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Effect of Particle Size on the Properties of Highly-Filled Polymers for Fused Filament Fabrication

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Abstract. Fused Filament Fabrication (FFF) could replace injection molding as the shaping step in a process similar to powder injection molding (PIM). Herein after shaping by using a highly-filled polymer the part is debound and sintered to obtain a solid part of metal or ceramic. New feedstock materials have been developed that can be printed using conventional FFF equipment. And after debinding and sintering stainless steel parts can be obtained. However, there are many parameters that can affect the performance of the FFF feedstock materials. One important parameter is the particle size distribution of the filler particles. In this paper, feedstocks containing 316L steel powder with different particle size distributions were characterized in terms of viscosity and mechanical properties, and tested regarding the printability using a conventional FFF machine. It has been observed that the particle size significantly affects the properties of feedstock materials and thus their ability to be printed.

Keywords: additive manufacturing, fused filament fabrication, highly-filled polymer, suspension, viscosity, mechanical properties
PACS: 62.20.de, 62.20.fq, 66.20.Ej 81.05.Bx, 81.05.Lg, 81.05.Qk, 81.05.Rm, 81.20.Ev, 81.40.Jj, 81.70.Bt, 83.50.Uv, 83.60.Fg, 83.80.Hj, 83.80.Tc, 83.85.Jn

INTRODUCTION

Thermoplastic-based Fused Filament Fabrication (FFF) is one of the most widely used additive manufacturing processes in the world. The main reasons of its increasing popularity and use have been its reliability, safe and simple fabrication process, low cost, and the availability of a variety of thermoplastics for printing [1].

In FFF the printing material is heated until it melts or softens so far that it is able to flow and then it is extruded from a nozzle onto a substrate to build a structure in a layer-by-layer manner. The extrudate solidifies when its temperature decreases due to air convection [2].

As with other additive manufacturing techniques, FFF is able to produce parts with very intricate shape, for this reason it is believed that FFF could be used to replace injection molding machines when the number of parts needed is small or when the geometrical requirements are very demanding. This includes also Powder Injection Molding (PIM). In PIM a highly-filled polymeric system with metal or ceramic powders is shaped by injection molding, the polymeric system is then removed without affecting the shape of the molded part, and finally the part is sintered to obtain a solid part of metal or ceramic [3].

In order to successfully perform FFF, the quality of the filament is very important. This includes the material properties and the geometrical shape of the filament. In this paper, we investigated one important factor that affects the material properties strongly of highly filled systems: particle size distribution (PSD) of the filler powders [4, 5]. In short, this paper presents the effect of PSD on the modulus, elongation at break and apparent viscosity of highly-filled filaments with 316L stainless steel.

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MATERIALS AND METHODS

The highly-filled filaments for FFF were composed of a multicomponent binder system and filler particles. The main binder component in the binder is a thermoplastic elastomer (Kraiburg TPE GmbH & Co. KG, Germany). The backbone was a grafted polyolefin (BYK Chemie GmbH, Germany). The filler particles in the filaments were 316L stainless steel of two particle size distribution: P_S (Smaller) (Epson Atmix Corporation, Japan) and P_L (Larger) (Carpenter Powder Products AB, Sweden). All filaments had a 55 vol-% content of metal powder.

The particle size distribution was measured by laser scattering. Scanning electron microscopy was used to observe the shape of the two different powders. The particle size distribution and the shape of the two types of powders are shown in Figures 1 and 2, respectively.

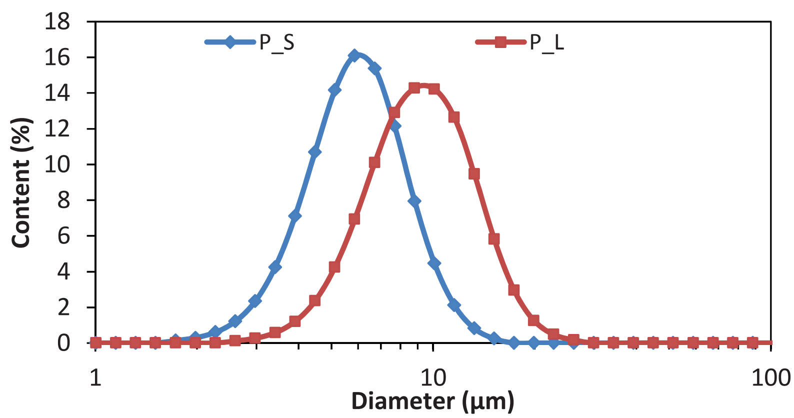

FIGURE 1. Particle size distributions of 316L stainless steel powders (P_S, small and P_L, large) measured by laser scattering.

