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Exploring high-stiffness pellets as filaments in fused filament fabrication

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Abstract

In Fused Filament Fabrication, there is increasing interest in the potential of composite filaments for producing complex and load-bearing components. Carbon fibre-filled polyamide currently has highest available strength and stiffness, but promising variants are not in filament form. This paper investigates filament production of commercially available, high-filled PA-CF pellets by modifying a tabletop filament extruder. We show filament production is possible by improving cooling. The FFF printed specimens show an average UTS of 135.5 MPa, higher than most commercially available filaments.

Keywords: additive manufacturing, production design, high-performance polymers, 3D printing

1. Introduction

Additive Manufacturing (AM) and High-Performance Polymers (HPP) have gained much attention due to the high demand for strong, complex, lightweight structures. Combining additives like carbon fibre (CF) with AM makes it possible to produce complex geometries and improve mechanical properties such as tensile strength and Young's modulus (Lionetto, 2021; Ning et al., 2015; Van De Werken et al., 2020). With increasing mechanical properties in the AM process, the designers are able to create new innovative solutions in load-bearing applications where high strength and stiffness are required. AM enables new designs by advanced geometries with close to no restrictions from the manufacturing process, and therefore AM is considered a new industrial revolution (Berman, 2012). AM is particularly beneficial for creating specialised advanced components, prototyping, and low-quantity production. Of all the available AM processes, Fused Filament Fabrication (FFF), also known as Material Extrusion (MEX) in the ISO/ASTM 52900 standard, is by far the most widespread, affordable and easy to use (Dey et al., 2021; Pandžić et al., 2019; Pandzic and Hodzic, 2022; Velu et al., 2021). However, the materials with the best mechanical properties are typically highly filled grades that are not commonly available in filament form. Exploring filament production with such materials can lead to new possibilities for the existing large-use base of FFF printers, enabling low-cost production and new design possibilities for load-bearing components. This article focuses on pushing the limits of currently available polymer composite materials by applying them in FFF. One of the most promising commercially available HPPs is short-fibre carbon-filled polyamide (PA-CF). While FFF generally results in highly anisotropic components**,** PA-CF has been highly modified and tailored for AM and has demonstrated a reduction in anisotropy when manufactured correctly (Bjørken et al., 2022).

Analysis of currently available materials reveals that materials developed for FFF generally possess much lower strength and stiffness properties compared to available pellets. Examples of top-rated commercially available PA-CF FFF filaments, PolyMide PA6-CF (Polymaker, Shanghai, China) with 20wt% CF, has a Young's modulus of 7.4GPa and Ultimate Tensile Strenght (UTS) of 105MPa in the X-Y direction and 4.3GPa and 67.7Mpa in the Z direction. Another commercially available filament, Luvocom® 3F PAHT CF 9891 BK (Lehmann & Voss & Co, Hamburg, Germany), a PA12-based material, filament with 15wt% CF has a Young's modulus of 11.5GPa and 130MPa UTS in the X-Y direction with no information on Z direction in the datasheet. In comparison, LUVOCOM 3F PAHT® CF 9743 BK, a pellet Luvocom claim is unsuited for filament production ("LUVOCOM," 2023) with 25wt% CF, has a Young's modulus of 28GPa and UTS of 250MPa (X-Y). The aim is to print this material, which is claimed to be unsuitable for filament production, by understanding how to make filament with high CF concentration and investigating the material properties printed by FFF. Many high-performance polymers are also available for injection moulding (IM) with high strength and stiffness. LUVOCOM® LFT 6-50581CF10NT is a pellet made with 40wt% CF made for IM with no publicly available datasheet, but the manufacturer claims high strength and stiffness, which is expected at such high CF fill grades. There are many other carbon fibre pellets made for IM, and starting to explore how these materials can be used in FFF has potential for both new designs and high-performance component production using complex geometries, allowing for novel designs. The greatest restriction of FFF when producing functional parts is the limited availability of suitable filament materials, which underscores the importance of exploring new filament materials to enhance mechanical properties and expand applications across diverse fields (Dey et al., 2021). The open-source community has the potential to share knowledge related to hardware on how to produce filaments of unsuitable materials, expanding the possibilities in FFF to create major advancements in high-performance polymers for FFF (HPPFFF). Creating open-source AM designs can contribute to a knowledge boost in the research field (Birkelid et al., 2022; Øvrebø et al., 2023; Pearce, 2012). The primary challenge in producing filament of LUVOCOM 3F PAHT® CF 9743 BK is related to its high wt% of CF, making it unsuitable for filament production (LEHVOSS Group, 2023). High CF content introduces various failure possibilities in filament production, most notably poor carbon fibre distribution and dispersion (Giles Jr et al., 2004; Øvrebø et al., 2023). CF can introduce the formation of gaps/voids in the material, making it brittle (3devoC, 2023). Additionally, the hygroscopic nature of PA worsens the issue, as any moisture in the material during the extrusion transforms into steam, causing the material to become foamy and brittle. Moisture in the material also causes adhesion loss at the interface between the fibre and the polymer matrix (3devoC, 2023; Kikuchi et al., 2020). An additional challenge emerges due to the abrasive characteristics of CF, causing wear and tear on the equipment (Giles Jr et al., 2004; LEHVOSS Group, 2023). A relatively low-cost filament maker, Composer 450 (3devo, Netherlands) in the 7500-9500€ range, operates by a puller wheel stretching the material into the correct diameter, a process complicated by the CF bonding in the PA matrix (3devoC, 2023). Additionally, high concentrations of CF may cause nozzle clogs, negatively impacting extrusion flow and resulting in uneven material surface and diameter variations. Lastly, According to Blanco et al. (2022), CF can increase the specific heat capacity in the final polymer composite, making cooling of the filament more essential.

