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Material Extrusion (MEX) Based Additive Manufacturing with Highly Filled Carbon Polymer Compounds

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Abstract

Today highly filled carbon compounds are used in special applications with the need for high electric or thermal conductivity at high chemical resistance processed only by conventional manufacturing technologies. The additive manufacturing of highly filled carbon polymer compounds enables new possibilities in design, prototyping and manufacturing on demand in these fields of application. Recent studies either concentrate on additive manufacturing of compound composites with low electric conductivity or high mechanical stability.

This paper investigates the electric conductivity in special of different carbon compound formulations with filler share rates of up to 80%. This paper also shows material behavior, influence factors and challenges in the use with material extrusion. The results of investigations lead to new insights and conclusions for product design and material processing in additive manufacturing of highly filled carbon compounds. The process shown in this work has the potential for application in industrial additive manufacturing of carbon products like graphite bipolar plates used in hydrogen fuel cells. This automatically leads to new innovations in these fields of application due to the potential of additive manufacturing in general.

Keywords: Additive Manufacturing, Material Extrusion, Highly Filled Compounds, Carbon Polymers and Electric Conductivity

Introduction

In recent years, additive manufacturing has developed into a serious and economical alternative to conventional manufacturing processes in many areas [1,2]. In addition, additive manufacturing processes enable new design options that lead to innovations in various areas of application [3,4]. A widely used additive manufacturing process is material extrusion (MEX). In the MEX process, a raw material that is either still solid or already viscous is applied in layers in a highly viscous form using energy and a nozzle [5]. One of the best-known MEX processes for example is Fused Deposition Modeling (FDM) [6,7].

3D printers that work according to the FDM principle use a solid raw material in coil form, so called filaments. These filaments are based on various plastics such as Acrylonitrile Butadiene Styrene (ABS) or Polylactide (PLA) [8,9]. Pure plastics limit the variety in possible areas of application. In order to expand the potential of additive manufacturing and the area of application for MEX processes, new materials are needed with properties specifically tailored to different applications, such as high thermal conductivity, chemical resistance or high electric conductivity. However, there are currently only very low electric and thermal conductive filaments on the market that are suitable for niche applications with low conductivity requirements, such as ESD protection. Due to their chemical structure, plastics have insulating electric and thermal properties. This means that many applications requiring high electric or thermal conductivity are reserved for other material groups such as metals.

Electric or thermal conductivity can be created by incorporating conductive fillers such as graphite and carbon black into plastics, known as compounding [10]. Graphite and carbon black are different forms based on carbon. Graphite has a crystalline structure, whereas carbon black has a disordered structure. Gnanasekaran have already considered the

positive influence of carbon as filler on the electric conductivity of filaments for FDM 3D printers [7]. But in this work only compound with a filler share of less than 10% are considered in order to be able in processing these filaments on common desktop 3D printers.

This work only considers highly filled carbon polymer compounds with a filler share of more than 50%. The physical properties of these highly filled compounds are determined by both the fillers and the polymer. On the one hand, certain electric and thermal conductivities can be precisely adjusted using various compound formulations. On the other hand, mechanical properties that are important for use or processing, such as rheology and mechanical stability, are influenced. To date, such highly filled carbon polymer compounds have only been further processed into products with the desired properties using subtractive or formative manufacturing processes. In addition to the known limitations in the realization of certain product geometries, these are also associated with procedural limitations in the processing of such compounds. Another option is to develop a process for the additive manufacturing of products made from highly filled carbon polymer compounds.

Carbon polymer compounds are used, for example, in actual very popular applications such as hydrogen fuel cells or electrolyzes, which require high electric and thermal conductivity as well as chemical resistance to hydrogen. A bipolar plate made from a carbon polymer compound, for example, is the central component within a PEM fuel cell and has the task of separating the individual cells from each other and thereby conducting the generated power per cell through the fuel cell stack. Therefore, electric conductivity and stable permeation of hydrogen are crucial criteria for the use of a graphitic bipolar plate in a fuel cell. So far, the processing of carbon polymer compounds into bipolar plates for PEM fuel cells has been primarily done by hot pressing or injection molding and in some cases also by milling. All these actual methods come with significant limitations in their application.

