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Solid Additives and their Lubrication Effects on Polyetheretherketone Polymers – A Review

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Abstract

This review investigates and analyses the effects and usage of solid lubricants on high melting polymer printed via fused filament fabrication (FFF). The analysis indicated that most FFF printed parts suffer non-uniformity in mechanical properties and such printed pieces are generally weaker than conventionally produced counterparts. About 95% of researches on FFF were about solving the weakness and non-uniformity of mechanical properties, with 98% ignoring the tribological effects. Addition of laminar solids to polymer not only improves the tribological properties but also some mechanical properties. Nevertheless, the effects of laminar solid results on polymer might not be predictable as the outcome depends on relative properties of such polymer, solids and FFF processing parameter. It is suggested that further research should be carried out on the effects of laminar solids on FFF processed polymer.

Keywords: Additive Manufacturing (AM), 3D printing, Material Extrusion (ME), Fused Filament Fabrication (FFF), Fused deposition Modelling (FDM), Polyetheretherketone (PEEK), solid lubricants, laminar solids

1 INTRODUCTION

Additive Manufacturing (AM) was defined by ASTM F 2792-12a (2013) as “a process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies”. It also categorises the AM technology into seven classes based on the production of layer mechanism, namely “Material Extrusion (ME), Vat Photopolymerization (VAT), Material Jetting and Binder Jetting. Other classes were Powder Bed Fusion (PBF), Direct Energy Deposition (DED) and Sheet Lamination” (ASTM International, 2013).

AM can also be grouped according to input material form, namely: wire-based form or filament as seen in ME process, liquid form as seen in VAT method and powder form as in PBF method (Gibson *et al.*, 2015).

Furthermore, AM was further categorised into two groups, namely: Direct Printing techniques (DIP) and Indirect Printing Techniques (IPT) by Travitzky et al., (2014) and Zocca et al., (2015). Direct Printing methods generally use direct extrusion through nozzles, and sometimes direct hardening of polymers or drop on demand technique. In contrast, Indirect printing technique is majorly a powder-based AM technology, in which the powder is usually laid first and fused thermally or with adhesive before a new layer of powder is spread over the existing layer.

AM has a significant advantage over the traditional way of manufacturing with a high degree of freedom during production as it is a layer by the layer manufacturing process. The high degree of freedom makes it easy to be used to fabricate objects with complex geometries, relatively low error, lower production cost, and minimal material wastage (Gibson *et al.*, 2015; Thompson *et al.*, 2016). These advantages make AM an optimum technique for adoption in manufacturing technology. The degree of freeform made it easily applicable in the manufacturing of complex engineering components for biomedical, defence, and other specialized areas (Adikari Appuhamillage, 2018).

Fused Filament Fabrication (FFF), also known as Material extrusion (ME) or Fused Filament Modelling (FDM) process is the most popular and most used in AM technology due to its cheapness and easy setup design for the printer (Adikari Appuhamillage, 2018). The production of AM printed parts generally was projected in 2016 to have about 4900% monetary increase by the year 2030 (Vitale and Cabral, 2016; Adikari Appuhamillage, 2018).

2. FFF AND PROCESSING PARAMETERS

FFF is a 3D fabrication process whereby material in wire form is heated to a semi-solid state and then forced out through an orifice called extrusion nozzle. The movement of the extrusion nozzle depends on the input parameters of the 3D stereolithographic computer file (STL file) to produce a 3D model (Wong and Hernandez, 2012; Ngo *et al.*, 2018). Also, FFF printing technique can be further explained as the process of manufacturing where thin wire plastics are fed into a machine. The machine heats the melt and ejects the material as a semi-plastic form; this is then laid layer by layer at a typical thickness of 0.25 mm (Wong and Hernandez, 2012). The nozzle is usually in the extrusion head which alters the temperature, pressure and feed rate as required by the computer input.

FFF method is attracting a lot of attention due to its simplicity, cheapness, easy setup and wide range of material usage. However, FFF printed parts are generally plagued by anisotropicity in its properties that originated from the layerwise structure (Xia *et al.*, 2020). Most of FFF produced parts only features 60 % to 8 0 % mechanical strength of traditionally produced counterparts (Ngo *et al.*, 2018; Lin *et al.*, 2019; Liu *et al.*, 2019; Wang *et al.*, 2019; Golbang *et al.*, 2020; Park *et al.*, 2020; Ramesh and Panneerselvam, 2020; Yadav *et al.*, 2020).