This paper explore the production of high-stiffness pellets as filaments by filament production and FFF manufacturing of LUVOCOM 9743 BK. The filament was selected due to 25wt% CF, 5% higher CF wt% than the highest CF concentration produced by FFF to date. First, influencing process parameters are investigated and optimised using the Composer 450 with additional added cooling capabilities by dimensional accuracy while monitoring the thermal process from extruder screw to filament. After satisfactory filament production, ISO 527 samples are produced by FFF and tensile tested.

2. Materials and methods

This section outlines the method, equipment and modifications needed to explore and develop printable high CF-wt% filaments in FFF. The section describes the essential steps and modifications required to adapt the material to filament production. It addresses material preparation, optimisation of process settings, and a cooling solution to achieve the desired filament characteristics for 3D printing.

2.1. Method overview

Figure 1 shows an overview of the steps used in the article to explore creating a printable filament and material testing of a filament unsuited for FFF. The pellets used in this article is LUVOCOM 3F PAHT®

9743 BK (Lehmann & Voss & Co, Hamburg, Germany) with 25wt% CF with a Young's modulus of 28GPa and potential UTS of 250MPa. The 9743BK material was chosen for its high stiffness >25GPa and high potential in UTS.

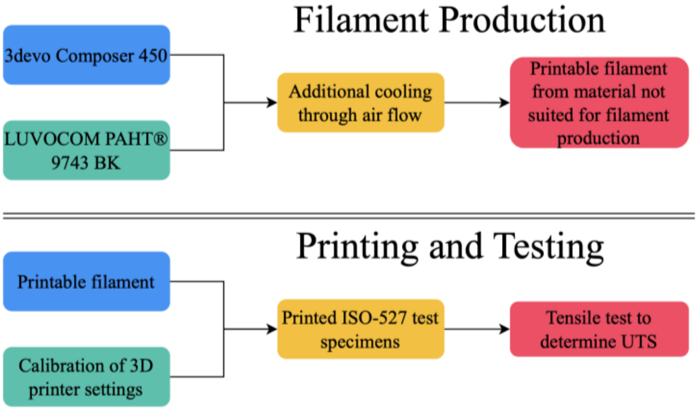

Figure 1. Flow chart describing the process for "Filament Production" and "Printing and Testing"

A series of events are required to process the pellets to filament, as seen in Figure 2. In accordance with the datasheet recommendation, the pellets were dried on a rack in an oven for 6-8 hours at 130°C (LEHVOSS Group, 2023). This step is essential to remove any moisture content that can disrupt the extrusion process, especially since PA is very hygroscopic (3devoC, 2023; Giles Jr et al., 2004; Kikuchi et al., 2020). After drying according to recommendations, the processed pellets were fed into the extruder's hopper using the filament maker Composer 450 (3Devo, Netherlands) (Figure2, step 2.).

