Solid Freeform Fabrication 2017: Proceedings of the 28th Annual International
Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference
Reviewed Paper

PROCESS – PROPERTY RELATIONSHIPS IN ADDITIVE MANUFACTURING OF NYLON-FIBERGLASS COMPOSITES USING TAGUCHI DESIGN OF EXPERIMENTS.

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Abstract

Composite Filament Fabrication (CFF) process, similar to Fused Deposition Modeling (FDM) can extrude a fiber along with a plastic. The process has two nozzles, one that can extrude Nylon and another that can extrude a fiber such as Fiberglass, Kevlar or Carbon Fiber. The mechanical properties of the parts produced by this process are dependent on the process parameters. To determine the effect of these process parameters and design parts for optimal properties, the relationship needs to be determined. This research works with Nylon-Fiberglass composite material. This study focuses on five different process parameters and their effect on mechanical properties such as tensile strength, elastic modulus and elongation to fracture. 36 experiments based on Taguchi design of experiments methodology are conducted and the analysis of variance of the results is used to find the important parameters. The results show that some process parameters are more significant than others in affecting the mechanical properties. It is found that the fiber volume % in the composite plays the most significant role in the mechanical properties.

Introduction

Additive Manufacturing (AM) is a manufacturing process in which material is added in a layer-by-layer fashion to produce the final part. The process involves slicing a CAD file into thin layers and depositing these layers in a manner so that the final part can be obtained without the use of any molds or dies (Gibson et al., 2015). According to the ASTM 529000-15 Standards (ISO/ASTM52900-15, 2015), there are 7 families of AM processes: vat polymerization, powder bed fusion, binder jetting, material jetting, sheet lamination, material extrusion and directed energy deposition.

Out of these families, the most prominent and widespread usage of AM is in the material extrusion category. Material extrusion involves melting of a material (usually a polymer) and extruding it through nozzle to create layer-by-layer cross section of the desired geometry. Till recently, material extrusion was limited to materials such as ABS, HIPS, PLA and Nylon. However, Markforged Inc. (www.markforged.com) invented a new process called Composite Filament Fabrication (CFF) in 2014 that has brought versatility to this AM family.

CFF process is similar to the typical material extrusion process such as FFF (Fused Filament Fabrication) and FDM (Fused Deposition modeling), except for the fact that it can extrude a fiber along with a plastic. The process has two nozzles, one that can extrude Nylon and another that can extrude a fiber such as Fiberglass, Kevlar or Carbon Fiber (US Patent 20150108677 A1, 2015). The resulting part can have multiple layers of the Nylon along with layers of the fiber embedded between them. The advantages of this process are that the resulting part can have better mechanical properties than a typical FDM or FFF part, the composite created by this process does not need any molds or autoclaves typically required by wet layups, vacuum bagging or resin transfer molding processes and the process can create complex geometries with design freedom of selecting the polymer and fiber layers. Other researchers have done similar work with systems which are based on similar technology but differ from the way the fiber is reinforced. Li et. al. (2016) studied the tensile properties of carbon fiber reinforced polylactic acid (PLA) composites by 3D printing. In their study the carbon fiber was reinforced within the PLA matrix and was not deposited as a separate layer. Leigh et. al. (2012) formulated a conductive filament (termed 'carbomorph') using a conductive carbon black filler in a matrix of a polycaprolactone and used FFF process to manufacture electronic sensors. Mori et. al. (2014) manually laid carbon fiber between the layers of ABS using the FFF process and calculated the tensile and fatigue properties of these parts.

Before the CFF process can be used to create parts that can be used in prototyping and production stages, it is important to understand the interactions between the process parameters and the resulting mechanical properties of the parts. The aim of this research is to create a process – property interaction matrix that can help the designers and production engineers in selecting the optimum parameters for their requirements. Similar work has been done for evaluation of anisotropic properties of ABS in fused deposition modeling (Ahn et. al., 2002).