The primary materials used in AM industries are polymers, metals and ceramics. Polymers and its composites account for about 81 % of usage in AM industries (Dizon *et al.*, 2018). Also, compositing of polymers with other materials is done mostly to alleviate some mechanical deficiencies of pure polymers and increases its industrial application (Kuo et al. 2005; Lai et al., 2007; Chen et al. 2012; Zalaznik et al., 2 016; Garcia et al., 2019; Arif et al., 2020). Research studies on materials used in the FFF techniques indicated that polymer is the primary material for the FFF process (Gibson *et al.,* 2015; Dizon *et al.*, 2018; Popescu *et al.*, 2018; Arif *et al.*, 2020; Wu *et al.*, 2020). The widely used polymer in FFF manufacturing industries is a variety of Acrylonitrile Butadiene Styrene (ABS). Some other polymers like polycarbonate (PC), polylactic acid (PLA) and Polyetheretherketone (PEEK) are being experimented on and used as the base material in FFF for printing. The search for excellent mechanical properties, high thermal resistance and biocompatibility from these polymers are needed for broader applications in aerospace, medical and military. Also, the polymer used in FFF processes must generally be able to undergo phase change easily at extrusion point either chemically or thermally (Gibson *et al.*, 2015). These polymeric materials are those that can quickly solidify or turn into semi-solids at a specific temperature or chemical change. Most materials used for FFF printing must possess this particular feature.

Major processing parameters that are usually considered in any FFF manufacturing process are; temperature, building orientation and slicing information (Ngo *et al.*, 2018; Popescu *et al.*, 2018). However, other processing parameters are printing angle, post-printing treatment, printing speed, in-fill pattern and in-fill ratio.

1) Temperature: this affects the mechanical and morphological properties of the final fabricated parts. High-temperature settings lead to thermal degradation of the printed piece. In comparison, low-temperature environments result in weaker interlayer bonds which might result in easy delamination of the printed piece (Gibson *et al.*, 2015; Popescu *et al.*, 2018). Vaezi and Yang (2015) classified thermal management of the FFF process into three significant parts viz:

a) Bed temperature: this is the platform where the printing is layered on and is usually preheated to improve interlayer adhesion. The preheating ranges a few degrees of about 10 ºC to 30 ºC below the transition temperature of the material or polymer intended to be printed (Popescu *et al.*, 2018)

b) Extrusion temperature: the temperature at the nozzle, is close to the melting temperature of feed material to maintain it in semi-solid form and to avoid thermal degradation. Thermal degradation also varies per material and the density of the material, which is usually above the melting point (Yang *et al.*, 2017; Popescu *et al.*, 2018).

c) Environmental temperature: It is the atmospheric temperature of the enclosed space where the parts are printed on the building bed. The enclosed area is often preheated to a close glass temperature of about 5% lesser to input material melting temperature and regulated to achieve best interlayer layer adhesion. This environmental temperature is mostly used for a polymer with a high melting temperature (Vaezi and Yang, 2015; Yang *et al.*, 2017; Popescu *et al.*, 2018).

2) Building orientation: these are the directions of builds with coordinate axes. The test specimen is usually vertical, horizontal or laterally oriented, but other build orientations might be used with the need of supporting materials. Basic build orientations and print angles that are being used in FFF method were illustrated in Fig. 2.1 (Domingo-Espin *et al.*, 2015; Zaldivar *et al.*, 2017; Popescu *et aol.*, 2018).

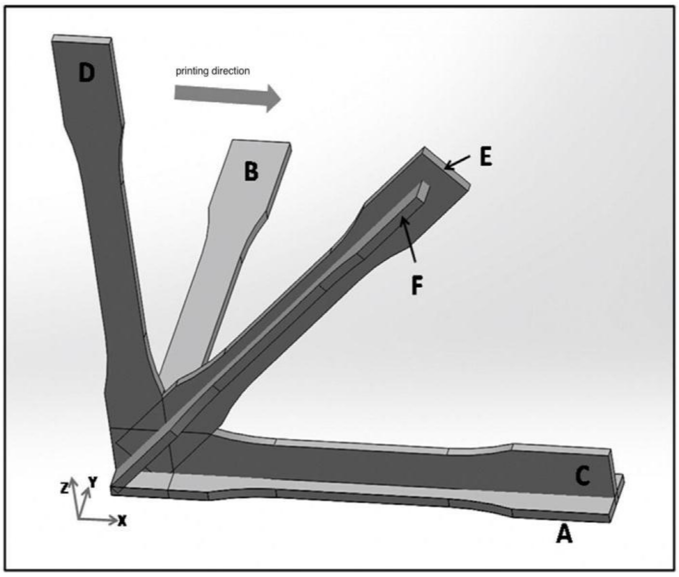

Fig. 2.1 The dog-bone specimen, illustrating several building orientations (Zaldivar *et al.*, 2017).