The filler share in injection molding is limited due to the increasing viscosity as the filler share increases. Hot pressing, on the other hand, has long cycle times and high energy consumption due to the heating and cooling of the solid steel pressing mold. When milling, there are difficulties in realizing detailed geometries. The use of highly filled carbon polymer compounds in additive manufacturing using material extrusion presented in this work represents a new alternative to produce bipolar plates for PEM fuel cells, for example to create new design options, flow fields or special variants as well as to realize small series of PEM fuel cells cost-effectively [11].

Materials and Methods

Materials

Inserting fillers into polymers as part of a compounding process enables new materials with special properties to be created. The characteristic properties of these fill-er-containing polymers largely depend on the properties of the used polymer and the properties of the used filler material. By specifically inserting various fillers, the properties of the individual components can be combined in a similar way to metal alloys.

By inserting conductive fillers such as carbon into a polymer, its electric conductivity can be increased significantly. For this purpose, carbon is introduced into a thermoplastic polymer as part of a compounding process and dispersed homogeneously [10]. To achieve high electric conductivity, a high proportion of filler must be achieved in the compound. Drummer and Lopez have already investigated the effects of the filler share on the material properties of carbon compounds [12,13]. Figure 1 shows schematically the basic dependence for carbon compounds between various material properties and the filler share, reproduced from these investigations.

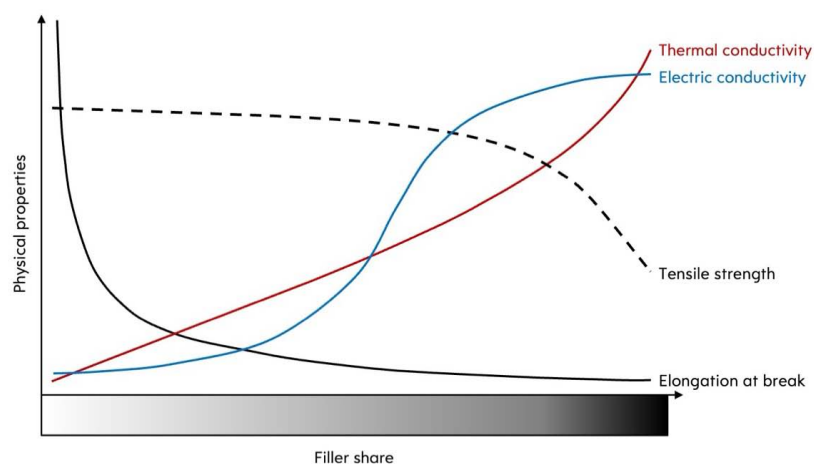


Figure 1: Illustrated Development of Physical Compound Properties at Carbon Polymers with Increasing Filler Share [10,12 and 13]

Mechanical parameters such as elongation at break and tensile strength show a clear negative dependence on the filler share. However, due to the distribution of the filler in the polymer and the formation of conductive paths in the

compound, both the electric and thermal conductivity increase with increasing filler share. With regard to electric conductivity, this effect can be described by the percolation theory. Here, probabilities for the occupancy of individual fields of a matrix are considered independently of the neighboring fields [14,15]. Relating to highly filled carbon polymer compounds, this explains how, as the proportion of conductive filler particles increases, and there are more and more points of contact in the otherwise electric insulating polymer matrix, which lead to conductive paths within the compound. The percolation threshold characterizes a range in which the resistance behavior changes by several orders of magnitude depending on the filler share [16,17]. In this area, the first continuous conduction paths within the compound are created between the individual filler particles.

In this study, two flexible polypropylene types with low modulus of elasticity and high tensile elongation serve as the base polymer. Two types of graphite and one carbon black are used as filler materials. The two PP types are a copolymer with a melt flow rate of 100 g/10min (PP100) and a homopolymer with a melt flow rate of 18 g/10min (PP18). The two graphite's used are a synthetic graphite with an average grain size < 45 µm (G45) and a microcrystalline graphite with an average grain size < 10 µm (G10). For a three-phase compound, a conductive carbon black (R) with a specific surface of 66 m²/g is also used. In compounds, carbon black usually acts as a "bridge" between individual graphite particles and thereby significantly increases electric conductivity. Table 1 lists the compound formulations considered in this study.

