Development of in-house composite wire based feed stock filaments of fused deposition modelling for wear-resistant materials and structures

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ABSTRACT

In the recent past many researchers have highlighted the development of in-house filament for fused deposition modelling (FDM) in order to enhance the application domain of the process. But hither to no work has been reported on the manufacturing of wear-resistant materials and structures with hybrid feed stock filament (comprising of Nylon matrix and Al_2O_3 powder) from process capability point of view. The Al_2O_3 powder as reinforcement in Nylon-6 matrix has been, e.g., used as rapid tooling in grinding applications for dentistry. The study started with establishing a melt flow index (MFI) of the polymer matrix composite (PMC) to be used as FDM filament. Further an effort has been made to establish the selected proportions of filler, matrix (Nylon-6) and extrusion load on the MFI of reinforced FDM filament as per ASTM-D1238-95 standard. The results of study suggest that FDM process with hybrid filament lies in ±4.5sigma (σ) limit as regard to wear of rapid tooling (grinding wheel selected as a case study) is concerned. This process ensures rapid production of pre-series technological prototypes and proof of concept at less production cost and time.

1. Introduction

Rapid prototyping techniques (RPTs), also known as additive manufacturing (AM) techniques, are one of the most suitable aids for tackling various problems relevant to biomanufacturing sectors. Numerous RPTs found their applications for fabricating patient specific bio-constructions [1]. The rapid manufacturing abilities of these techniques help the medical practitioners/doctors/students for their strategic planning on a prototype. According to Wohlers report of 2010, RPTs emphasised with robotic dispensing technique have been particularly designed for medicine and tissue engineering [2]. The layer-by-layer manufacturing of these techniques produced the best fit parts with negligible material wastage when compared to conventional manufacturing class. In recent past, RPTs techniques have been employed in the field of dentistry for the development of crowns, dentures and bridges [3-5]. The fabrication of the medical implants start with MRI or CT scan data (patient specific) and its conversion into a 3D geometry through some commercially available software such as: MIMICS, 3D-Doctor, etc. The output available in .STL (standard triangulation language or stereo-lithography format) is best suitable for any type of rapid prototyping (RP) system. The use of AM technologies can be helpful in producing sacrificial patterns for conventional casting of best fitted removable partial dentures (RPDs) [6].

Most of the RPTs require the use of organic solvents as binder for the powder-based aliphatic polyesters [7]. Some of the research groups have used fused deposition modelling (FDM) for designing bioresorbable scaffolds, highlighted that this technique does not require any solvent [8]. The FDM method is one of the most widely used, after stereo-lithography, and economical class of RPTs which is used for the fabrication of real 3D models through computer interface. The input to interface could be any of the MRI, CT, CAD, jpg, etc. created through a sophisticated 3D image digitizing system. Most of the commercial FDM system comes with a temperature controlled chamber (closed chamber) wherein the whole fabrication takes place. For prototyping, a thin filament wire is forced to pass through a heated zone i.e. head of the system. The temperature of the head is material dependent and is selected in a way that the filament after passing through it turns to semi-molten stage. This molten filament material

found its escape through the nozzle tip and finally layered down on a flat platform one after one. The FDM head moves in x and y direction whereas the platform moves in z direction. Pictorial view for different parts of FDM system is shown in Fig. 1. The head-platform path generated by system friendly software is temporarily stored by the system.



Note: (a) Controlling display; (b) Model material rack; (c) Support material rack; (d) Base platform; (e) Roller screw; (f) Mechanical drives; (g) Building prototypes

Fig. 1.Pictorial view of FDM system [9].

Till date, various researchers have developed different categories of alternative FDM filament materials in order to increase the application domain of this technology by reducing its running cost. Development of new FDM materials based on metals or ceramics is a tedious task as the filament required for FDM system is very specific in-terms of its size, mechanical properties and rheological properties [10, 11]. It has been found that the thermal and mechanical properties of iron and copper reinforced ABS polymer matrix were better as compared to pure ABS polymer [12]. It has been found the addition of 10% of iron powder reinforcement in FDM graded ABS material enhanced the thermal properties and storage modulus of the resulting filament [13]. Tribological properties compared by a research group highlighted that Al₂O₃ reinforcement in Nylon-6 has improved the wear resistant properties of rapid tooling prepared with FDM system [14]. Addition of carbon fibres in pure plastic specimen increased tensile strength and Young's modulus whereas, other properties such as: toughness, ductility, and yield strength were decreased [15, 16]. Recently, a research group of

Taiwan has developed an eco-friendly filament material consisted of starch based biomass polymer for FDM [17].