In this work tensile testing of nylon-fiberglass composites is conducted on a design of experiments matrix based on Taguchi Design. Process parameters such as fill density, fiber volume fraction, fill pattern and fiber-layering technique are varied and the samples are tested for their dimensions, productivity, yield strength, elastic modulus, ultimate tensile strength and elongation. The results are then used to determine the interactions between the parameters and the properties of the material. The results show that some parameters affect the properties to a greater degree than some other parameters. This information can be used to design the parts and the process so that the productivity and the material properties can be optimized for a given problem.

Composite Freeform Fabrication (FFF) Process

CFF is a novel material extrusion process that essentially comprises of 2 nozzles or print heads for extrusion:

1. Print Head 1 that extrudes the matrix material such as nylon
2. Print Head 2 that extrudes the reinforcement fiber such as fiberglass, kevlar or carbon fiber.

The part geometry in a 3D CAD format is first sliced into thin layers of cross sections. Each layer can then be assigned either the matrix material, the reinforcement or a combination of the two. The CFF process then lays down each layer by heating the print heads to the required temperatures and extruding the required material from either the print head 1 or 2. After the part is completed in this manner, it can be removed from the print bed and cleaned to remove any support structures. The machine and the nozzles are shown in Fig. 1.

Fig. 1: The CFF AM Equipment (MarkForged MarkOne) and the Print Head with 2 nozzles

There are various process parameters that can be varied to produce the part. They are as follows:

1. Fiber Volume Fraction: The fiber volume fraction can be varied by specifying the number of layers of fibers and nylon in the part. Apart from this, each layer of fiber can be adjusted to change the volume occupied by the fiber.
2. Fill Density: This is the density of the matrix material in the middle sections of the part. The part can be completely solid (fill density = 100%), or can be specified to be of any density ranging from 10 – 100%.
3. Fill Pattern: When the fill density of a part is less than 100%, it is possible to specify the kind of fill pattern in the part. The possible fill patterns include rectangular, triangular or hexagonal.
4. Fiber Fill Type: In the layers in which the reinforcement fiber is deposited, it is possible to change the fiber fill type to a concentric type (with varying number of concentric layers) or an isotropic type (with varying angle of deposition).

The different parameters are illustrated in Fig 2-4.

