Extrusion of Carbon Fiber-Reinforced Polyetheretherketone

Manufacture of FFF-Filaments

The manufacture of high-quality plastic filaments for 3D printing is demanding. The challenges vary depending on the used polymers and polymer fillers. As part of a research study at the Institute of Polymer Materials and Plastics Technology at the Technical University of Clausthal the requirements of the process were investigated, especially with regard to the necessary preparations and conditions of the plant periphery.

Fused Filament Fabrication (FFF) is an additive manufacturing process used to process thermoplastics. This can be both amorphous and semi-crystalline thermoplastics. Material processing based on a filament is specific to this process. For the production, the thermoplastics must be extruded and wound.

The challenges of this procedure vary depending on the respective polymers and the plastic fillers which are used, for example in the form of different processing temperatures, flow properties, abrasion behaviors or elasticity.

Polyetheretherketone (PEEK) is one of the most common high-performance polymers. It is used particularly in the automotive and aerospace industries. The thermoplastic is characterized by high strength values and high temperature resistance [1]. The melting temperature of PEEK is around 340 °C. The related process temperature is in the range from 370 to 420 °C [2]. This results in a high output requirement for the entire processing peripherals.

In additive manufacturing, only a few 3D printers can process polyetheretherketone these days. This is mainly due to the required temperature control. Due to the relatively high cooling rates in the FFF process, the crystallization behavior of the high-performance polymer has a significant impact on the quality of the print results. Pure polyetheretherketone tends to severe material distortion (warping) [3, 4], especially when large, flat parts with high infill rates are printed. For this reason, leading filament manufacturers are increasingly using carbon fibers (short fibers <600 µm). The high rigidity of the carbon fiber counteracts the part distortion in the 3D printing process [5].

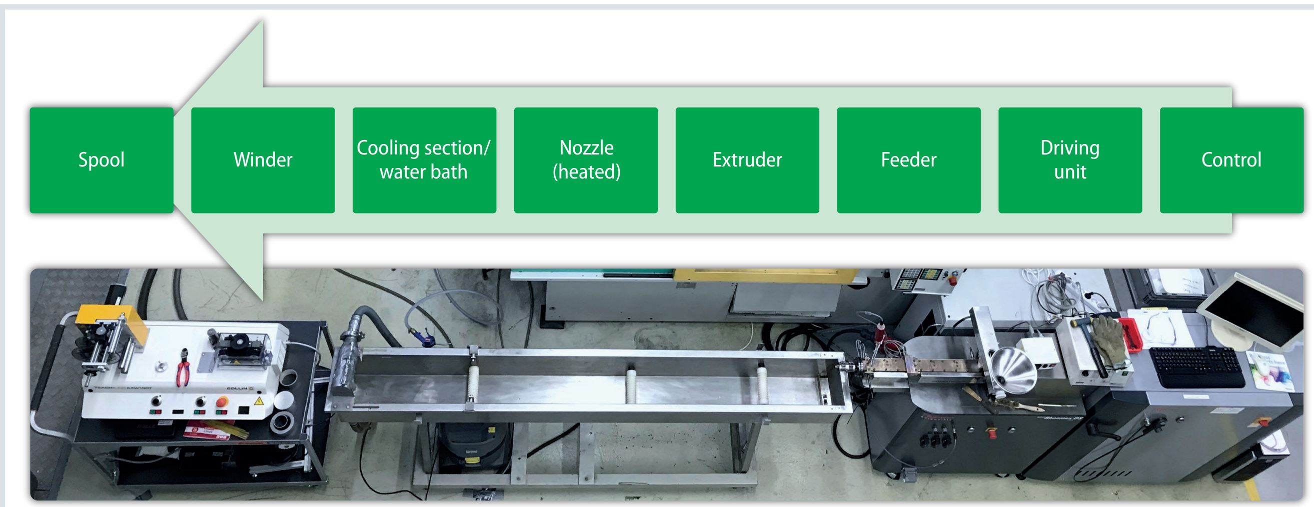

Fig. 1. Schematic representation of the production line for the manufacturing process of plastic filament. © Prigann/Institute of Polymer Materials and Plastics Technology, TU Clausthal

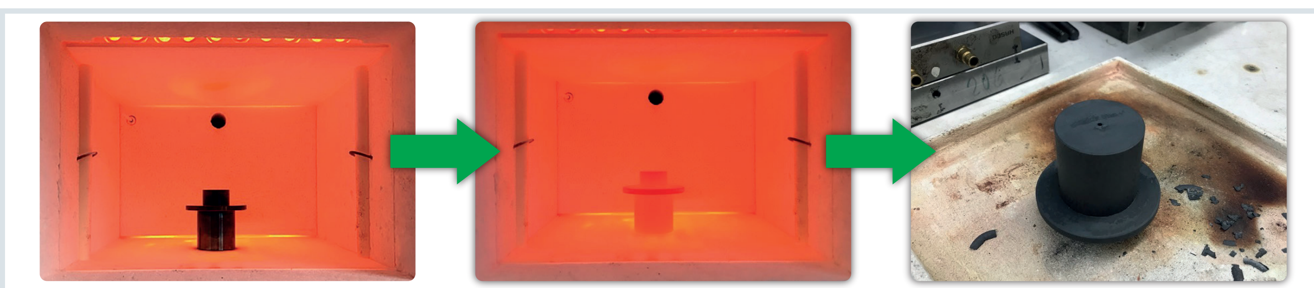

Fig. 2. Necessary surface hardening of the extruder nozzle for filament production of carbon fiber filled plastic (standard hardening process 42CrMo4). © Prigann/Institute of Polymer Materials and Plastics Technology, TU Clausthal

