3D computed tomography as Quality Control Tool in Advanced Composite Manufacturing

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Abstract
3D computed tomography (CT) is used as a quality control tool in composite manufacturing. The analyzed materials include natural fiber reinforced polymers (thermoplastics and thermosets), porous carbons, and silicon carbide (SiC) ceramics. Multi-step manufacturing processes were used for the production of the composite materials. One manufacturing route includes the production of natural fiber reinforced composite (NFC) parts by extrusion of thermoplastic NFC filaments followed by 3D printing (fused layer modelling - FLM). In the other route, extrusion of wood polymer composites (WPC) is followed by a carbonization process for production of porous carbon templates, which are then converted to SiC ceramics by a liquid silicon infiltration process. 3D CT was used for the analysis of the intermediate as well as the final products. The results showed that 3D CT is a suitable tool for analysis of samples. Of particular interest is that defects like air inclusions and fiber agglomerates, which significantly diminish the quality of the final products, namely the 3D printed composites and the bio-based SiC ceramics, can already be found in the intermediate products, the extruded NFC filaments and the extruded WPC profiles, respectively. Therefore, 3D CT has the potential to be used as quality control tool during both manufacturing processes used in this study.

Keywords: 3D printing, computed tomography, extrusion, fused filament fabrication, porous carbon, SiC ceramic, wood polymer composite, natural fiber composites

1 Introduction
Bio-based composite materials, natural fiber reinforced composites (NFC), and wood polymer composites (WPC) have some beneficial properties compared to the conventional composite materials, which usually are fossil-based matrix polymers with inorganic fillers. Besides cost saving aspects and expected ecological benefits, the low density of bio-based fillers is the main advantage, enabling lighter weight structures compared to conventional composite materials. In addition, NFC and WPC can be processed similar to conventional composite materials, but offer advantages with regard to equipment wear. [1]

Besides injection molding and extrusion, 3D printing represents a well-known and fast growing material processing technology. A printed part is formed by adding material layer by layer until the part is finished without requiring any additional tools. Therefore, using 3D printing can save time and costs in comparison to commercial production methods, particularly when manufacturing complex prototypes or small batch series. Another advantage of 3D printing is its freedom of design. 3D printing of NFC could offer further fields of applications. [2]

The quality and performance of 3D printed NFCs is defined by the morphology and density of the polymer composite. The main influencing factors can be classified in four categories: First, the properties, quality and performance of matrix itself, second the quality and performance of the added natural fiber, third the polymer matrix bonding quality and fourth, when talking about 3D printing, the quality and performance of the used additive manufacturing technology. [3]

A big issue is to analyze the mentioned properties and the quality of the NFC parts. To the best of the author’s knowledge, no standard procedure for characterization of combinations of the materials and manufacturing processes is available.

In a previous study WPC-based materials have been investigated using 3D CT scans [4]. The process included extrusion of WPC, followed by a carbonization process for production of porous carbon templates, which were then converted to SiC ceramics by a liquid silicon infiltration process. 3D CT was used for analysis of phase composition of different samples, intermediates as well as in the final state of the production process. CT slices of the individual production steps of C/Si/3C-ceramics are shown in Figure 1.
Figure 1: Detailed view of CT slices of a WPC (left) and a C-template type 1 (middle) after carbonization of the same sample. The different sizes of the images represent the shrinkage during the carbonization process. The C/Si/SiC-ceramic (right) obtained by liquid-Si-infiltration of a C-template mainly shows the denser Si/SiC and the less dense C/air phases. Voxel Size 1.f.t.r: (2.5 µm)³ / (2 µm)³ / (3 µm)³.[4,5]

In the WPC (left), the chipped wood particles and pores could be distinguished from the polymer matrix. Additionally, inclusions with higher density can be recognized. After carbonization, the polymer matrix disappears in the C-template (middle) and only pores and the denser inclusions remain. In the final C/Si/SiC-ceramic (right) four different phases are visible, but the differences in grey values between air / C and Si / SiC are quite low. [4, 5]

In this application CT scans were successfully used for the qualitative non-destructive evaluation of different manufacturing steps of WPC-based ceramics. It could be shown that there are some phase structures of higher density in the WPC, which cannot be eliminated by the carbonization and Si-infiltration steps. 3D CT was shown to work in general as characterization method for WPC. Furthermore, it was shown that changes during the various production steps can be visualized using 3D CT scans of intermediate and final products. Therefore, the use of 3D CT as a quality control tool in fused layer modelling (FLM) of WPC and NFC materials seemed to be promising and was evaluated in this study.

2 Materials and Methods

2.1 Materials for NFC-Compound

For the production of the NFC compound, two different materials were used. As for the polymer matrix, pre-dried polylactic acid (PLA) type Ingeo 3251 D from NatureWorks LLC, a low viscosity injection molding grade with MFR (210 °C/ 2.16 kg) = 80 g 10 min⁻¹, was used. For PLA reinforcement wood flour with trade name ArboCell C100 supplied by Rettenmaier&Söhne GmbH&CoKG was used.