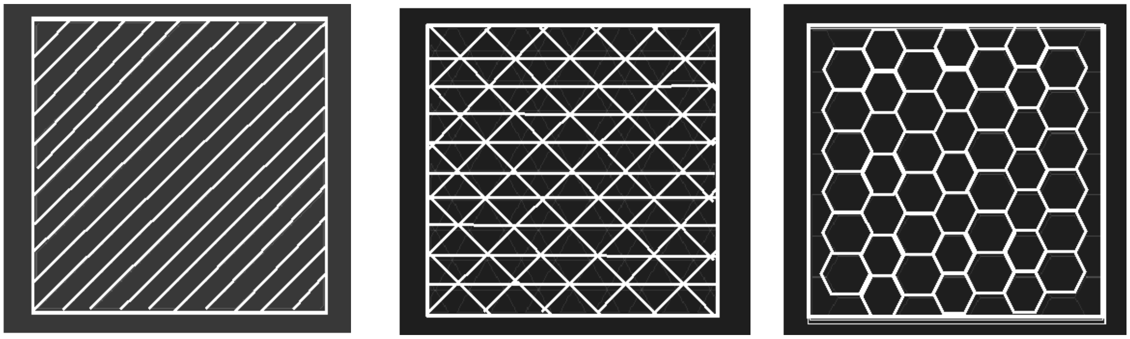

Fig. 2: Fill Pattern: (Left to Right) – Rectangular, Triangular and Hexagonal

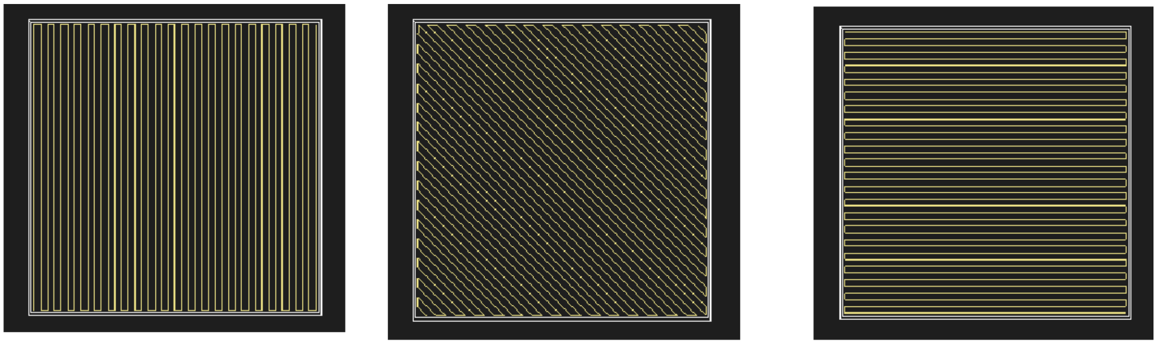

Fig. 3: Fiber Fill Type, Isotropic (Left to Right) – 90 degree, 45 degree and 0 degrees

Fig. 4: Fiber Fill Type, Concentric (Left) – 10 layers of fiber, (Right) – 18 layers of fiber

Experimental Plan: Design of Experiment

A design of experiment (DOE) is a method in which a series of input variables are changed and the output is observed in a manner that reduces the cost and time of doing the experiments (Montgomery, 2013). Statistical analysis is then performed on the output to determine the input variables that affect them and also the manner in which they affect them. Some of the most common kinds of DOE designs are factorial designs, response surface designs, mixture designs and Taguchi design (Antony, 2014).

A Taguchi design is based on a set of orthogonal arrays that utilizes minimum number of experiments to determine the affect on input variables on the output (Kuehl and Kuehl, 2000). Taguchi design can be employed on two-level, three-level or multi level variables. An example of a Taguchi design is shown in Table 1. This is called a L4 Design (4 runs of the experiment). There are 3 factors (or variables) – A, B, C. Each of these factors has 2 levels each (0 and 1). If a full factorial DOE were run on these factors, it would require 8 runs, but the Taguchi design can be run with only 4 experiments.

Experiment No.	Factor A	Factor B	Factor C
1	0	0	0
2	0	1	1
3	1	0	1
4	1	1	0

Table 1: A Taguchi L4 Design

Various different Designs of experiments in general and Taguchi design in particular have been used over the years for determining the properties of additive manufactured parts. Chen and Zhao, 2016, have written a concise review of use of Taguchi method in various AM processes. They also used the Taguchi method to determine the process parameters for improving the surface quality of binder jet additive manufacturing process. Barclift and Williams (2012) examined the variability of mechanical properties of parts manufactured by polyjet 3D printing using the DOE method. Krishnan el. Al. (2014) determined the most significant process parameter influencing macroscopic properties of AlSi10Mg parts manufactured by DMLS process using a full factorial DOE.

Materials and Specimen

To study the mechanical properties of the nylon-fiberglass composite, tensile testing of the material was done based on the Taguchi DOE. ASTM D3039 (ASTM D3039, 2014), standard for testing of composites was used for this study. The samples were prepared in 2 steps. The main section was manufactured first based on the DOE and the tabs were prepared separately. The main section was measured for dimensional details, weighed and glued to the tabs for the testing. Each experimental run had 3 samples manufactured at the same time. The specimen dimensions are shown in Fig. 5

Mechanical Testing

The samples were tested on a MTS 22 ton hydraulic machine with extensometer mounted on the samples in the gage area. The testing was done at a speed of 0.125 mm/min till the fracture was attained. The test set up is shown in Fig. 6.

To understand the interaction between the different process-parameters as outlined in Section 1.1 and the mechanical properties of Nylon-Fiberglass composite, Taguchi design of experimental plan was created in this research. The factors and the levels of each factor chosen on the study are shown in Table 2. The actual DOE matrix is shown in Table 3. As can be seen from the tables, a total of 36 experiment runs were conducted for the testing, 18 for each fill type. Three samples were tested for each experimental run.