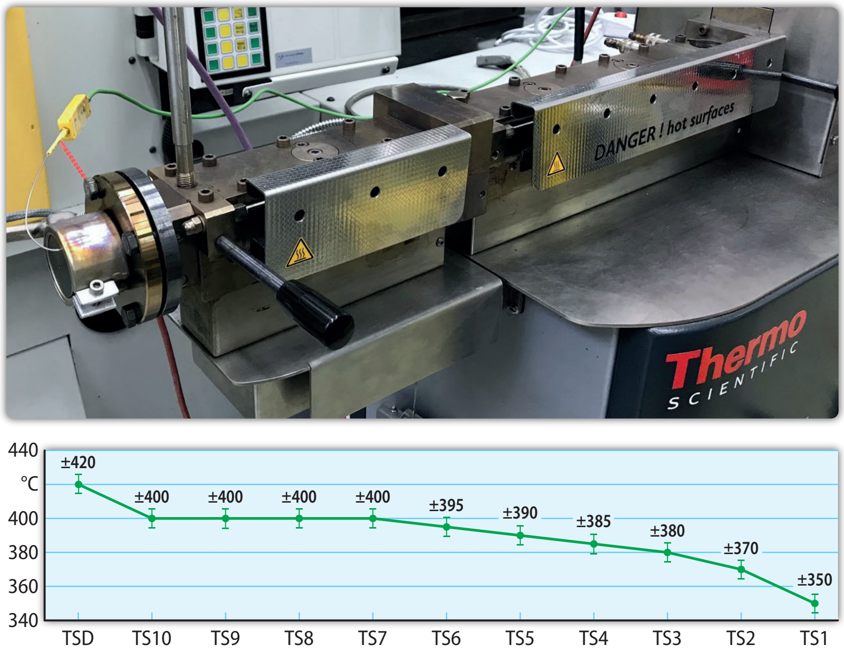

Fig. 3. Temperature control in the extrusion process of carbon fiber-reinforced PEEK. © Prigann/Institute of Polymer Materials and Plastics Technology, TU Clausthal

Periphery for Filament Production

The filament production of polyetheretherketone with a carbon fiber content of 30 % was tested as part of a research study at the Institute of Polymer Materials and Plastics Technology at the Technical University of Clausthal, Germany. The research background was to investigate the precautions that are necessary to achieve sufficient process stability along the entire production line of the filament manufacturing process. Basis of the investigation is the PEEK 450CA30 granulate from the polymer manufacturer Victrex. The underlying production line for filament production can be seen in the schematic representation in **Figure 1**.

The production line consists of a twin-screw extruder (Haake Rheomex OS PTW16) including the associated drive unit (Haake PolyLab OS RheoDrive 7) from Thermo Fisher Scientific. The plastic granules are fed in by a feeder (Haake Metering Feeder MF 1) from the same device manufacturer. The extruder nozzle is an in-house construction made of 42CrMo4 (hardened steel). This material was surface-hardened because preliminary tests with carbon fiber-filled polyetheretherketone showed high abrasion on the nozzle (**Fig. 2**). The inner diameter of the nozzle in this research study is 2.00 mm ± 0.10 mm with a length of 40.00 mm ± 0.10 mm (L/D ratio: 20). This value is based on already validated manufacturing processes for commercially available 1.75 mm filament.

The extrusion of high-temperature plastics requires an external nozzle heater. A commercially available heating sleeve was used for this (power 300 W; supply voltage 230 V). To cool the extruded plastic, it is guided through a cooling section. This is usually a temperature-controlled water bath. However, this is not the case for polyetheretherketone because the temperature gradient between the melting point and the ambient temperature is too high. For this reason, air cooling is sufficient. The difficulty lies in the rapid cooling of the plastic and the associated stiffening of the filament, which is why the cooling section must either be kept short or tempered. The filament was wound using a filament winder (Teach Line AZW130T) from the system manufacturer Collin Lab & Pilot Solutions as well as a self-made stepper motor-controlled coil. Welding wire spools are preferable due to the brittle fracture behavior of carbon fiber filled polyetheretherketone. Compared to conventional 3D printing coils, these have a larger outer diameter.