3) Slicing parameter: these are sets of computer instruction about specific parameters, such as layer thickness or diameter, raster, and nozzle diameter about the 3D STL model and file.

a) Layer thickness or diameter: this refers to the movement of the nozzle in an upward direction, i.e. Z-direction during fabrication (Wu *et al.*, 2017; Salazar-Martín *et al.*, 2018).

b) Raster angle: this is the angle between the nozzle path and the X-axis of the build platform (Wu *et al.*, 2017). The raster angle is represented as θ within Fig 2.2a and graphically annotated on dog bog specimen in Fig 2.2b

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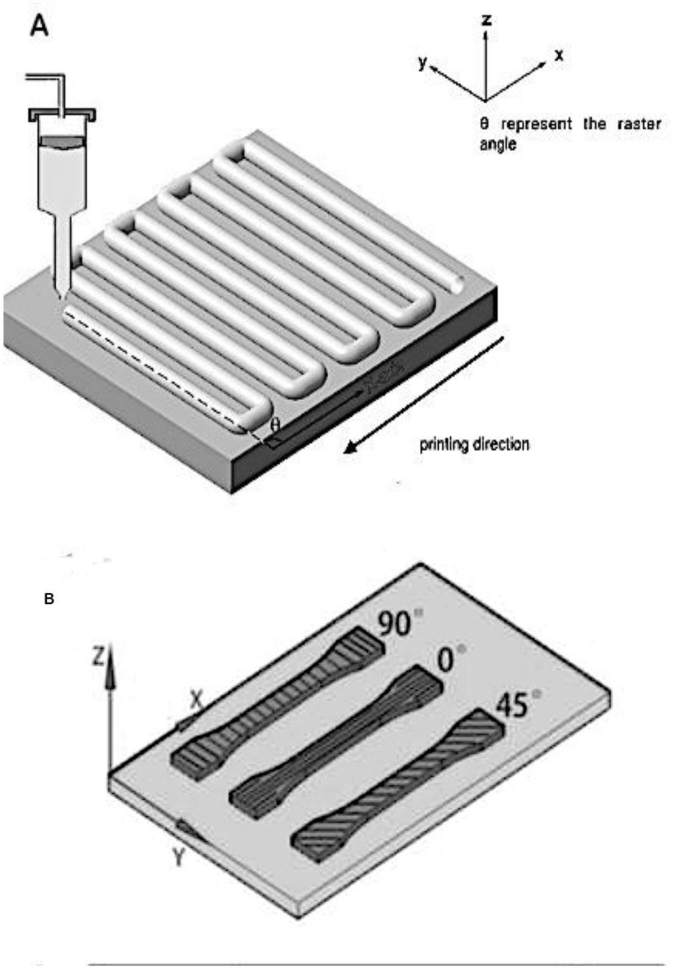

Fig 2.2 (a) The raster angle and print direction of the FFF process (Wu *et al.*, 2015) (b) dog bone specimen indicating various raster angles (Xia *et al.*, 2020).

c) Extrusion diameter: this determines the width of the road or raster width; it is the diameter of the nozzle or orifice where the feed material is forced out. The diameter must always be smaller than the layer thickness to achieve the best resolution during fabrication. The smaller the diameter, the more detailed the resolution and the more time-consuming (Gibson *et al.*, 2015; Salazar-Martín *et al.*, 2018).

Review of several studies indicated that FFF printed objects have anisotropic mechanical properties which are also the factor of processing parameters and input material characteristics (Ziemian *et al.*, 2012; Torrado and Roberson, 2016; Chen *et al.*, 2017; Meng *et al.*, 2017; Ngo *et al.*, 2018; Popescu *et al.*, 2018; Somireddy *et al.*, 2019). This property is prompting many FFF researchers to research into the improvement of tensile properties and the reduction in anisotropic properties across the printed parts or specimens.