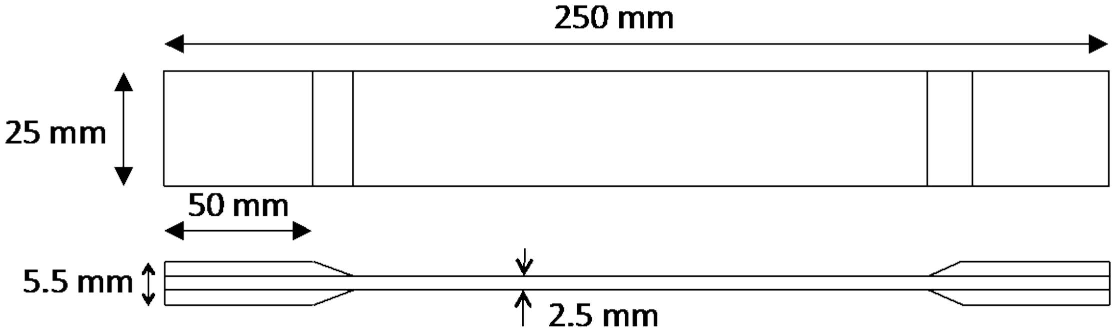

Fig. 5: Specimen dimensions for tensile testing

Fig. 6: (Left) – 3 Samples on the print bed (Right) – Sample being tested

Factors	**Levels**		
No. of layers of fiber	0	25%	50%
Fill Density	20%	60%	100%
Fill Pattern	Hexagonal	Triangular	Rectangular
Fill Type	Isotropic	Concentric	-
Fill Type Category (Isotropic)	0 degree	45 degree	90 degree
Fill Type Category (Concentric)	4 layers	8 layers	12 layers

Table 2: Factors and Levels for the Taguchi Design

Expt. No.	**Fill Type**	**Fill Density (%)**	**No of layers (%)**	**Fill Pattern**	**Fill Type Category**
1	Isotropic	20	0	Hex	1
2	Isotropic	60	25	Triag	2
3	Isotropic	100	50	Floor	3
4	Concentric	20	0	Hex	1
5	Concentric	60	25	Triag	2
6	Concentric	100	50	Floor	3
7	Concentric	20	0	Triag	3
8	Concentric	60	25	Floor	1
9	Concentric	100	50	Hex	2
10	Concentric	20	0	Floor	2
11	Concentric	60	25	Hex	3
12	Concentric	100	50	Triag	1
13	Isotropic	20	25	Floor	1

14	Isotropic	60	50	Hex	2
15	Isotropic	100	0	Triag	3
16	Isotropic	20	25	Floor	2
17	Isotropic	60	50	Hex	3
18	Isotropic	100	0	Triag	1
19	Isotropic	20	25	Hex	3
20	Isotropic	60	50	Triag	1
21	Isotropic	100	0	Floor	2
22	Concentric	20	25	Triag	3
23	Concentric	60	50	Floor	1
24	Concentric	100	0	Hex	2
25	Isotropic	20	50	Triag	1
26	Isotropic	60	0	Floor	2
27	Isotropic	100	25	Hex	3
28	Concentric	20	50	Triag	2
29	Concentric	60	0	Floor	3
30	Concentric	100	25	Hex	1
31	Concentric	20	50	Floor	3
32	Concentric	60	0	Hex	1
33	Concentric	100	25	Triag	2
34	Isotropic	20	50	Hex	2
35	Isotropic	60	0	Triag	3
36	Isotropic	100	25	Floor	1

Table 3: DOE based on Taguchi Design for the study

The fill type category column shows “1/2/3” in the Table 3. This corresponds to the factors in Table 2 based on the fill type. For example, if the fill type is Isotropic and the category is 2, it means orientation with 45-degree fiber layout. Similarly fill type of concentric with the category of 1 means concentric fiber layout with 4 layers.

Experimental Results

The mean UTS (Ultimate Strength), YS (0.2% offset yield strength), Elongation, Elastic Modulus, Weight and Fiber volumes for the different experiments are shown in Table 4. The build time has been standardized with the time for experiment 1 represented as 1.0.