#	Polymer	Filler	Filler share	Compound
1	PP100	G10	75 %	75_25_G10_PP100
2	PP100	G10	80 %	80_20_G10_PP100
3	PP100	G45	75 %	75_25_G45_PP100
4	PP100	G45	80 %	80_20_G45_PP100
5	PP18	G45	75 %	75_25_G45_PP18
6	PP18	G45	80 %	80_20_G45_PP18
7	PP18	G45+R66	70 % + 7 %	70_7_23_G45_R_PP18

Table 1: Produced Highly Filled Carbon Compounds for Comparison in Additive Manufacturing with Material Extrusion

These compound formulations were produced on a Rheomex PTW 25/36p twin screw extruder from Thermo Electron with a constant mass throughput of 5 kg/h and an extruder screw speed of 200 min⁻¹. The polymer and filler supplies were realized using three gravimetrically controlled metering units from K-Tron. The polymer was dosed directly into the main feed, while the graphite and carbon black were fed into the melted plastic via side feeders. The air and other volatile components that were inevitably inserted with the large amount of filler were removed from the mass flow with the help of two degassing zones. The compound formulations were then extruded into individual strands and ground into granulates with a grain size of < 2mm. This entire production process is shown in Figure 2.



Figure 2: Production Process of Highly Filled Carbon Compounds for Additive Material Extrusion

MEX Process

In additive material extrusion (MEX), a thermoplastic polymer is fed through a heated metal nozzle [18]. The melted polymer is applied layer by layer to a build platform using a kinematic structure. A common kinematic structure, for example, are portal systems with the three spatial axes X, Y and Z. According to DIN EN ISO/ASTM 52900, for the material feeding at the print head, a distinction can be made between feeding filaments, granulates and dispersions. The feeding of granulate offers a high degree of flexibility when processing different starting materials in terms of their form and composition [5,19]. Figure 3 shows the schematic structure of a MEX 3D printer with a granulate extruder.

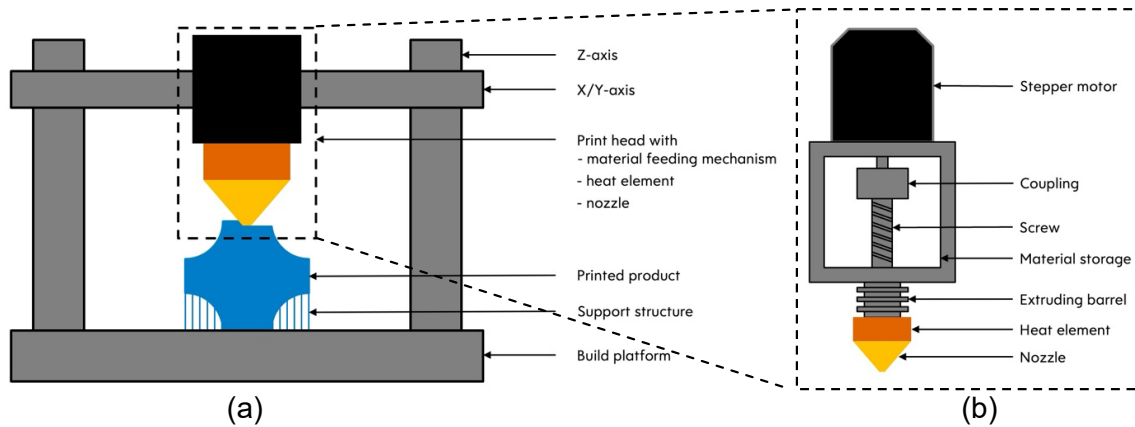


Figure 3: Sketch of a Material Extrusion Printer (A) With A Granulate Extruder (B)

For the production of test specimens from carbon polymer compounds a 3D printer based on the RepRap platform was designed and built. The resulting modular structure allows the use or exchange of central components to carry out various test series and iterative optimization of the respective manufacturing process for various compound formulations. With this structure, all influencing variables can be recorded and their effects on the entire manufacturing process in terms of the extrusion process can be analyzed and disturbances can be eliminated. For this purpose, the manufacturing of test specimens was carried out on a granulate extruder with an 8mm thick screw extruder which is driven by a NEMA stepper motor with a power of up to 0.45 Nm.

