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3D PRINTING CONDITIONS DETERMINATION FOR FEEDSTOCK USED IN FUSED FILAMENT FABRICATION (FFF) OF 17-4PH STAINLESS STEEL PARTS

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Fused filament fabrication combined with debinding and sintering could be an economical process for 3D printing of metal parts. In this study, compounding, filament making and FFF processing of a feedstock material containing 55 vol. % of 17-4PH stainless steel powder and a multicomponent binder system are presented. For the FFF process, processing windows of the most significant parameters, such as range of extrusion temperatures (210 to 260 °C), flow rate multipliers (150 to 200 %), and 3D printing speed multipliers (60 to 100 %) were determined for a constant printing bed temperature of 60 °C.

Keywords: stainless steel, powder, additive manufacturing (AM), FFF, scanning electron microscopy (SEM)

INTRODUCTION

Additive manufacturing (AM) comprises a group of technologies used to build physical parts by adding material in a layer-by-layer fashion from a computer aided design (CAD) file, as opposed to subtractive manufacturing methods, such as machining [1]. AM is also referred as 3D printing, solid freeform fabrication (SFF) and rapid prototyping (RP) [2]. Over the last three decades, many AM technologies have been developed for the production of polymeric, metallic or ceramic parts [3].

One of the most commonly used AM technologies for the production of metal parts is selective laser sintering (SLS). Its biggest disadvantage is the dependency on high power lasers, which can be very costly. Therefore, fused filament fabrication (FFF) has shown great potential as a cost effective alternative [4]. In FFF, the building material is supplied in the form of spooled polymer-based filaments into a heating unit with a nozzle using counter-rotating drive-wheels. The heating unit is controlled to move in the X-Y plane, and as it moves, the material is extruded through the nozzle on a platform that moves in the Z-direction [3].

FFF was first developed to work with polymeric filaments. For the fabrication of metal parts, filaments made of a polymer highly-filled with metal particles can be used. After shaping the filament into what is referred to as the green part, the binder system is removed from the part and then sintered to obtain a final metal part.

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This paper describes a process for compounding a new feedstock formulation consisting of a proprietary binder system and 17-4PH stainless steel powder, the process of filament making, and the determination of 3D printing processing windows for the most significant parameters related to FFF.

MATERIALS AND METHODS

Materials

Feedstock materials for FFF are composed of a polymeric binder system and filler particles. Here, the binder system had three components: the main binder component, the backbone polymer and a compatibiliser. The main binder component was a soft and flexible thermoplastic elastomer (TPE). The polyolefin-based backbone and a commercially available compatibiliser were used in the feedstock. The fillers were 17-4PH stainless steel particles, whose size, measured by laser diffraction, is shown in Table 1.

Table 1 **Particle size data on 17-4PH stainless steel**

D10 / μm	4,2
D50 / μm	12,3
D90 / μm	28,2

Compounding

Feedstocks were compounded in a co-rotating twin-screw extruder (Leistritz Extrusionstechnik GmbH, Germany), designed for compounding highly-filled polymers with metal powders. All the binder components were premixed in solid state. The metal powder and the binder were fed using two gravimetric feeding units. The result was a feedstock material with 8,9 wt. %

of binder and 91,1 wt. % of steel powder. The extruded material was pulled away from the die with a conveyor belt and later granulated in a cutting mill.

Filament production

Filaments were prepared using a single-screw extruder (Dr. Collin GmbH, Germany). A round capillary with a diameter of 1,75 mm and length of 20 mm was used. At the exit of the die a conveyor belt was placed to pull the filament as it was extruded. After the filament left the conveyor belt it was guided to a spooling device where it was wound onto spools. A device to measure the diameter and ovality of the extruded filament was placed between the haul-off unit and the spooling device. According to the reading of the measuring device the extrusion and haul-off speeds were manually regulated to obtain a filament with appropriate geometry.

Printing trials

Each material to be used in an FFF machine needs to be checked for the conditions which lead to good quality printed parts. Printing trials were performed to determine the range of temperature, flow rates and printing speeds at which this novel material can be processed by means of FFF. Printing trials were performed on a Duplicator i3 v2 FFF machine with a nozzle diameter of 0,6 mm. The printing surface was glass coated with hair spray to enhance the adhesion of the feedstock to the glass. The printed parts were dog-bone specimens with a length of 70 mm, a thickness of 3 mm, a width at the narrow section of 4 mm and at the wider section of 12,5 mm.