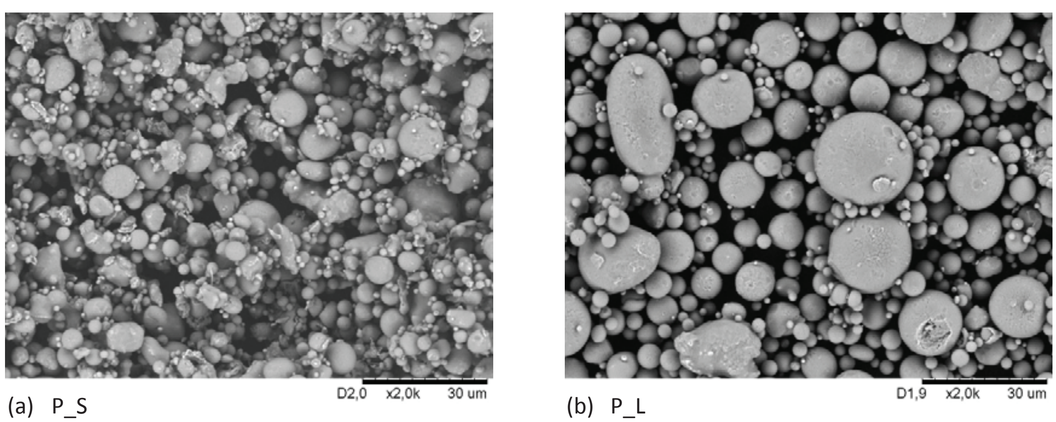

FIGURE 2. Scanning electron microscopy images of 316L powder (a) P_S, small and (b) P_L, larger particles.

P_S has a smaller average particle size of approximately $D_{50} \approx 5.5$ µm, while P_L of approximately $D_{50} \approx 8.6$ µm (Figure 1). Both powders have a normal distribution with a similar broadness; both powders have their standard deviations around 35 % from the mean value. Figure 2 shows that the majority of the particles in both types of powders are spherical. Thus it can be said that the only parameter changed is the average particle size

The feedstocks were compounded in a kneader with counter rotating rollers (Plasti-Corder PL2000, Brabender GmbH & Co. KG, Germany). The kneader temperature was set at 200 °C and the total kneading time was 30 minutes. After compounding, the feedstocks were grinded in a cutting mill fitted with a sieve with square perforations of 4 mm in length (Retsch SM200, Retsch GmbH, Germany). Two types of feedstocks were prepared: FT-P_S containing the smaller particles and FT-P_L containing the larger particles.

Filaments were prepared using a high pressure capillary rheometer as a ram extruder (Rheograph 2002, Göttfert Werkstoff-Prüfmaschinen GmbH, Germany). A barrel of 15 mm in diameter was used to melt the material before pushing it into a capillary. A round capillary with a diameter of 2 mm and length of 20 mm was used. Extrusion temperature was set at 185 °C and the piston speed was 0.5 mm/s. At the exit of the die a Teflon conveyor belt was placed to pull the filament as it was extruded at an approximately speed of 45 mm/s. At the same time when the filaments were produced the apparent viscosity was measured at the apparent shear rate of 113 s^{-1}, which corresponds to the piston speed and the geometry used during the measurement.

The mechanical properties of filaments were tested in a material tensile testing machine fitted with pneumatic grips (Zwick Z001 2.5 kN load cell, Zwick GmbH & Co. KG, Germany). The pneumatic pressure was 3 bar. Measurements were performed at 23 °C and 50 % relative humidity. Gauge length was set at 75 mm. In order to measure the apparent secant modulus an extension rate of 1 mm/min was used, afterwards the rate was increased to 10 mm/min until the sample broke. The apparent secant modulus was estimated between 0.05 % and 0.25 % extension.

After the filament characterization was made, printing trials were performed on a HAGE 3Dp-A2 fused filament deposition machine (HAGE Sondermaschinenbau GmbH & Co KG, Austria). Printing parameters were kept constant for both materials investigated.

RESULTS AND DISCUSSION

The properties for the materials investigated are summarized in Figure 3. Three properties are shown in Figure 3, the apparent viscosity measured during filament preparation (η), the secant modulus (E) and the strain at break (ε_{br}) for the filaments.