2.2 Methods:

2.2.1 Preparation of NFC Compound

Prior to compounding, PLA and wood flour were dried at 60 °C for 12 h. After drying, NFC compounds consisting of 85 wt. % PLA and 15 wt. % wood flour were prepared using a lab scale co-rotating compounder TSE Brabender with screw diameters of 20 mm and L/D of 40. An Econ underwater pelletizing system (EUP 50) was used for pelletizing. After the compounding process, the prepared NFC compound was dried again at 60 °C for 12 h before further processing.

2.2.2 Filament production

The produced compound (in shape of granules) was further processed and transformed into a continuous plastic wire (filament). For this, a counter-rotating conical twin screw extruder equipped with a band conveyer for calibration, a haul-off unit for speed control and a winder was used. The diameter of the filament was set to 1.75 mm and controlled by a two laser measuring system (Beta LaserMike). In order to minimize the fluctuation of the filament diameter, various parameters like the diameter of the profile die, processing temperatures, the take-off speed, the humidity of the compound and the crystallinity of PLA have been changed and optimized.

2.2.3 FLM 3D printing

For the production of parts, the 3D printing technology FLM was used. The temperature of the heated print head equipped with a 0.6 mm die was 205 °C. During the printing process the travel speed of the print head was set to 30 mm s⁻¹. The solid
filament was continuously pushed through the printing head and the melt was deposited to the printing bed at a layer height of 0.3 mm. The printing bed was heated to 60 °C to improve the bonding of the printed object on the printing bed.

2.2.4 CT machine and measurement parameters
3D CT investigations were performed with a Nanotom 180 NF (GE phoenix|x-ray, Wunstorf, Germany) device. The system is equipped with a 180-kV nano-focus tube and a 2,304 × 2,304 Hamamatsu detector. Molybdenum was used as target material. Different types of samples were analyzed, shown in Figure 1. Sample size, voxel size and other parameters were optimized for each type of sample. For image reconstruction, a filtered back-projection algorithm was applied by using the Nanotom reconstruction software datos|x (GE phoenix|x-ray). Qualitative and quantitative data evaluation was mainly done in VG Studio Max 2.2 and 3.0 (Volume Graphics, Heidelberg, Germany). An overview of applied measurement parameters is given in Table 1.

<table>
<thead>
<tr>
<th>Sample / Material</th>
<th>VS [µm]</th>
<th>U [kV]</th>
<th>I [µA]</th>
<th>T int [ms]</th>
<th>T meas [min]</th>
<th>n</th>
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<tbody>
<tr>
<td>Figure 2: CT scans of three different filaments</td>
<td>2.5</td>
<td>60</td>
<td>150</td>
<td>500</td>
<td>66</td>
<td>1500</td>
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<tr>
<td>Figure 3: CT scan of an NFC filament</td>
<td>6.5</td>
<td>50</td>
<td>260</td>
<td>500</td>
<td>98</td>
<td>1900</td>
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<td>Figure 4: CT scan of two different NFC filaments</td>
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<td>50</td>
<td>200</td>
<td>900</td>
<td>120</td>
<td>1000</td>
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<td>Figure 5: CT scans of building parts out of NFC filaments</td>
<td>7</td>
<td>60</td>
<td>240</td>
<td>500</td>
<td>103</td>
<td>1700</td>
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<tr>
<td>Figure 6: CT scans of building parts produced via FLM and IM</td>
<td>8.5</td>
<td>60</td>
<td>250</td>
<td>700</td>
<td>120</td>
<td>1700</td>
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3 Results and Discussion
First, three filaments consisting of different material combinations were scanned and analyzed as shown in Figure 2. All three tested filaments were colored black and therefore could not be differentiated from each other by human eye. The filaments were screened and classified due to their density.

Filament A consists of PLA mixed with wood flour and wood fibers. Filament B was synthesized out of PLA mixed with low amount of glass fibers and talcum powder. Filament C is made out of PLA mixed with 30 wt. % glass fibers.

For better visualization of specific components of the filaments, parts having a certain density can be made invisible in the scans. In Figure 2-(3) all parts with the density of pure PLA are made invisible. Therefore, the only remaining parts are the fillers and fibers. Besides the quantitative and qualitative analysis of the added fillers and fibers, the orientation of the fibers within the filament can be analyzed. Fibers are mostly aligned parallel to the filament axes. This is due to the flow behavior of the polymer melt through the shaping die in filament production.