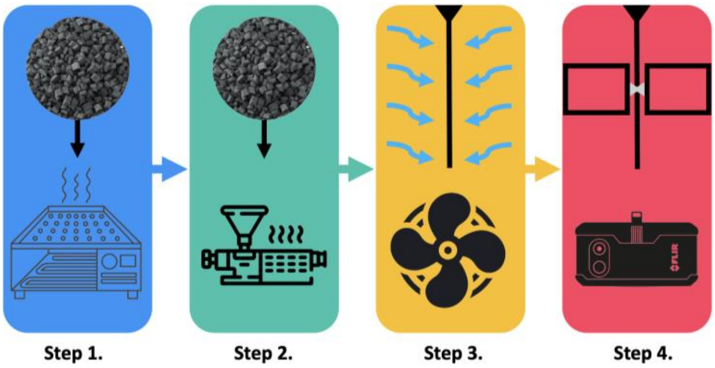

Figure 2. Step 1.) Drying PAHT-CF pellets at 130°C for 6-8h; Step 2.) Adding dried material to the filament extruder; Step 3.) Additional air cooling, cooling down the filament; Step 4.) Filament thickness- and thermal data collection

Initially, the filament was processed according to the technical datasheet with screw extrusion temperatures set to zone 4 (250 - 290°C), zone 3 (260 - 300°C), zone 2 (260 - 300°C) and zone 1 (260 - 300°C). The zones represents four parts of the screw, where zone 4 is closest to the material inlet, and zone 1 is closest to the nozzle. The extruded material was then pulled by a puller wheel to achieve the requested filament diameter, air-cooled when exiting the nozzle by two integrated fans (Figure 2, step 3). The material temperature during different stages of the process after exiting the nozzle was monitored by a thermal camera (Flir One Pro, Teledyne FLIR, USA). Due to the material not being suitable for filament production, it was challenging to achieve satisfactory filament quality using Composer 450 as the filaments did not achieve circular, even geometry, but a more flat cross-section, making them unprintable due to exceeding the 3mm extruder diameter. The quality of a filament is considered good when it has a consistent diameter (2.85±0.15mm) throughout the length of the filament and is free of contaminants or other impurities. Additionally, the filament should have a uniform texture and be free of warping or other defects. Warping happens when the extruded material is not uniformly cooled, bending in the direction of cooling. Visual inspection showed that high extrusion temperature with low extrusion speed gave better filament quality, but it was hypothesised that additional cooling would further increase the quality.

2.2. Experimental setup

To determine the optimal settings for PA filament fabrication with 25wt% CF, a systematic approach of iterative testing was conducted. For heaters 1-4, temperatures were set within the limits provided in the technical data sheet for the material (LEHVOSS Group, 2023). Furthermore, an extrusion speed of 6-8 rpm (rounds per minute) proved to produce the highest quality filament. Using the insights from the initial explorations, an experiment was designed to optimise the filament quality (Table 1). All tests were conducted in ventilated laboratory conditions at 23 °C.

Table 1. Overview of 3devo Composer 450 Settings

Setting ID	Heater 1	Heater 2	Heater 3	Heater 4	Extrusion speed	Fan speed
1	285 ˚C	290 ˚C	275 ˚C	260 ˚C	7 rpm	100 % = 0.8 m^3/min airflow
2	285 ˚C	290 ˚C	275 ˚C	260 ˚C	7 rpm	80 %
3	285 ˚C	290 ˚C	275 ˚C	260 ˚C	7 rpm	60 %
4	285 ˚C	290 ˚C	275 ˚C	260 ˚C	6 rpm	100 %
5	285 ˚C	290 ˚C	275 ˚C	260 ˚C	8 rpm	100 %
6	290 ˚C	295 ˚C	285 ˚C	270 ˚C	7 rpm	100 %

The 3devo Composer 450 filament extruder is a compact desktop filament maker solution to create custom 3D printing filament at home (3devoA, 2023). It is an all-in-one machine that processes pellets and uses four heaters and a single screw extruder to melt and blend the material before releasing it through the nozzle. See Figure 3B for a front view of the Composer 450 with marked areas where the fans are pointed. Two air fans are cooling the filament below the nozzle (Cooling 1). A puller wheel will then draw the filament through a thickness sensor to measure the adjustable filament diameter. Using the stock Composer 450, the initial best results all used the 100% cooling 1 configuration. Multiple cooling configuration prototypes were made to assess and optimise filament quality. The final setup featured two MagLev PSD1209PLV2-A fans (Sunon, China) mounted on a stand, positioned to direct airflow midway between the nozzle and the puller wheel (Cooling 2). The two fans increased the cooling airflow to 5.3 m^3/min (Sunon, 2009). These fans were placed 100mm from the Composer 450. A picture of the setup, including the additional fans and a thermal camera, can be seen in Figure 3A.