The software Slic3r was used to prepare the G-code for printing. In this software, the following parameters were kept constant: layer height of 0,15 mm, first layer height of 0,2 mm, infill density of 100 %, rectilinear fill pattern for all layers, fill angle of 45°, speed of printing perimeters of 60 mm/s, infill printing speed of 80 mm/s, extrusion width of the first layer of 200 %, infill overlap of 15 %, and printing surface temperature of 60 °C. The varied FFF printing parameters are shown in Table 2.

Table 2 **Investigated printing parameters**

	Extruder temp. / °C	Flow rate / %	Printing speed / %	No. of perimeter lines
1.	260	100	50	1
2.	250	100	50	1
3.	240	100	50	1
4.	260	150	50	1
5.	260	150	70	2
6.	260	150	60	1
7.	240	150	60	1
8.	230	150	60	1
9.	220	150	70	1
10.	210	150	60	1
11.	260	200	100	1
12.	260	200	100	2

Microscopy

In order to observe the powder distribution in the binder, a filament and a printed part were investigated by means of optical and scanning electron microscopy (SEM). The filament and the printed part were cryogenically fractured under liquid nitrogen.

RESULTS

Filament quality

A constant geometry of the filament is important for its continuous transportation in an FFF machine. For this reason the diameter and the ovality of the produced filament were monitored during production. Please note that ovality is defined as the difference of the diameter in the horizontal direction and the diameter in the vertical direction. Thus, a truly round filament will have an ovality of zero as both diameters are equal.

The diameter had a normal distribution with an average diameter calculated to be 1,732 mm and a standard deviation of 0,020 mm. This diameter is acceptable even though one of the standard diameter sizes for FFF filaments is 1,750 mm. It has been observed by other researchers [5] that as long as the diameter is not below 1,700 mm, the material should be able to be printable without major problems. The ovality distribution was more or less constant with an average of 0,030 mm and a standard deviation of 0,017 mm, which is sufficiently low to consider the filament to be round.

The roundness of the filament can be seen in Figure 1. One can see that the stainless steel particles (brighter spots) are equally distributed in the filament, which is important to have an even distribution of particles in the printed and sintered parts.

The insert in Figure 1 shows a SEM image where the good compatibility between the particles and the binder can be seen, since there are no gaps between the binder and the individual particles. Additionally, cavities in the filament can be seen; these cavities could be the result of air trapped during the production of filaments via ex-