Expt. No.	**Build Time (Std.)**	**Fiber Vol. %**	**Mean UTS (Mpa)**	**Mean Elon. (%)**	**Mean E (Gpa)**	**Mean Wt. (gms)**
1	1.00	15.28	76.96	1.14	6.81	10.83
2	1.48	22.93	24.84	4.42	1.40	17.07
3	2.15	44.05	26.62	1.15	2.08	18.32
4	1.13	5.83	22.22	0.80	2.05	9.62
5	1.02	18.96	94.69	1.06	8.61	14.07

6	2.55	41.32	233.44	2.76	8.45	19.49
7	1.44	14.15	275.98	2.77	9.96	10.66
8	1.84	6.64	49.71	3.08	1.61	14.98
9	2.19	29.68	189.15	3.12	6.05	17.08
10	1.30	10.75	49.02	2.72	1.80	12.68
11	1.72	28.86	143.58	2.82	5.09	13.59
12	1.80	16.05	105.97	3.14	3.34	16.45
13	1.45	22.58	139.33	2.56	5.45	18.23
14	1.99	45.22	29.46	10.85	0.44	19.53
15	1.13	15.57	11.55	32.85	1.46	13.52
16	1.27	22.90	27.93	2.82	1.22	17.22
17	1.17	41.77	19.75	19.15	0.35	19.70
18	1.33	12.00	79.69	3.02	2.64	16.51
19	1.20	23.49	18.49	31.90	0.26	17.88
20	1.77	41.69	278.27	2.81	9.91	11.44
21	1.69	9.46	26.72	2.85	1.32	11.50
22	1.22	22.11	143.85	2.99	4.82	17.18
23	1.12	16.47	96.35	3.03	3.18	16.72
24	1.27	9.45	49.10	3.14	1.57	13.89
25	1.42	43.68	93.10	2.10	4.43	19.24
26	1.06	15.14	16.64	38.05	0.19	13.98
27	1.64	26.64	16.37	19.27	0.29	16.17
28	1.27	30.70	161.20	2.56	6.12	17.57
29	0.95	13.77	70.95	3.05	2.33	13.94
30	1.25	9.15	44.76	3.00	1.49	14.58
31	1.49	41.98	151.50	1.79	8.49	19.49
32	0.94	5.33	18.84	2.12	0.89	12.42
33	1.02	18.02	79.01	2.53	3.13	14.79
34	1.91	45.22	35.08	10.08	0.91	19.73
35	1.16	14.70	13.69	31.75	0.20	14.11
36	1.33	20.04	154.80	3.06	5.07	18.41

Table 4: Results of the DOE

Exploratory Data Analysis

The first step after the experimentation was to divide the data into two distinct groups: one for isotropic fill type and one for concentric fill type. According to Gibson (Gibson, 2016), the mechanical properties of composites are dependent on the direction of fibers with respect to the loading direction. The box plots for the mean UTS for the different fill types, fill density and fill pattern are shown in Fig. 7 – 9 along with representative stress strain curves.