An artificial dentures/tooth frequently needs micro-machining operation prior to final fitting and the tool for the same can be manufactured in a customized manner with FDM system by using reinforced filament. In the present research work, an effort has been made for the development of new filament material for rapid tooling application in dentistry. Proportion of alternative FDM filament (made of Nylon-6/Al₂O₃) has been established by maintaining its MFI suitable to available FDM system. Test specimens fabricated with FDM system were tested for their wear resistance properties as per ASTM standard. Statistical analysis carried out on wear results highlighted that the process capability indices ($C_P \& C_{PK}$) are greater than 1 hence the process is suitable for producing such tools.

2. Experimentation

Table 1

The present work is focused on enhancement of application area of FDM machine by developing an alternative FDM filament material for wear resistant materials and structures, such as, e.g., grinding applications in dentistry; the reinforcement of cementitious materials in civil engineering; and the 3D printing of sport shoes. Nylon-6 granules (E-35grade; specific density- $1.2g/cm^3$) and Al₂O₃ powder (particle size: $125-149\mu$ m) were selected as filament ingredients. A set of pilot experiments were performed on melt flow indexer (MFIer) as per ASTM-D-1238 standard in order to achieved the desired rheological properties of alternative material proportion the various proportions of these two filament ingredients. It has been found that the MFI of OEM-ABS-P430 filament of uPrint SE-FDM system (make: Stratasys Inc.) is 2.41gm/10min. So, it was necessary to develop alternative filament material with similar rheological characteristics as any change in the FDM system was prohibited. Experiments were performed as per Table 1 and it has been found that proportion with 50% (wt.) of Nylon-6 and 50% (wt.) of Al₂O₃ powder has similar MFI value.

Proportion (Nylon-6/Al ₂ O ₃ powder)	MFI (gm/10min)										
in gm/20gm	#1	#2	#3	#4	#5	#6	#7	Mean			
16/4	7.405	7.48	7.68	6.40	6.945	6.795	6.255	7			
14/6	4.725	5.76	6.785	6.965	5.19	6.995	7.215	6.234			
13/7	3.56	4.225	5.455	2.655	4.48	5.68	3.45	4.215			
12/8	1.925	1.64	1.965	2.11	3.54	4.315	4.21	2.841			
10/10	2.435	2.83	2.325	2.325	1.49	1.49	1.55	2.363			

MFI results for different proportions of Nylon-6/Al₂O₃ powder.

This proportion has been selected and used for the development of alternative FDM filament wire on single screw extruder as shown in Fig. 2(a). Fig. 2(b) shows the finally developed FDM filament wire. During extrusion of filament wire all process parameters of single screw extruder such as: die temperature, barrel temperature and screw speed were kept constant to 180°C, 155°C and 40rpmrespectively. However, take-up unit speed was adjusted in order to maintain filament wire diameter to 1.78mm. The filament fabricated was used to prepare test specimens of 10mm diameter and 30mm length at solid density option of uPrintSE-FDM system (see Fig. 3).



Fig. 2. Development of alternative FDM filament (a) and alternative filament wire (b).

At room temperature, the wear tests were performed on pin-on-disc type tribo-meter (DUCOM-TL-20) according to ASTM-G-99 standard under dry sliding condition [14].



Fig. 3. Preparation of specimen for wear test.

As polymers and their composites results into less wear when rubbed against a steel disc due to formation of thin protective film [18]. So, in the present case wear of the test specimens was calculated by making some additional modifications in the existing tribo-meter. For this, SiC emery paper (#600) was pasted on steel disc and test specimens were rubbed onto it. Normal load, track diameter and speed of rotation were maintained at 5N, 80mm and 200rpm respectively and test was performed for 10min. Table 2 shows the results of wear test.