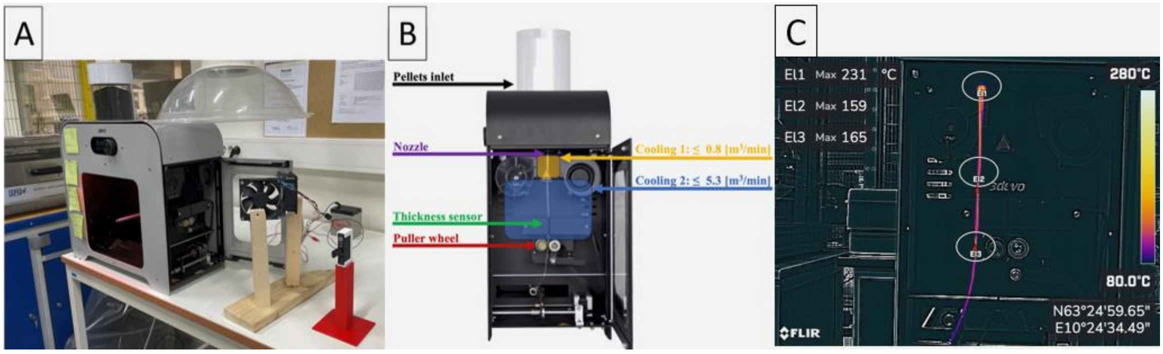

Figure 3. A) Experimental setup with cooling prototype and thermal camera for temperature measurement. Fan stand placed 100mm from the Composer 450 during extrusion; B) Cooling zones and extruder front view; C) Thermal data acquisition

When extruding the filament using the Composer 450, the influencing factors on filament quality are temperature when exiting the nozzle, speed (time spent within the cooling zones), and cooling rate. Monitoring the actual temperature of the material exiting the nozzle, the material temperature in the cooling zone, and the temperature before entering the puller wheel will allow for process analysis and replication of the experiment using different filament extruder setups. The thermal camera (Flir One Pro, Teledyne FLIR, USA) was used to gain deeper insights into filament temperature and cooling dynamics. The camera was positioned to capture consistent thermal images at positions El1 (nozzle), El2 (middle), and El3 (puller wheel) throughout various tests. A thermal image is shown in the figure below (Figure 3C). After the exploration phase, in the final experiment, 12 different settings were explored using different extrusion screw temperatures with and without additional cooling. Thermal data allowed direct comparisons between the previous test setup and the new one with extra cooling (Figure 3). The 12 different production configurations were used to produce filament to determine the width ratios. After stabilising the process by extruding a minimum of 200g material, maximum and minimum thickness were measured at minimum 5 points along a selected 300mm of extruded filament from a longer filament spool. In addition, visual defects were also measured and included in the average calculations. The filament was measured with a digital calliper, and the average cross-section was calculated from the different measurements.

2.3. Specimen printing and testing

The process of FFF for producing tensile test specimens according to the ISO 527 1B standard was done using a modified Vivedino Troodon CoreXY (FORMBOT, China) with a modified high-flow, high-temperature hot-end with 100mm heated zone using a 400W 230V heater. The filament with a diameter of 2.85mm (± 0.15mm) was 3D printed using optimal settings for PAHT. Five test specimens were printed using the highest quality filament in the X-Y direction, which is the strongest (Podsiadły et al., 2021), and then tested for tensile strength using the MTS Landmark 370 - 50kN Axial Load tensile test machine. The UTS was measured in accordance with the ISO 527 standard at a test speed of 1 mm/min. The cross-sectional area was measured using a digital calliper around the fracture, and the tensile strength was calculated thereafter.