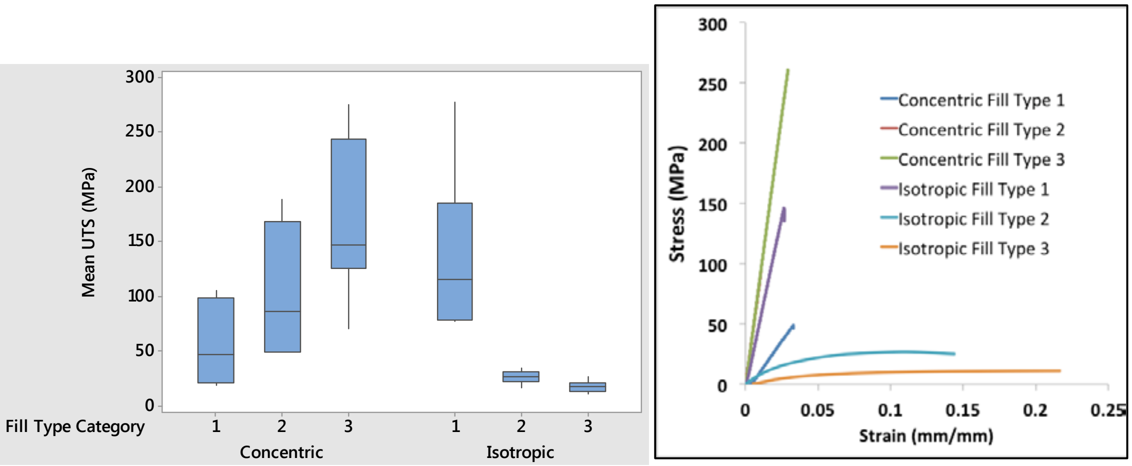

Fig. 7: Distribution of mean UTS with the fill type category and stress strain curves