Process Parameterization for Filament Production of PEEK-CF30

The processing temperature of Victrex polyetheretherketone is in the range of 350 to 400 °C [6]. Preliminary tests showed a stable manufacturing process with a temperature profile of 350 to 420 °C (**Fig. 3**). Depending on the heating rates and due to the thermal inertia, sufficient holding times must be planned after the heating-up phase in order to ensure the set process temperatures. The hold time in the research study conducted here was 60 min. ± 5 min. An additional temperature comparison of two independent temperature measurement methods is advantageous to that (**Fig. 4**).

To adjust the entire production line, consisting of feeder, extruder and winder, it is necessary to coordinate the conveying speed of all devices. The discharge speed of the extruded material (filament) and the peripheral speed of the winder must be designed to be congruent. When material is discharged, it has to be ensure that the convey- »

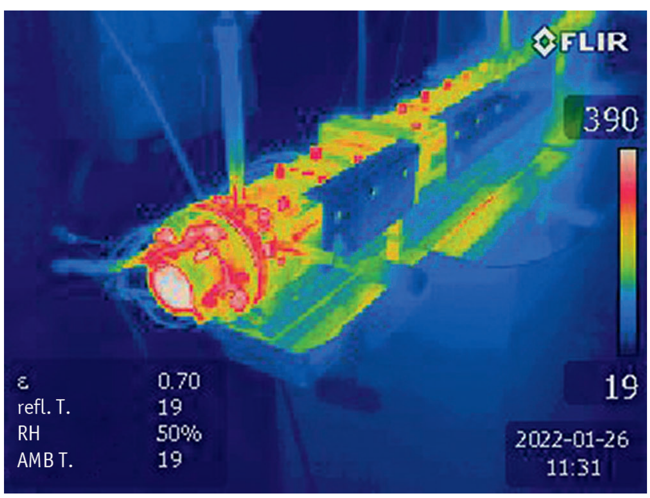

Fig. 4. Thermography of the extruder and nozzle temperature in the extrusion process of carbon fiber-reinforced PEEK. © Prigann/Institute of Polymer Materials and Plastics Technology, TU Clausthal

Fig. 5. Temperature loss during filament production of high-temperature plastics (without additional post-tempering processes).
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ing rate remains below the critical ratio of conveying speed to the nozzle length, otherwise there will be a loss of surface quality (e.g. shark skin) [7]. Based on results from preliminary tests, the conveying speeds for polyetheretherketone should be kept low in order to achieve sufficient homogenization in relation to the amount of heat introduced.

The speed of the drive unit was 30 rpm ± 0.1 rpm, with a resulting torque of 100 Nm ± 10 Nm. The pressure in front of the nozzle inlet rose to 95 bar ± 5 bar. These values vary depending on the design and configuration of the extruder which is used.

After extrusion, the plastic filament is conveyed through the cooling section. In this study no additional temperature control was used, which is why the filament winding took place immediately after the extruder nozzle outlet. Otherwise, the filament would break when winding up during the deflection process, as described above, due to its brittle material behavior.

At a room temperature of 20 °C ± 2 °C, the temperature loss within the first 100 mm after the extruder outlet was around 150 °C (**Fig. 5**). With the described production line and based on the presented process parameters, it was possible to produce PEEK-CF30 filament with a diameter of 1.2 mm ± 0.2 mm (**Fig. 6**). In consideration of the L/D ratio of the extruder nozzle which was used here, the diameter reduction between the nozzle and the extruded filament was around 40 %. One of the decisive factors is the filament pulling speed of the winder, which significantly reduces the filament diameter due to the resulting tensile force on the extrudate.

Regarding to the material diameter of commercial 3D printing filaments, a nozzle diameter of 3.00 mm ± 0.10 mm is recommended. In this case, however, a new parameterization of the entire production line is to be expected. Validation attempts for this research hypothesis are currently still pending at the Institute of Polymer Materials and Plastics Technology. ■

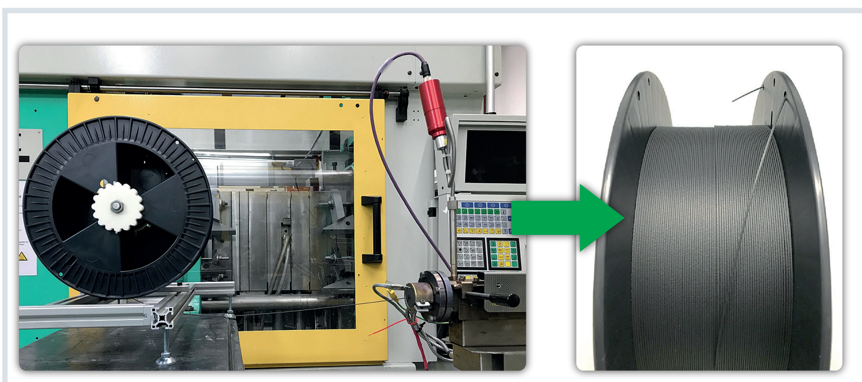

Fig. 6. Winding of carbon fiber-reinforced PEEK (welding wire spool). © Prigann/Institute of Polymer Materials and Plastics Technology, TU Clausthal

Info

Text

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