3. Results

This section shows the improvement in filament quality achieved through additional cooling. It includes the outcomes of tensile tests on ISO 527 specimens printed using the newly produced filament. These results are compared with the quality and strength of PolyMide™ PA6-CF (Polymaker, 2023), a commercially available PA-CF filament, one of the few high-performance materials where out-of-plane (ZX) properties are provided in the technical datasheet.

3.1. Calibration and validation

A direct comparison between filament quality is done by testing all the different settings IDs listed in Table 1, with and without additional cooling. The resulting cross-section ratios (largest diameter divided by smallest diameter) are shown in Figure 4.

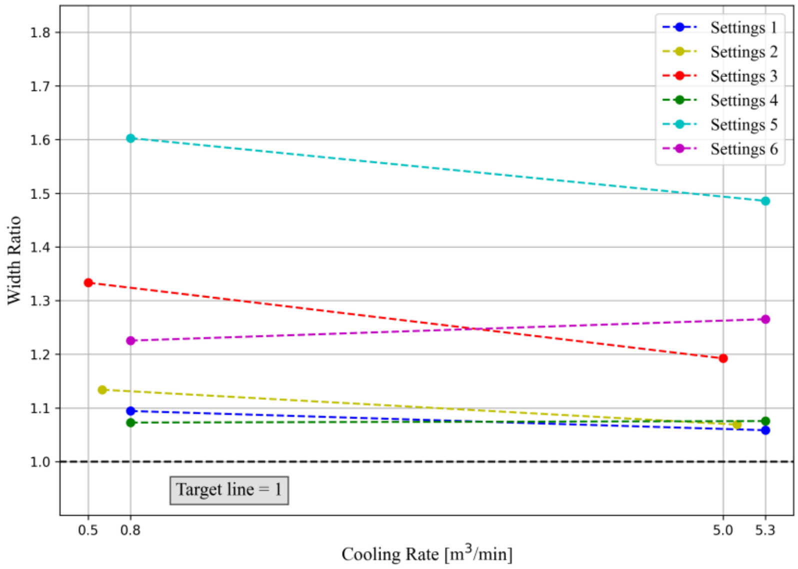

Figure 4. Width ratio or cross-section ratio after producing filament with the six different settings, with and without additional cooling

Looking at the width ratios for the highest cooling rate, setting 1 produced the best filament closest to 1.0, while setting 5 had the highest ratio. Figure 5 shows the gathered temperature data at key locations during filament extrusion, comparing the same settings used with and without additional cooling in each plot.