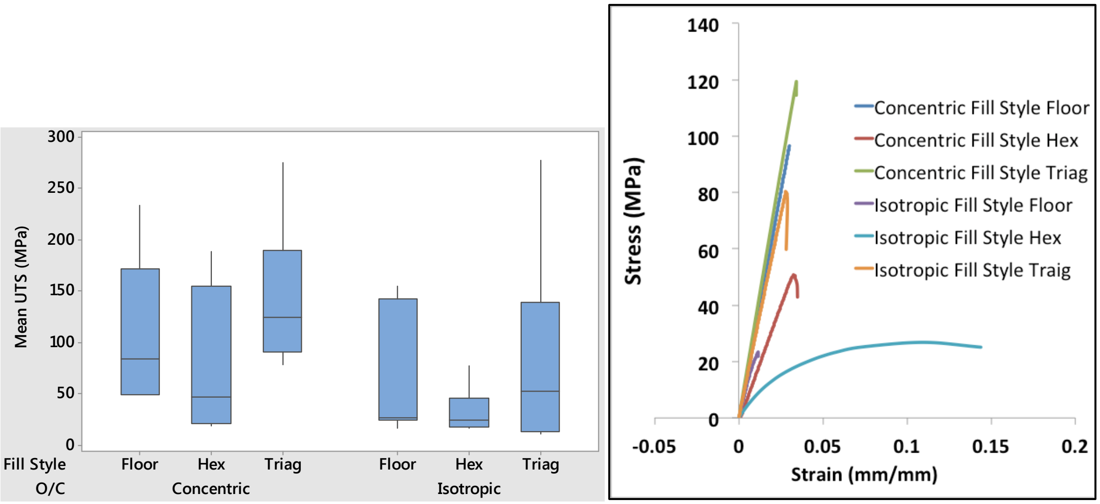

Fig. 8: Distribution of mean UTS with the fill style and stress strain curves

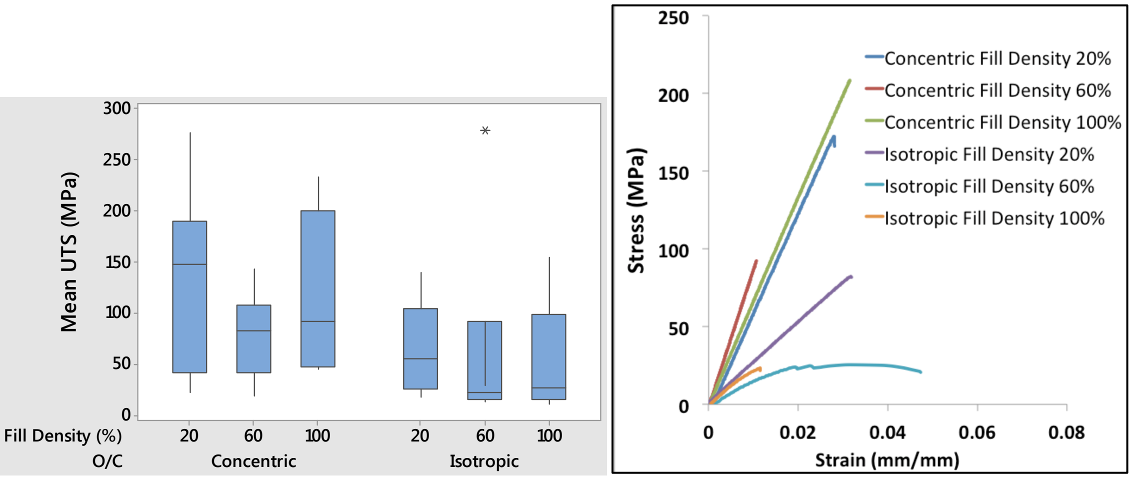

Fig. 9: Distribution of mean UTS with the fill density and stress strain curves

The box plots and the stress strain curves show the following:

a) The mean UTS increases for the different concentric fill types. It is highest for the 0 degree isotropic type and decreases for the 45 and 90 degree orientation
b) For both the concentric and isotropic fill types, the fill styles (floor, hexagonal and triangular) do not show any trends or differences in the mean UTS
c) The fill density also does not seem to affect the UTS in a considerable manner. The mean UTS are however higher for the Concentric fill type than the isotropic.
d) The stress strain curves show brittle failure for most of the cases except for isotropic fill types (across different fill styles and densities). The isotropic fill type with 45 degree and 90-degree orientation are more ductile with higher strain to failure.

Analysis of Variance (ANOVA)

The exploratory data analysis shows that there is lot of difference in the mechanical properties (UTS, Elastic Modulus, elongation etc.) based on the two fill type categories. Hence it is important to separate these two categories and then do the analysis for them.

Analysis of variance (ANOVA) is a statistical tool that helps in determining the significance of each factor on the response. ANOVA was conducted separately on the Isotropic and Concentric fill types to see the effect of different parameters on the Elastic Modulus and UTS. The results are shown in Table 5 and 6.

Control Factor	**Degree of Freedom (Df)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**F value**	**Probability (>F)**
Category	1	42683	42683	16.99	0.001 *
Density	1	958	958	0.38	0.548
Layers	1	6664	6664	2.65	0.129
Style	2	1909	955	0.38	0.691
Residuals	12	30137	2511		

Table 5: ANOVA table for isotropic fill type with UTS as response

Control Factor	**Degree of Freedom (Df)**	**Sum of Squares (SS)**	**Mean Square (MS)**	**F value**	**Probability (>F)**
Category	1	38698	38689	16.56	0.001 *
Density	1	411	411	0.17	0.682
Layers	1	18349	18349	7.85	0.015 *
Style	2	4427	2213	0.94	0.414
Residuals	12	28040	2337		

Table 6: ANOVA table for concentric fill type with UTS as response

Note: * denotes significance at 90% confidence level

The analysis is similar for both the UTS and the Elastic modulus. The ANOVA shows that the fill type category is the significant factors in both concentric and isotropic fill types. It also shows that the fill density and fill style do not play any significant role in determining the mechanical properties of the composite. The number of layers is significant at the 90% confidence level for the isotropic fill type while it is significant at the 85% confidence level for the isotropic fill type. Thus the only factors that affect the mechanical properties of CFF based fiberglass-nylon composites are the fill types and the number of layers of the fiber. The other design features such as the fill density and the fill style do not affect these properties in any significant manner.

Discussions

According to Gibson (2016), the mechanical properties of composites, if determined from micromechanical analysis, follow the rule of mixtures. The rule of mixtures for the elastic modulus can be generally written as:

$$E_c = \eta E_f V_f + E_m V_m \qquad (1)$$

Where,

E_c = Elastic modulus of the composite
E_f = Elastic modulus of the fiber
E_m = Elastic modulus of the matrix
V_f = Volume fraction of the fiber
V_m = Volume fraction of the matrix
η = composite efficiency factor (based on the angle of fiber). It is equal to 1 for 0 degree, 0.25 for 45 degree and 0 for 90 degree orientation

Similarly, the tensile strength of a composite is defined as:

$$\sigma_c = \sigma_f V_f + \sigma_m' V_m \qquad (2)$$

where,

σ_c = Tensile strength of the composite
σ_f = Tensile strength of the fiber
σ_m' = Tensile strength of the matrix at the strain at which the fiber fails

Both these equations show that the elastic modulus and the tensile strength depend on the volume % of fiber in the composite. To understand this, the mean UTS and elastic modulus of the fiberglass-nylon composite were plotted against the fiber volume % (Figure 10). The graphs show that the UTS and E for the concentric fill type follow a linear trend with the fiber vol. % and are scattered for the isotropic fill type. A closer look reveals that the points that do not follow a linear trend in the isotropic fill type are for orientations of 45 degrees and 90 degrees.