Table 2

Results of wear.

	Wear (µm)															
#1	#2	#3	#4	#5	#6	#7	#8	#9	#10	#11	#12	#13	#14	#15	#16	Mean
161	158	161	163	162	159	160	160	163	160	162	158	161	158	162	162	160.625

Wear of test specimen prepared with OEM-P430 (made with FDM system) was also calculated and it was found that wear resistance of composite specimens was far better. Fig.4 shows the wear tracks of Nylon- $6/Al_2O_3$ composite and OEM-P430 specimen. From Fig. 4, it has been observed that the developed material is highly resistive against wear and could be used for the micro-machining of acrylic based dentures, or different wear-resistant materials and structures.

Further, scanning electron microscopic (SEM) analysis of test specimen (refer Fig. 5) highlighted the even distribution of Al₂O₃ particles in Nylon-6 matrix.



Fig.4. Wear tracks of Nylon-6/Al₂O₃ composite (a) and OEM-P430 specimen (b).



Fig. 5. SEM micrograph of test specimen.

3. Statistical Analysis

In order to establish the process as industrial standard, statistical analysis has been carried outby using the wear results. Presently, statistical process control (SPC) and quality improvement (QI) methods were which are used for measuring uniformity and for monitoring relevant process characteristics such as process capability indices (PCIs).QI Macros (2015) statistical software package was used for calculating PCIs of the collected data and for plotting control charts through the adoption of specified procedure [19]. Fig.6 shows the process capability histogram for obtained wear results.



Fig.6. Process capability histogram.

It has been observed that the obtained values of process capability indices i.e. Cp and Cpk are 1.37 and 1.23 respectively. Generally, a process with Cp value greater than 1 is assumed to be capable for produced desirable process characteristics [20]. Similarly, a process can be treated as an industrial benchmark if its Cpk value is greater than 1 [21]. In the present case both Cp and Cpk are greater than unity hence there are strong possibilities that the process is statistically controlled for production applications which mean, e.g., that machining of artificial polymeric dentures will not affect by changing the grinding tool. Fig.7 and Fig. 8 shows R-chart and X-chart respectively for the obtained wear values.



Fig.7.R-chart for obtained wear values.



Fig.8. X-chart for obtained wear values.

Fig.7 is not showing the presence of any special cause/effect on the collected wear results. This may be due to the adoption of the precision while performing wear test. However, due to the existence of only one sub group, it can't be ensured that the variation within the subgroup is due to any common effect as it may be due to the non-uniform mixing of the Nylon-6 and Al₂O₃ powder while filament preparation. Further in Fig.8, the shifts of X-values about the mean are dispersed evenly around both sides (\pm). For *Cpk* value of *1.23*, the area under normal curve is *0.983198* and non-conforming parts/million (ppm) are6.8. The results of study suggested that wear of the grinding tools lies in \pm 4.5 sigma (σ) limit.

4. Conclusions

On the basis of experimental results and statistical observations, following conclusions may be drawn:

In the present research work, an alternatively fabricated reinforced FDM filament has been successfully fabricated for the manufacturing of wear-resistant materials and structures, with special focus on grinding applications of artificial polymeric dentures. Test results highlighted that due to the reinforcement Al₂O₃ particles in Nylon-6 matrix the resistance of composite specimens against wear has been improved significantly. Further from SEM analysis, it has been found that the distribution of Al₂O₃ particles in Nylon-6 matrix is quiet uniform. Statistical analysis performed on obtained wear results highlighted that the process is having high capability of producing desirable wear results highlighted that the process is having high considered as industrial benchmarks (Cpk>1). Further, the results of study suggested that wear of the grinding tools lies in ±4.5 σ limit as non-conforming ppm was 6.8. Future research work may focus on different types of reinforcement material in order to compare their suitability for actual clinical applications. Additional future extensions of the present study will focus on the additive manufacturing via FDM of wear-resistant fibers and bars for the reinforcement of cementitious materials [22-25], and impact-protection metamaterials [26-27]-

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