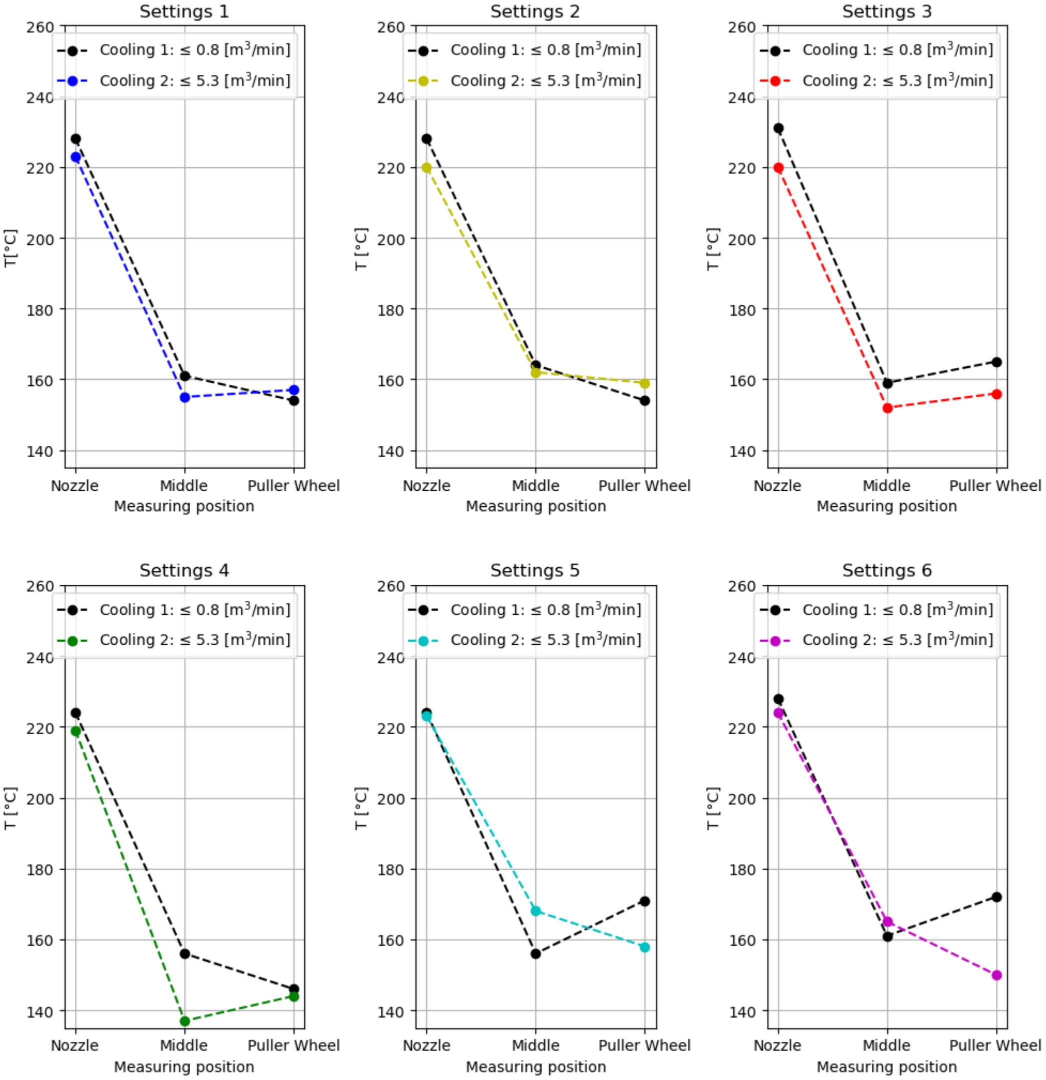

Figure 5. Temperature distribution across measuring positions for two different cooling levels

Figure 5 shows a slightly lower material temperature when comparing the original cooling 1 and additional cooling 2 when the material exits the nozzle through all configuration settings. In settings 1 and 2, the temperature seems to have stabilised when measuring the middle position, with minor material temperature changes compared to the puller wheel. In setting 3, the final temperature is similar to 1 and 2, but the material has not reached a steady state with a temperature increase after the cooling zones. In settings 4, 5, and 6, there is an observed temperature change, either a decrease or an increase, based on the various cooling configurations. Setting 1, 2 and 4 with additional cooling provided the best width ratio (Figure 4). Figure 6 shows microscopy pictures of the filament produced with the setting 1 configuration with different levels of external cooling. A microscopy picture of PolyMide™ PA6-CF has been added as a direct comparison. The filament in the left picture in Figure 6 exhibits unsatisfactory characteristics, characterised by uneven quality and a width ratio of 1.09. Furthermore, in the image in the middle of Figure 7, the filament has a much smoother surface and roundness, with a width ratio of 1.01—falling within the desired tolerance range of (2.85 ± 0.15 mm).

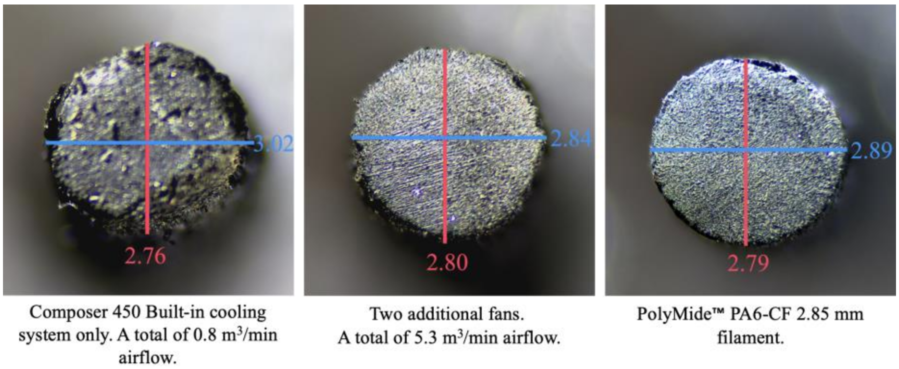

Figure 6. Microscopy pictures of filament produced using settings 1 with three different amounts of airflow cooling. Compared to PolyMide™ PA6-CF

3.2. Tensile strength

Figure 7 shows the tensile testing results of seven specimens that were produced using FFF from filament made with LUVOCOM 3F PAHT® 9743 BK pellets using setting 1. The average tensile strength is 135.5 MPa, with a standard deviation of 6.24 MPa. When comparing the results to the values provided in the PolyMide™ PA6-CF technical data sheet, high-stiffness pellets can be produced by FFF with approximately 30MPa higher UTS in the XY-direction (Polymaker, 2023).