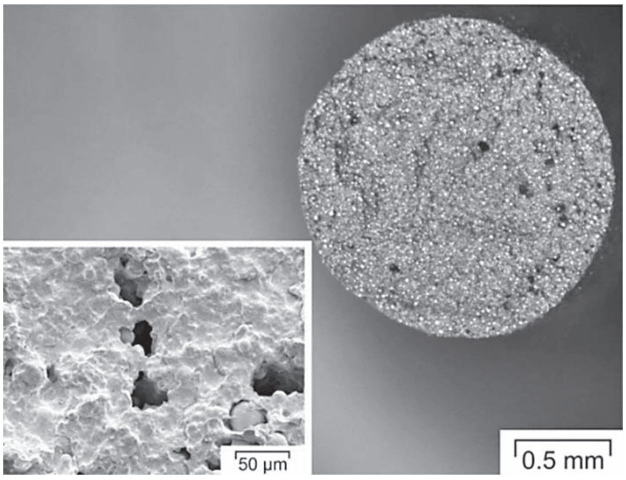
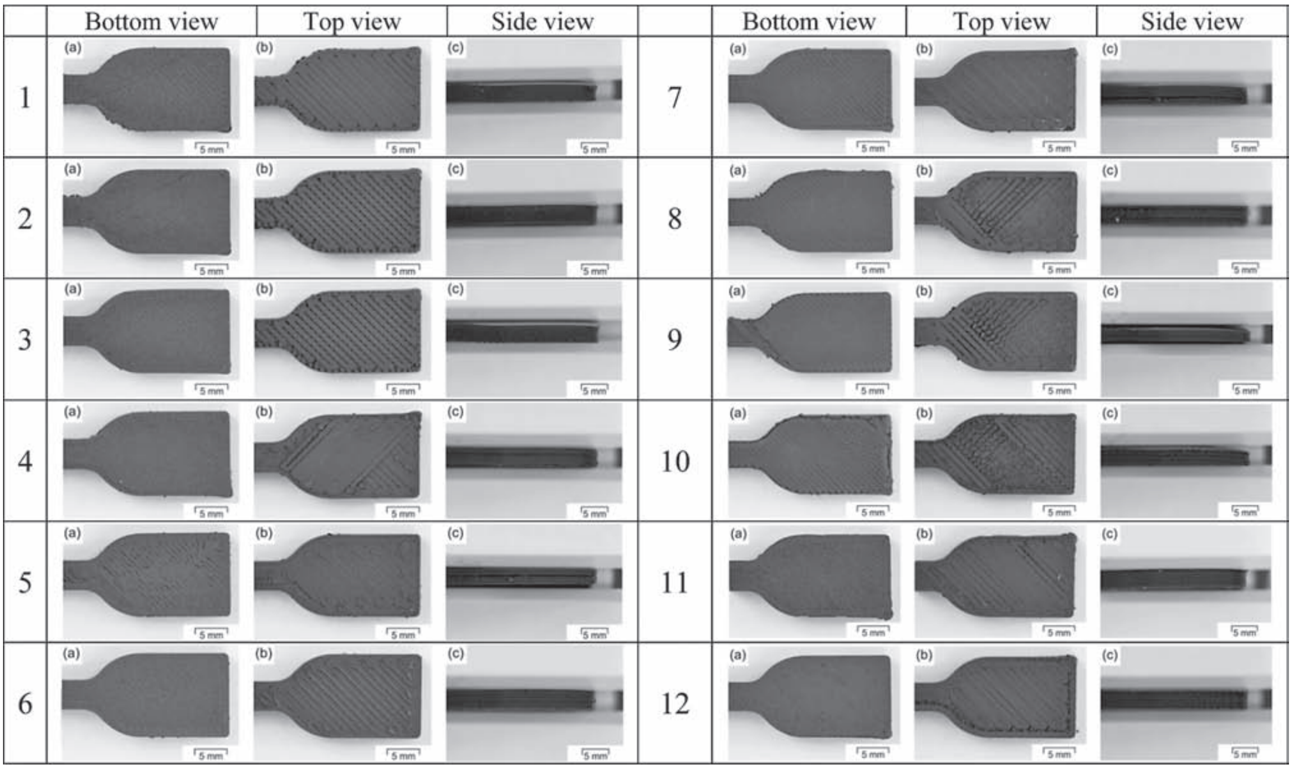


Figure 1 Overall view and detail of the filament filled with stainless steel particles.

Table 3 **(a) Bottom, (b) top and (c) side views of printed parts 1 to 12**



trusion. Cavities are undesirable because they weaken the mechanical properties of the filament, but if there are not too many cavities and they are small enough, the filament is still printable. As it will be shown in the next section; this was the case for the filaments produced in this investigation.

Printing trials results

The results of the printing trials are summarised in Table 3. In almost all printed parts the layer in contact with the printing bed had a smooth and continuous surface (column (a) in Table 3). The two parts with the worst bottom section are parts 5 and 10. In the case of part 5, the printing speed was not properly matched to the flow rate of the material, so there are voids between the layers. In the case of part 10, the extrusion temperature was the lowest; therefore, the material did not flow correctly to coalesce the printed strands. In general, the higher the extrusion temperature the smoother the surface looks (parts 2 to 4).

When the parts are looked at from the top view (column (b) in Table 3), there is a more noticeable difference among them. For parts 1 to 3, only the extrusion temperature was changed and the other parameters remained constant. It can be seen that the printed strands did not coalesce as the temperature was lowered from 260 to 240 °C. When the flow rate was increased (part 4 compared to part 1), the coalescence between the printed strands improved, but there is a risk of supplying too much material and blobs of material could appear. Blobs can be corrected by increasing the printing speed (parts 5 and 6). The printing parameters used for part 6 yield acceptable quality. Decreasing the temperature from 260 to 210 °C, while keeping the flow rate constant, generally leads to a decrease in the quality of the printed parts (parts 6 to 10).

