃ (Grade 150)

COROLLARY. : For Eg., for last composition in Table in which

for same composition For Yield Strength The prepared model was analysed for yield strength and linear model was proposed for yield strength, the equation of which is developed as following:

Yield Strength = |2.66776 A - 0.74963 B - 1.41875 C

+ 2.78802 D|

where, A is composition of Nylon 6 B is composition of Al₂O₃ (Grade 100) C is composition of Al₂O₃ (Grade 120) D is composition of Al₂O₃ (Grade 150)

COROLLARY. : For Eg., for last composition in Table 5 in which

for same composition **For Young's Modulus**

The prepared model was analysed for Young's Modulus and linear model was proposed for Young's Modulus, the equation of which is developed as following:

Young's Modulus = | - 4.44512 A + 220.15468 B + 144.26259 C - 22.85559 D|

where, A is composition of Nylon 6

B is composition of Al_2O_3 (Grade 100) C is composition of Al_2O_3 (Grade 120)

D is composition of Al_2O_3 (Grade 150)

COROLLARY. : For Eg., for last composition in Table 5 in which

 $\begin{array}{l} A(Nylon-6)=0.6\\ B(Al_2O_3\ grade\ 100)=0.1334\\ C(Al_2O_3\ grade\ 120)=0.1333\\ D(Al_2O_3\ grade\ 150)=0.1333\\ Young's\ Modulus=-4.44512(0.6)+220.15468(0.1334)\\ +144.26259(0.1333)-22.85559(0.1333)=42.88(approx.),\\ which\ is\ approximately\ near\ to\ observed\ value\ of\ 43.14\ for\ same\ composition \end{array}$

4. Conclusions

The conclusions drawn from the present study are:

Alternate feed stock filaments with Al₂O₃ reinforcements as SPS, DPS and TPS in Nylon 6 matrix have been successfully developed. The MFI of feed stock filaments prepared with SPS, DPS and TPS were made comparable to the ABS filament used conventionally in FDM.

The mechanical properties (like: Percentage elongation, Young's Modulus, Tensile strength, Yield strength) have been optimized to increase the application domain FDM. The empirical relations for mechanical properties have been successfully developed and counter verified. Finally it is concluded that in-house prepared FDM feed stock filament with tailor made properties can be successfully used (based upon industrial applications).

We address to future studies the development of highperformance nylon-6 fibers and bars for the reinforcement of cementitious materials [18–24], and the assembly of mechanical metamaterials [25–27], through a closed-loop approach including the computational design and the additive manufacturing of physical models via FDM.

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Annexure 1: MFI values of pilot experimentation.

S.	No	Α	В	С	D	E	А	В	С	D	E	А	В	С	D	E
		SPS	;				DP	S				TPS	5			
		50	0	0	50	14.22	50	0	5	45	8.92	50	5	5	40	24.45
		50	0	50	0	5.23	50	0	10	40	8.54	50	5	10	35	22.24
		50	50	0	0	8.9	50	0	15	35	6.25	50	5	15	30	16.58
		60	0	0	40	15.96	50	0	20	30	4.92	50	5	20	25	15.58
		60	0	40	0	6.61	50	0	25	25	3.25	50	5	25	20	17.49
		60	40	0	0	7.8	50	0	30	20	5.55	50	5	30	15	10.26
							50	0	35	15	6.11	50	5	35	10	9.48
							50	0	40	10	5.25	50	5	40	5	7.54
							50	0	45	5	10.15	50	40	5	5	14.55
							60	0	5	35	11.98	50	35	5	10	9.59
							60	0	10	30	10.54	50	30	5	15	10.55
							60	0	15	25	5.96	50	25	5	20	12.55
							60	0	20	20	5.82	50	20	5	25	14.87
							60	0	25	15	8.25	50	16.66	16.67	16.67	4.12
							60	0	30	10	8.35	50	15	5	30	12.54
							60	0	35	5	15.55	50	10	5	35	14.59
							50	5	45	0	16.45	50	35	10	5	5.48
							50	10	40	0	12.25	50	30	15	5	6.52
							50	15	35	0	12.35	50	25	20	5	8.54
							50	20	30	0	7.58	50	20	25	5	9.64
							50	25	25	0	4.29	50	15	30	5	10.55
							50	30	20	0	10.29	50	10	35	5	10.54
							50	35	15	0	12.84	60	5	5	30	28.12
							50	40	10	0	13.84	60	5	10	25	25.56
							50	45	5	0	18.52	60	5	15	20	17.55
							60	Э 10	30	0	14.20	60	5	20	15	10.10
							60	10	30	0	14.28	60	5	25	10	10.12
							60	10	20	0	9.65	60	20	50	5	12.59
							60	20	15	0	4.72	60	25	5	J 10	19.40
							60	20	10	0	4.9J 9.45	60	20	5	10	14.57
							60	30	5	0	0.45	60	15	5	20	16.40
							50	5	0	45	3.24 8.12	60	10	5	25	18.88
							50	10	0	40	6 35	60	13 3/	12 22	13 32	4 80
							50	15	0	35	3.45	60	5	5	30	14 54
							50	20	0	30	2.98	60	25	10	5	8.29
							50	25	0	25	2.82	60	20	15	5	9.55
							50	30	0	20	3.25	60	15	20	5	11.59
							50	35	0	15	5.29	60	10	25	5	11 73
							50	40	0	10	5.84	60	5	30	5	14.59
							50	45	0	5	9.8	00	-		-	
							60	5	0	35	9.25					
							60	10	0	30	8.36					
							60	15	0	25	4.92					
							60	20	0	20	4.15					
							60	25	0	15	8.54					
							60	30	0	10	10.59					
							60	35	0	5	12.25					

Where, **A** is Nylon 6 (in wt. %age), **B** is $Al_2O_3 - 100$ grade (150 micron) (in wt. %age), **C** is $Al_2O_3 - 120$ grade (120 micron) (in wt. %age), **D** is $Al_2O_3 - 150$ grade (100 micron) (in wt. %age) and **E** is MFI (g/10min).

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