With regard to the design of the test specimens, a design suitable for determining electric conductivity was chosen in the form of a square with an edge length of 30 mm and a thickness of 2 mm. Since this work is not about determining mechanical characteristics and quality classes, the design of these test specimens bases on the investigations made by Gnanasekaran and creates a comparability of the results with low-filled and low-conductive polymer filaments [7].

In the series of manufacturing tests, the extrusion temperature was set to the usual working range for PP filaments of 200 °C to 220 °C. A uniform nozzle diameter of 0.4 mm, which is common in additive manufacturing using material extrusion, was chosen. Based on VDI 3405, all test specimens were printed fully filled [20].

Characterization of Electric Conductivity

The area-specific volume resistance is recorded to characterize the produced test specimens made from different compound formulations with regard to their electric conductivity. The measurement of resistances is based on measuring the voltage drop with a constant current applied to the specimen. For this purpose, the produced test specimens are clamped between two gold-plated copper poles ($A = 3.75 \text{ cm}^2$) whose contact force can be linearly controlled using compressed air. Gas diffusion felt (GDL) is applied to each pole, whose contact force and pressure-dependent resistance curve is known. The current was then conducted via the measuring poles along the entire measuring section. Two measuring poles are available for this purpose. One without and another with deenergized measuring needles, so that the measured resistances can be broken down into contact and bulk resistance. The measuring needles are located concentrically in the measuring pole and are electrically isolated from it and powered off. Thanks to the measuring setup with a measuring needle, the total resistance obtained can be broken down into its partial resistances. The total resistance R_{TOT} describes the resistance of the entire measuring section. The volume resistance R_V describes the resistance of the produced specimen (= total resistance minus the known GDL resistances). The bulk resistance R_{Ω} describes the resistance that is measured via the passively measuring tips that penetrate the specimens. The contact resistance R_C describes the simple contact resistance from GDL to the test specimen.

The measurement was carried out at four different contact pressures (5 bar, 10 bar, 20 bar, 30 bar). At each measuring point, four different currents (0,5 A; 1,0 A; 1,5 A; 2,0 A) were passed through the test specimens and the required voltage was recorded. The total resistance R_{TOT} of the specimen was determined from the slope of the best-fit line of the four measuring points through the origin. After subtracting the known GDL resistances, this results in the specific volume resistance R_V of the specimen. In order to generate reliable measured values, five identically produced test specimens were measured for each processing point (variation of pressing time and pressing temperature) and their measured values were then averaged.

Results and Discussion

Extrusion Behavior

Following Gnanasekaran it is not possible to meaningfully extrude the highly filled carbon compounds, described in 2.1, with the filament 3D printers available on the market [7]. Additive material extrusion fails here because of the material feeding mechanism. Due to the high filler share, filaments made from carbon compounds have a very low elongation at break. Mechanical material feeding, for example via pinions, quickly leads to the filaments breaking and thus to an interruption in the feeding process. Furthermore, the high thermal conductivity of highly filled polymers leads to

difficulties during the melting process. In common filament 3D printers, the power of their heat elements often is not sufficient to melt carbon compound filaments homogeneously and consistently. This can be explained by a sharply decreasing temperature gradient towards the middle of the filament, which results from the high thermal conductivity in the outer layers of the filament. The inconsistent melting behavior in turn leads to the print head becoming clogged and thus also to an interruption in the material feeding process.