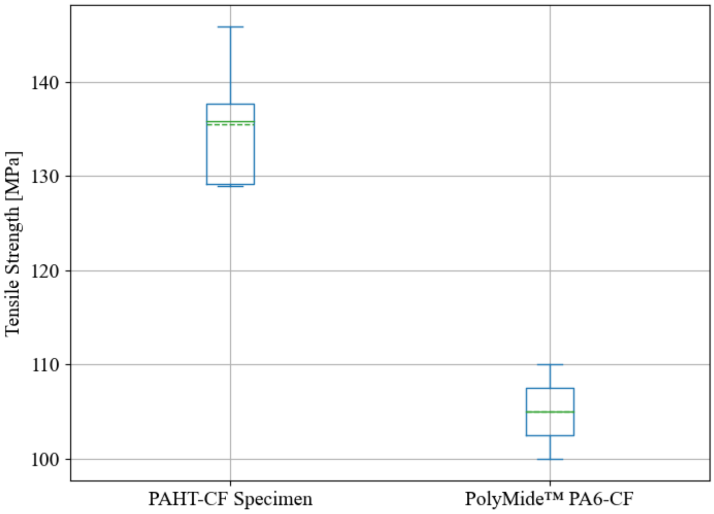

Figure 7. Tensile strength box plots of printed PAHT-CF specimen compared to PolyMide™ PA6-CF

4. Discussion

In Figure 5, the filament temperature exiting the nozzle, in the cooling zone and exiting the puller wheel provide insights into how to produce dimensionally accurate filament suitable for FFF. While setting 4 showed the lowest puller wheel temperature of approximately 145 °C, setting 1, with a puller wheel temperature of approximately 158 °C, offered the best dimensional accuracy. Comparing the two configurations, setting 1 obtained close to thermal stability with similar temperature recordings in the middle cooling zone and puller wheel, while setting 4 increased by almost 10°C exiting the cooling zone. We, therefore, argue that the additional cooling while maintaining thermal stability will enable the production of high-stiffness pellets to filaments. Inspecting the microscopy images (Figure 6), the filament quality produced without implementing the new cooling solution is considered unsatisfactory. It has a width ratio close to 1.10 and is, therefore, unusable as a printable filament. Immediate improvements in filament quality were demonstrated in this article by prototyping and testing with more cooling through airflow. The PolyMide™ PA6-CF filament has a 20 wt% carbon fibre, compared to the Luvocom pellets that have 25 wt%. Increased carbon fibre wt% leads to greater heat capacity and thermal conductivity in the material, meaning that higher temperatures and more cooling is needed to create a well-blended filament (Blanco et al., 2022). Due to the high carbon fibre content, achieving a more consistent filament diameter than ±0.15 mm was problematic with the low-cost experimental setup. The filament appeared uniform, though, and still printable. The width ratio results (Figure 5) show the impact the filament extruder settings have, especially the screw's RPM. By turning the RPM down to 6 or up to 8, the pellets were not well-blended, leading to low filament quality, visually and in terms of roundness. Therefore, the testing showed that 7 RPM produced the highest filament quality.

4.1. Filament production challenges

Creating filament of a material unsuitable for filament production had several challenges. The amount of cooling was the main problem after optimising the settings on the machine. The two small fans integrated in the Composer 450 provided minimal cooling to the PAHT filament, leading to flat filament when pulled through the wheels. This problem is the main reason the experimental setup was developed, with additional airflow cooling and the opportunity to take temperature data at critical locations in the machine. Thermal data comparison was used to better understand the cooling dynamics and the impact on filament quality. Uniform cooling was essential in preventing material warping, ensuring consistent filament measurements by the sensor. On the other hand, if the filament was cooled down too rapidly its stiffness made the puller wheel unable to create the correct diameter. Finding the optimum trade-off was key for high-quality filament creation. Inspecting the thermal data plots (Figure 5), the optimum seemed to be around 150-160 ˚C which complies with being slightly beneath the material's crystallisation temperature. The filament had notably better quality when the temperature difference between the middle and nozzle position was small, like in settings 1. Based on a similar material (9742 BK), but with another wt% CF, this temperature is approximately 175 ˚C (Dul et al., 2021). Assuming the air in the lab had the same temperature when cooling the different extrusion settings meant quantifying the airflow as volume per minute, which made it easy to compare different fan models and prototypes.