Using the equations 1 and 2 and fitting the values as depicted in figure 10 reveals the following two equations for the values of E_c and σ_c:

$$\sigma_c = 499.6\, V_f + 4.01\, V_m \qquad (3)$$

with a R^2 value of 0.89 and

$$E_c = \eta * 19.83V_f + 0.04V_m \quad (4)$$

with a R^2 value of 0.96

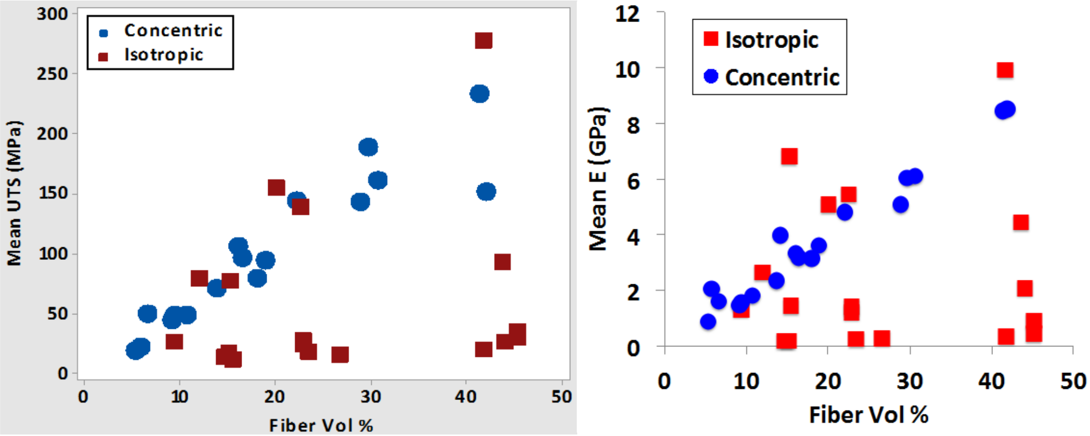

Fig. 10: Variation of mean UTS and mean Elastic Modulus with the fiber volume %

Conclusions

CFF (Composite filament fabrication) process is a novel additive manufacturing process similar to the typical material extrusion process such as FFF (Fused Filament Fabrication) and FDM (Fused Deposition modeling), except for the fact that it can extrude a fiber along with a plastic. The process has two nozzles, one that can extrude Nylon and another that can extrude a fiber such as Fiberglass, Kevlar or Carbon Fiber. The resulting part can have multiple layers of the Nylon along with layers of the fiber embedded between them.

Before the CFF process can be used to create parts that can be used in prototyping and production stages, it is important to understand the interactions between the process parameters and the resulting mechanical properties of the parts. The aim of this research was to create a process – property interaction matrix that can help the designers and production engineers in selecting the optimum parameters for their requirements.

In this work tensile testing of nylon-fiberglass composites is conducted on a design of experiments matrix based on Taguchi Design. Process parameters such as fill density, fiber volume fraction, fill pattern and fiber-layering technique are varied and the samples are tested for their dimensions, productivity, yield strength, elastic modulus, ultimate tensile strength and elongation. The results are then used to determine the interactions between the parameters and the properties of the material. The results show that the fill type and fiber orientation play the most important role in the mechanical properties. Traditional composites micromechanical theory of rule of mixtures can be used to predict the properties of composite using the fiber volume % in the mixture. The empirical relationship between the tensile strength of composite and its individual components is derived and presented in this work.

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