The part with the worst quality is the part printed at the lowest temperature (part 10). Since not enough material was flowing, there are sections that were not properly filled and the strands did not coalesce. The results of the last two trials (parts 11 and 12) showed that the printing speed can be doubled (compared to part 1) if the flow rate is doubled, which still leads to acceptable quality. However, care must be taken when selecting the number of perimeter lines to be printed. For example, using two perimeter lines led to improper filling of part 12, but using one perimeter line leads to good filling in part 11. This can be attributed to the number of strands that can be completely fitted in the width of the printed part; the printer cannot deposit fractions of strands so it skips a strand if it does not fit. Printing parameters used for part 11 yield the best quality.

The side view (column (c) in Table 3) of the printed parts helps to determine the stability of the printing process. For example, part 1 appears to have good quality from the top and bottom view, but in the side view it is visible that there is a space between the printed layers. Another example is part 7, whose side view reveals that one layer was not properly printed, so there is a gap between two sections of the part. These errors during printing can arise from many places, for example from unexpected external vibrations, changes in environmental conditions, or even changes in the geometry of the filament.

Based on the results shown in Table 3, one can estimate a range of printing parameters within which the highly-filled filament presented can be processed by FFF. The extrusion temperature can be between 210 and 260 °C. The flow rate multiplier should be between 150 and 200 %. The printing speed should be between 60 to 100 %. The combination that seems to yield the best quality is using an extrusion temperature of 260 °C, flow rate multiplier of 200 %, and printing speed multiplier of 100 % and in this case one perimeter line. Because higher temperature decreases the viscosity of polymers, adding more material with lower viscosity improves cross-flow and results in a better contact between strands.

Microscopic morphology

The good distribution of the stainless steel particles (brighter spots) in the printed parts can be also seen in the cross-section of the printed specimens (Figure 2). Moreover, the individual printed layers can be seen in the SEM image shown in the insert in Figure 2. The arrows indicate the printed layer thickness. Some defects can also be observed as a result of errors occurred during printing, for example small voids between the layers.

CONSLUSION

A new material to be used in fused filament fabrication (FFF) has been developed. This new material is highly filled with 17-4PH stainless steel particles and could be used for the production of full metal parts after it is shaped, debound and sintered.

It was observed that this material can be processed by FFF when the extrusion temperature is between 210 and 260 °C, the flow rate multiplier between 150 and 200 %, and the printing speed between 60 to 100 %. The best quality was observed when using an extrusion temperature of 260 °C, flow rate multiplier of 200 %, and printing rate multiplier of 100 %. The printed surface was a glass mirror heated to 60 °C. Under such conditions an acceptable surface on the top and bottom sections of the printed parts is achieved.

The stainless steel particles are evenly distributed in the filament and also in the printed part. So it could be expected that sintering can be performed without destroying the printed shape as this binder system has already been proven to be solvent debindable and sinterable [6].

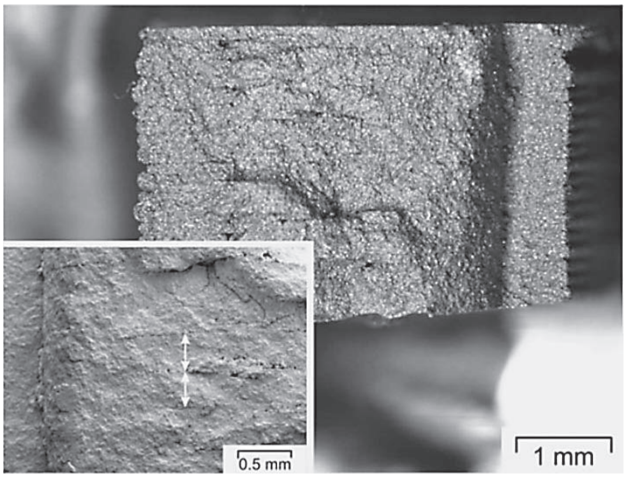


Figure 2 Overview and detail of fractured section of printed part

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Note: Responsible person for English translation is S. Kereković, Croatia