A granulate feeding mechanism according to Liu et al. on the other hand, allows the continuous and reliable material feeding of highly filled materials without interruptions if the extruder and its dimensioning are designed accordingly [19]. With the help of the implemented experimental setup, described in 2.2, and the torque applied in the experiments, it was possible to achieve a consistent melting behavior in the extruder and thus a continuous mass flow of the melt through the extruder nozzle. It was observed that a different run-in phase is required depending on the filler share so that a continuous and high-grade mass flow with little dimensional deviation from the nozzle diameter can be achieved. Furthermore, it can be observed that changes in speed of the extruder screw during the manufacturing process as well as frequent changes in direction of the extruder have a direct influence on the part quality that can be achieved. Figure 4 shows the result by this additive manufacturing process with material extrusion of test specimen for characterization of physical compound properties with increasing filler shares.

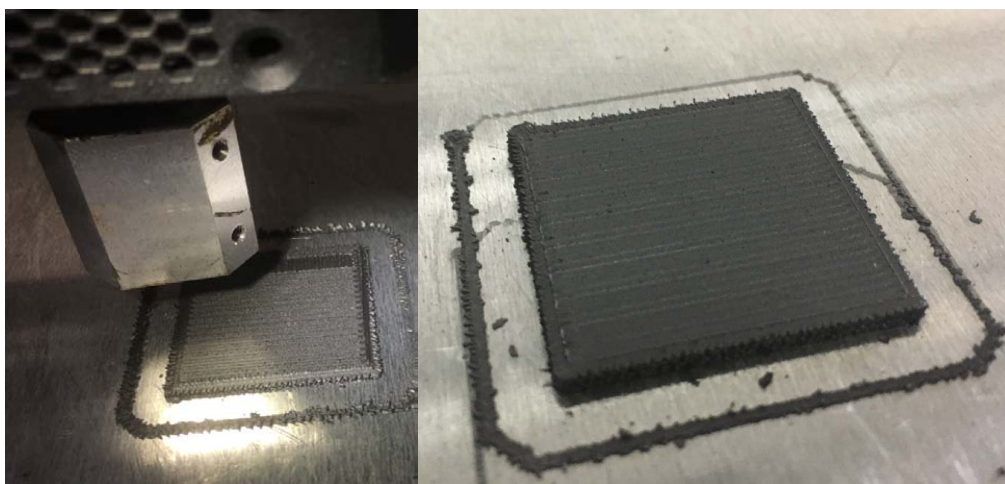


Figure 4: Additive Manufacturing with Material Extrusion of Test Specimen for Characterization of Physical Compound Properties with Increasing Filler Shares

With the extruder structure used, a graphite amount of 80 percent proved to be the maximum filler share that could be processed. In compound formulations with carbon black as a secondary filler, the proportion of graphite as a primary filler had to be reduced accordingly. As the extrusion tests progressed, increased abrasive wear was observed on the extruder, the screw and the nozzle. This wear is due to the high filler share of graphite and, similar to injection molding, leads to fluctuations in the manufacturing process as it progresses. These observations are confirmed by investigations on directly extruded compound strands using the methods of Ehrenstein [21].

As the filler share increases, the porosity in the processed compound also increases. This in turn leads to an immediate increase in the probability of contact between the individual slate-shaped graphite particles and the rotor, stator and the nozzle of the extruder. Figure 5 provides an impression of the described extrusion behavior of highly filled carbon polymers in additive manufacturing using material extrusion.