Given the non-uniformity of properties of the FFF printed parts, the addition of other polymer materials, chemicals and tubes as fillers to mitigate or alleviate the effects of such anisotropicity of such fabricated parts are necessary (Christ *et al.*, 2017; Rajpurohit and Dave, 2018; Tambrallimath *et al.*, 2019; Kumar and Senthil, 2020; Wu *et al.*, 2020). These additives often referred to as nanofillers, are used to improve some specifically targeted properties like tensile strength and Young modulus of elasticity (Dorigato *et al.*, 2017; Wu *et al.*, 2020). Other properties are tribological properties, crystallisation, hardness, electrical properties, flame retardancy and sometimes colouration (Shofner *et al.*, 2003; Dorigato *et al.*, 2017; Golbang *et al.*, 2020; Wu *et al.*, 2020). For instance, Tambrallimath *et al.*, (2019) used 0.8 wt% graphene as nano-filler to increase the Young modulus of ABS-PC composite to about 60 % to pure ABS-PC composite's value. Similarly, Dorigato *et al.* (2017) melted multiwalled carbon-nanotubes with ABS using a twin-screw extruder of 16mm screw diameter. The twin extruder was configured to length-diameter (L/D) ratio of 25, with temperature ranging from 180 ^{o}C to 240 ^{o}C at 5 revolutions per minutes. This addition process altered the electrical conductivity and elastic modulus of FFF produced ABS by 70 % and 40% increase respectively. However, it is strongly building orientation dependent.

In another experiment by Olesik *et al.* (2019), the effects of glass filler particle size on Low-density polyethene (LDPE) were characterised by Young's modulus of elasticity, morphology and friction properties. LDPE was mechanically mixed with glass particles of an average diameter of 0.5 mm and dried at 70 ^{o}C. Young's modulus of elasticity increased by 13.5 %. Nevertheless, the friction coefficient reduced drastically, with 15% of glass-filled LDPE when compared to 30 % glass filled LDPE. However, the friction properties depend on the relative friction direction and print direction. Also, LDPE filled with 30 % glass had a higher wear rate than the 15 % glass-filled LDPE irrespective of the frictional direction due to polymer matrix and filler concentration. Nevertheless, the addition of glass particles reduced the wear rate and increased the Young modulus when compared with pure LDPE.

The addition of fillers to polymers might improve some properties and cause degradation in some other properties; this prompts the need to fully understand the effect of such addition to the polymer used in the FFF method (Rajpurohit and Dave, 2018; Popescu *et al.*, 2018; Ngo *et al.*, 2018). Additionally, the reaction or output properties of traditionally fabricated parts are quite different from the FFF printed parts. Mechanical strength of FFF parts only accounts for about 60-80% of traditionally manufactured parts, irrespective of the polymer used in printing or fabrication (Hossain *et al.*, 2013; Ngo *et al.*, 2018; Popescu *et al.*, 2018; Rajpurohit and Dave, 2018).

Furthermore, Aumnate *et al.* (2019) showed that fillers and polymer matrix are factors of dispersion; Oversized particle-sized organoclay filler dispersed poorly in the Polypropylene matrix, tends to aggregate and might cause buckling at extrusion nozzle. Similarly, Sanes *et al.*, (2020), confirmed the effect of particle size and concentration of graphene on the polymer's mechanical properties, which was similar to the findings of Aumnate *et al.* (2019) on the varied particle size of organoclay fillers.

The works of Olesik *et al.* (2019), Aumnate *et al.* (2019) and Sanes *et al.* (2020) on filled polymer usage in FFF method confirmed the need for effective dispersion method for fillers in the polymer matrix, which is vital to the homogeneity of mechanical properties.

Some polymers with high melting points, e.g. PEEK, tend to be viscous due to early crystallization at high temperature (Zalaznik, 2016; Golbang *et al.*, 2020). The viscosity might cause warpage and dimensional inaccuracy. Besides, an increase in temperature might also result in thermal degradation (Vaezi and Yang, 2015).

3. POLYETHERETHERKETONE

Polyetheretherketone (PEEK) is a material that possesses excellent mechanical strength, flame retardant, chemical inertness, high-temperature resistance and low weight to strength ratio. It changes from liquid to gel-like form quickly, and it has excellent biocompatibility with good mechanical properties and high-temperature resistance. The melting point is about 343ºC and thermally degrades at a temperature above 575 ºC (Patel *et al.*, 2010). Due to its mechanical and thermal properties, it satisfies the necessary use conditions in aerospace, construction, automobile and medical industries. Additionally, PEEK was considered to be very close to human bone in both mechanical and physical properties, and this makes it widely acceptable in dentistry and prosthesis application (Gibson *et al.*, 2015; Najeeb *et al.*, 2016; Geng *et al.*, 2019). Additionally, PEEK might be considered as a replacement for metal, due to its semi crystalline-nature, high thermal resistance, chemical inertness, flame retardancy and good mechanical properties which make it suitable for aerospace industries.