Furthermore, the pellets produced a significant amount of carbon fibre dust due to their high wt% of carbon fibre. Washing the pellets led to higher water absorption, making drying even more critical. The dust was contaminating the filament extruder, making the produced filament visually hairy and uneven, indicating poor distribution and dispersion of carbon fibre. Despite thorough washing, there is still a possibility that the extrusion was affected by the carbon fibre dust. The material pellets were initially meant for either injection moulding or direct pellet printing, methods that may be less susceptible to the effects of dust production. When producing filament, extruding well-blended pellets at a constant rate is essential to ensure roundness and even filament (Øvrebø et al., 2023).

4.2. 3D Printing and tensile testing

According to the technical data sheet, the tensile strength of "LUVOCOM 3F PAHT® 9743 BK" is 250 MPa. The strength is determined using an "ISO 3167 A" test specimen by injection moulding. Injection moulding creates better material bonding because it is uniformly heated, cooled, and pressurised (Striemann et al., 2020). During 3D printing, the layer adhesion is weaker, and when comparing ABS printed vertical, flat, and horizontal directions gives a tensile strength of 33%, 60%, and 65%, respectively, compared to injection moulding (Podsiadły et al., 2021). However, by properly investigating the layer fusion temperature using PolyMide PA6-CF from Polymaker, a study shows that layer bonding of PA6-CF is only 30% weaker printed horizontally (Bjørken et al., 2022).

With the right approach to filament production and 3D printer calibration, it becomes feasible to manufacture components that possess up to 65% of the strength of their injection-moulded counterparts using the same material. If such strength is achieved, filaments manufactured from IM pellets will outperform all currently available commercial filaments. The tested specimen of the PAHT filament had an average tensile strength of 135.5 MPa, 55.3% of the strength provided in the technical data sheet (LEHVOSS Group, 2023). This means there is major room for improvement in high-performance FFF filaments, where the future will lead to new design opportunities with increased strength and stiffness at low production costs.

Annealing the specimen before testing is an example of how to improve the strength. Annealing can increase the tensile strength by up to 14% (Arjun et al., 2022). The standard deviation of the box plot values is 6.24 MPa and indicates that the data points are spread out over a wide range. The main factor causing this is inconsistent filament diameter, which can change the overall extrusion rate during printing. Some specimens experienced under-extrusion, weakening them in the tensile tests. There is room for improvement in the way tensile strength is calculated. The use of callipers to measure cross sections did not account for voids or gaps caused by CF or under extrusion, resulting in inaccurate calculations. To achieve more precise results, compensating for these errors by utilising microscope images of the fracture would be beneficial.

5. Conclusion

In engineering design, new opportunities may drastically change current equipment designs as new production methods, such as Fused Filament Fabrication, become cost-efficient, reliable, and capable of producing components that sustain real-world load cases. FFF also has the potential to provide full design freedom by removing geometric limitations from manufacturing. The exploration of new materials will further increase these opportunities as the potential of short carbon fiber re-enforced pellets is significantly better than what is currently commercially available as filament. Recently, new relatively low-cost desktop solutions have become available. By combining off-the-shelf hardware with open-source hardware, exploring the potential of pellets as filaments may lead to a major improvement in material properties, thus adding new design possibilities for designers. This research demonstrates that with new cooling solutions, precise temperature control, and testing, materials that were once considered unsuitable for FFF can be transformed into high-quality filaments, expanding the range of usable FFF materials. These findings allow for more creativity and flexibility when selecting materials for FFF applications, demonstrating the adaptability of materials traditionally used for other processes and bridging the gap between what is possible in injection moulding and FFF. The materials were successfully converted from pellets to filament for FFF with an average strength of 135.5 MPa, which is a relative reduction to injection moulding, as reported by previous studies.

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