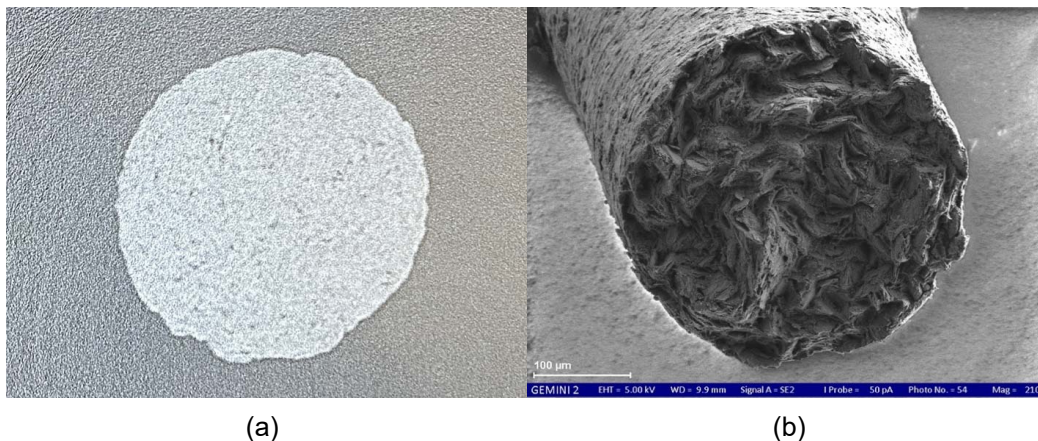


Figure 5: Examination of the Material Extrusion Behavior of Highly Filled Carbon Compounds by (A) Computed Tomography and (B) Scanning Electron Microscope

For further characterization, an important insight regarding the particle orientation in the extruded compound can be achieved. The existing literature assumes a three-layer structure in the polymer melt in common injection molding of highly filled carbon compounds which results in anisotropic properties in the molded parts. However, additive material extrusion results in a homogeneous polymer melt. In this case the anisotropic properties in the produced part arise through the layer-by-layer application of the material. This is a very important difference regarding the design process for later products that shall be produced out of specific carbon compounds by either injection molding or additive material extrusion [22].

With the additive manufacturing of carbon polymer compounds using material extrusion, limited post processing is necessary after the manufacturing process, depending on the compound formulation. On the one hand, any cavities must be freed of compound residues after removal from the installation space. In individual areas isolated material adhesions may have formed during production and must be removed. On the other hand, an abrasive processing, for example by grinding, is necessary on the top side facing the print head in order to minimize contact resistance.

Electric Conductivity

The produced test specimens were then electrically characterized, see Figure 6. For later comparison, the values of injection molded specimen (BK) from some compound formulations are listed here in addition. Surprisingly, the specimens from compound formulations with a high-grade extrusion behavior and a correspondingly well manufacturing result had the worst conductivity values. For PP100 with filler share of 80% values are missing, because the additive manufacturing of these specimens failed due to extrusion problems. Furthermore, it was shown that the smaller graphite G10 caused a lower resistance in the test specimens without shares of carbon black compared to G45. As expected, the specimens with graphite and carbon black as filler had the lowest electric resistances. This confirms the suspicion that the insertion of carbon black as a bridge between individual graphite particles significantly increases the electric conductivity, even in additive manufacturing using material extrusion.

When comparing additive manufactured to injection molded test specimens, the bulk resistance of additive manufactured specimens is significantly higher. The reason for this can be explained due to the contact resistances of the individual layers. In contrast to this the contact resistances of additive manufactured specimens tend to be lower compared to injection molded ones. This can be explained on the one hand by the rough surface of the additive manufactured specimens and on the other hand by the polymer-rich surface of the injection molded specimens.

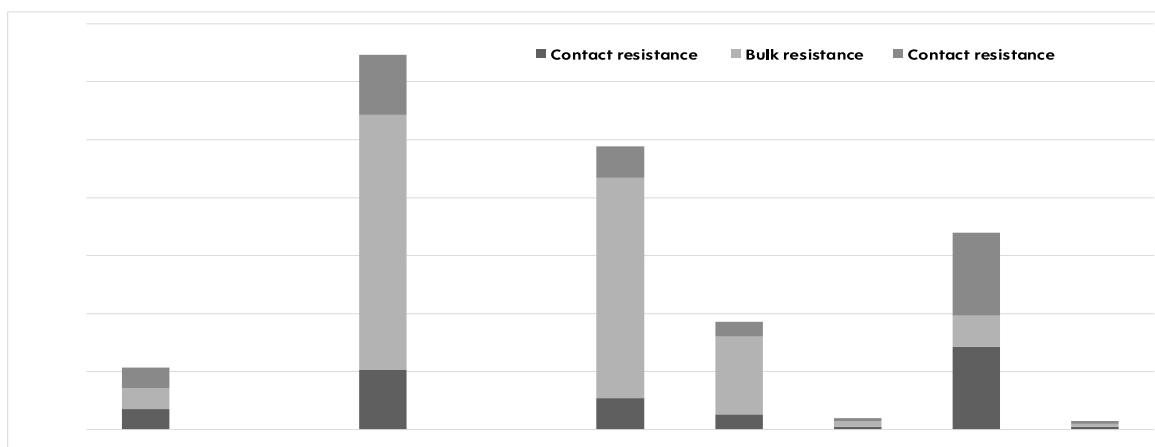


Figure 6: Electric Resistances of Test Specimen Made Out of Highly Filled Carbon Polymer Compounds by Material Extrusion Compared to Injection Molding (BK)