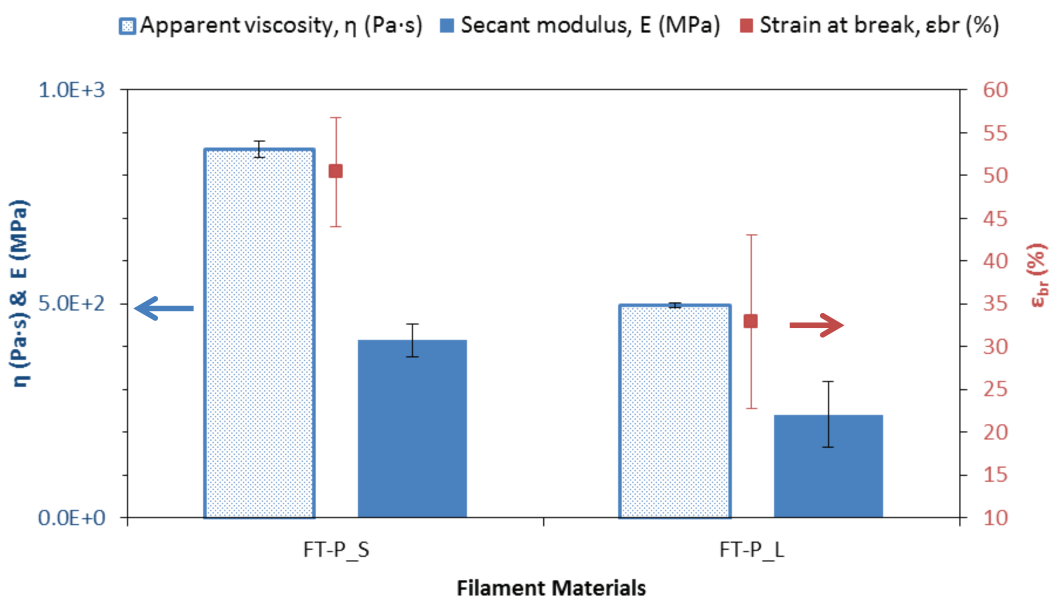

FIGURE 3. Apparent viscosity (η), secant modulus (E) and elongation at break (ε_{br}) for filaments with different particle sizes.

As it can be seen in Figure 3, increasing the average particle size of filler particles causes the apparent viscosity to decrease by ~42 %, the secant modulus decreases by ~42 % and the elongation at break decreases by ~35 %. A decrease in viscosity has also been reported in the literature when using coarser particles in PIM feedstocks [4]. Additionally, it has been reported in the literature of polymer-metal composites filled with particles that increasing the particle size decreases the modulus [5] and elongation at break [6] of the composite materials. Therefore all our results are in agreement with the literature. However, in order to determine if the filaments are useful or not, printing trials were performed.

Printing trials revealed that filaments FT-P_S can be printed and filaments FT-P_L cannot. Figure 4 shows some 3D printed parts produced with FT-P_S. Therefore it is clear that changing the average particle size of filler particles can lead to unprintable filaments. By looking at the results in Figure 3, one can speculate that decreasing the viscosity is not as crucial as maintaining a good level of modulus and elongation at break for the FFF printing process; nevertheless further investigation is need for conclusive evidence. But it can be concluded that in order to print feedstock materials with larger particles, adjustments to the binder formulation should be done in the future.

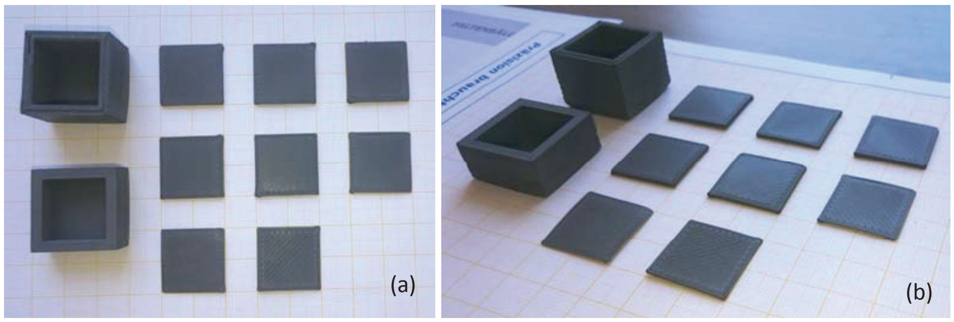

FIGURE 4. Parts produced by FFF using filaments with FT-P_S feedstock material (a) top view and (b) side view.

CONCLUSIONS

Highly filled composite materials (feedstock) have been prepared with 55 vol.-% of 316L stainless steel with two different particle size distributions. It was observed that increasing the average particle size from 5.5 to 8.6 µm decreases the apparent shear viscosity of feedstock, as well as the secant modulus and the elongation at break of filaments. The change in properties is significant since the filaments with smaller particles can be printed while the materials with larger particles cannot.

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