A considerable amount of research has been done on material selection, and mechanical properties of FFF processed parts and processes. Nevertheless, the focus on PEEK has been minimal due to its high melting temperature and high viscosity during extrusion, which affect the flowability during extrusion and print quality after printing. However, the need to solve the high viscosity problems of PEEK is critical, to make it printable, useful and in creating more functional parts via FFF technique. Furthermore, little has been done on the effects of solid lubricants on FFF processed polymers including PEEK (Wong and Hernandez, 2012; Mani *et al.*, 2014; Gibson *et al.*, 2015; Najeeb *et al.*, 2016; Arif *et al.*, 2018; Wu *et al.*, 2020).

Zalaznik *et al.* (2016) and Golbang *et al.* (2020) attempted to solve the high viscosity problem and improve the wear properties of PEEK by addition of various solid lubricants in varied weight ratio. They found out that the addition of such lubricants didn't only improve the rheology and flow rate but also increased some other mechanical properties of the composited PEEK. However, Zalaznik *et al.* (2016) research work used compression moulding technique which made the experiment not comparable with FFF technique. Nevertheless, the mixing method used showed higher dispersion and improved properties. (Golbang *et al.*, 2020).

4. SOLID LUBRICANTS

Solid lubricants are fillers added to the material to alter the tribological properties of the material, usually to reduce friction ($\mu < 0.2$) and wear rate ($k < 10^{-6} mm^3/Nm$) (Lancaster, 1972; Allam, 1991; Omrani *et al.*, 2017). Table 3.1 summaries the application of solid lubricants and their types according to the review on Solid lubricants for applications at elevated temperatures by Allam,(1991). Allam's (1991) study confirmed the reason why laminar solids are more applicable to FFF method, and specifically PEEK FFF fabrication.

Table 3.1 Summary of Solid Lubricants and Application. Summarised from Allam, (1991).

S/N	Types	Operating temperature range	Common examples	Environment where applicable
1	Polymers	27 ºC to 300 ºC	PTFE, Polyamides	Vacuum and cryogenic temperature
2	Laminar Solids	50 ºC to 450 ºC	MoS_2, WS_2, graphite	Vacuum and non-adsorption conditions
3	Metal Fluorides	500 ºC to 1000 ºC	Chemically stable oxides and chemically stable fluoride group I & II, e.g. LiF, CaF2, BaF2, CuO, V_2O_5, PbO, Bi_2O_3	Fused coating and composting above 500 ºC. as they only lubricant above 500 ºC

Most solid lubricants don't have a standard way of application or usage; it requires a significant volume of trial and error methods to understand better, the effects of solid lubricants on a specific material or environment (McCook *et al.*, 2006; Omrani *et al.*, 2017). Depending on the relative properties of both polymer matrix and solid lubricants. Solid lubricants can mitigate crack propagation and reduce the shear strength and sliding contact of the polymer (Blanchet and Kennedy, 1992; Ye *et al.*, 2015). However, none of these functions of solid lubricants can be used to predict their effects on polymer structure, loading pattern and wear rate of such polymer (Omrani *et al.*, 2017). These phenomena also buttressed the need for characterisation of solid lubricants and polymers for better understanding and industrial application.

5. CONCLUSION

FFF produced objects suffers from anisotropicity in both mechanical properties and surface quality. Also, FFF made parts can only account for a 65 to 80% of the mechanical strength of the traditionally manufactured part of the same dimension and material. This review-study shed some light and need for the addition of fillers usually inorganic material to solve the deficiency of anisotropicity and weakness of manufactured parts by the FFF technique.

Nevertheless, the homogeneity of the FFF produced part, and its mechanical property is highly dependent on the dispersion and other processing parameters like temperature and building direction.

Besides, some high melting polymer had a high level of dimensional inaccuracy when printed via FFF technique due to high viscosity. This brought about the use of laminar solids to alleviate the viscous issue and at the same time, improve the mechanical properties. Nevertheless, most FFF researches focused on enhancing the mechanical properties with little attention to its tribological properties. Also, the addition of filler to FFF processes is quite unpredictable in terms of its mechanical properties at the end; especially laminar solid whose behaviour in the polymer matrix is highly dependent on its relative properties.

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