Conclusions

The additive manufacturing of highly filled plastics on 3D printers, which normally work with starting materials in coil form or filaments, is associated with various problems, from filament production to material feeding and extrusion at the print head to layer application. Even during filament production, current extrusion systems cannot achieve a constant filament diameter over the entire length due to the inhomogeneous filler distribution. Due to the high modulus of elasticity of highly filled polymers, compound filaments cannot be formed into the usual coil form and fed on conventional 3D printers that work according to the FDM technology associated with the MEX process.

A material feeding process via guides, rollers or gears leads to a break in the filament after a short time and thus to an interruption of the feeding process. High filler shares of electric and thermal conductive compounds result in a high viscosity of the melt, which in turn leads to increased mechanical wear and blockages on the print head. The high thermal conductivity of carbon compounds also prevents the filament from effectively melting in the print head, meaning that safe processing of highly filled compounds is not possible on common additive manufacturing systems that can be assigned to the MEX process.

One solution for solving these issues is to additively process highly filled compounds in the form of granulate instead of in filament form. For this purpose, before the additive manufacturing process starts, the compound are first converted into granulate through an additional upstream production process [23]. This granulate can then effectively and reliably be feeded into the direction of a multi-zone heating section via an extruder screw, similar to the functional principle of a classic extruder [24]. Both during production and during feeding, care must be taken to ensure that there is no segregation of polymer and filler components, as this has a direct impact on the melting behavior during the manufacturing process and the quality of the end product in terms of the required electric, thermal, chemical and mechanical properties. Furthermore, material-specific temperature control must be ensured during the heating process [25]. For example, excessive cooling of the feed zone can cause the respective compound to soften too late or too slowly.

Insufficient cooling can result in the polymer portion of a compound overheating and causing the material to clump together. Both effects lead to the supply of material in the print head being disrupted and the manufacturing process having to be interrupted. The subsequent examination of the test specimens revealed that compound formulations with extrusion properties suitable for additive manufacturing using material extrusion have high volume resistances or low conductivities and therefore are unsuitable for applications that require high electric conductivity.

Outlook

The presented work shows the fundamental possibility of additively processing highly filled carbon polymer compounds on material extrusion systems. In addition to the established manufacturing processes such as milling, hot pressing or injection molding, additive manufacturing with its process related advantages now offers an additional alternative for using carbon polymer compounds in existing applications, for example to produce prototypes, spare parts and small series, without fixed costs, more cost-effectively and without the time-consuming production of tools to process faster. In addition, the development of new applications, e.g. in the field of electronics or mechanical engineering, is made possible, based on the specifically adjustable mechanical, electric, thermal and chemical properties of carbon polymer compounds in combination with the advantages of additive manufacturing in terms of design freedom, costs and production time.

However, the presented work also shows limitations in the additive manufacturing of carbon polymer compounds using material extrusion. The risk of process errors increases significantly as the filler share increases. This risk can be countered by measures such as expanding the nozzle diameter. Though, this affects the imaging accuracy and thus the quality of the product to be manufactured. A filler share rate above 80% seems to be difficult to achieve compared to conventional manufacturing processes. Compared to conventionally manufactured specimen, additively manufactured specimen made from carbon polymer compounds had just slightly poorer electric conductivity values.

The extent to which this also applies to the mechanical, thermal or chemical properties and can be remedied through changes in the design and manufacturing process or suitable measures in post processing was not investigated in this work. This needs to be examined in future investigations in order to enable the use of carbon polymer compounds in additive manufacturing for other areas of application.

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