The performance of printed NFCs is strongly influenced by the quality of the processed filament. Geometry of the filament, inhomogeneities and voids may be transferred into the constructed NFC part, thus resulting in bad part properties and performance. CT scans were used to analyze filaments in detail and screen their morphology. Figure 3 shows a CT scan of an NFC filament.
Figure 3: CT scan of NFC filament. The picture shows the NFC filament from top view (left) and from side view (right). The black areas represent voids

As can be seen in Figure 3 the filament fulfills specific geometry requirements for FLM, being round shaped with a diameter of 1.75 mm. The diameter shows very little fluctuation, which is fundamental to achieve a stable 3D printing process. Besides the geometry the homogeneity can be observed. In the scanned filament it can be clearly differentiated between polymer matrix, fiber and fillers and voids. The dark grey area represents the polymer matrix, while the bright spots and areas represent fibers and wood flour. The wood particles are well distributed and hardly any particle agglomerations can be found. The images show black spots within the filament, which can be characterized as large voids. They vary in number and size over the cross-section. Beginning from the center outwards the voids are getting smaller and the number of spots is also decreasing. Near the outer surface only little voids can be found.

Voids can act as stress concentration centers, introduce cracks and should not be part of the cross-section at all [6]. Via changing the tool die in the filament production line, voids should be avoided in the filament. Therefore, an extrusion die of different shape was installed. Using CT scans the influence of changing process parameters on the filament quality can be characterized. For comparison, two different filaments were scanned together. Figure 4 shows a comparison of two filaments, produced at different process parameters.

Figure 4: CT scan of two different NFC filaments

The change of the tool die had a big impact on the filament quality. While the matrix-fiber homogenization was not influenced, a big change concerning the voids was detected. The total number of voids decreased, while the size of the voids increased.
The biggest voids can be found in the middle of the filament cross-section. Beginning from the center the size of the voids is reduced and finds its minimum size near the outer surface.

Using CT scans the quality of different filaments can be analyzed in detail. In this way it is easy to show the influence of process parameter changes on the filament quality. CT scans can be used to accelerate optimization methods and help to find the best parameters for qualitative filament production. Concerning the filaments shown in Figure 4, the change in process parameter did influence the morphology but did not increase the quality of the filaments. It is necessary to modify the process in an alternative way to get rid of the voids in the cross-section or at least to reduce the number of voids.

Based on these results, the question arose, how the morphology of the filament influences the 3D printing process and the printed part. During 3D printing the filament is molten under pressure in the printing head and deposited layer by layer on the printing bed. Although the filament is completely molten, the quality of the filament has a big impact on the 3D printed part. Figure 5 shows a CT scan of a printed building part, processed from the scanned filaments in Figure 4.

![Figure 5: CT scans of building parts produced via material extrusion of porous filaments](image)

The CT scans clearly show a high number of defects in the building part cross section. Besides the polymer matrix and the fibers, a high number of voids and defects can be found. This means, the voids in the filament are transmitted to the building part. As mentioned before, these voids have a strong influence on the properties of the building part. They act as stress concentrators and lead to bad crack resistance. The expected performance of the building part is reduced the more voids existing in the cross section of the building part. CT scans clearly show that any defects and voids in the filament will be transmitted to the 3D printed part, thus the filament quality influences the quality and performance of the printed part.

Further CT scans were done to compare 3D printed parts and injection molded parts. Figure 6 shows CT scans of a printed and an injection molded test specimen. The 3D printed part was made out of the filament shown in Figure 3. For production of this filament and injection molding of the test specimen, the same material compound was used.
In this scan the used processing technologies (FLM and injection molding) can be compared to each other. Although the material source is the same, big differences in the cross section of the parts can be seen. The injection molded part shows high density while the 3D printed part is full of defects and voids. This is due to differences in the process technologies and leads to differences in the mechanical properties of the produced test specimen. The injection molded parts achieve higher mechanical properties in tensile tests and also higher fracture toughness than the 3D printed parts. Besides the porous structure, the geometry of the 3D printed part is rectangular, while the injection molded part shows a furrow in the middle. This is likely due to the material shrinkage and thermal stress in the cooling process during injection molding of the highly densified polymeric material compound. [7, 8, 9]

4. Conclusion and Outlook
CT scans can be used to analyze various properties of NFC filaments and 3D printed parts thereof.

The morphology of NFC materials concerning fiber orientation can be characterized. This is of big importance concerning NFC, as the fiber orientation dominates the mechanical and thermal part properties. By virtually removing parts of specific density (using the CT software), like the polymer matrix itself, the fiber orientation and distribution in the matrix can be obtained. Based on this knowledge the mechanical and thermal properties of the NFC might at least be predicted or estimated. Furthermore, CT scans can be used to get quantitative information about the NFC composition. E.g. it can be used to calculate the volume content of the fibers in the matrix, when the composition is unknown and information are not available from the manufacturer of the compound or building part.

Using CT scans the filaments can be analyzed in detail. CT scans help to detect inhomogeneities and voids in the filaments. Comparison of scans of the filament and the printed part showed that voids present in the filament are transferred to the printed part despite melting of the filament during FLM processing. Therefore, optimization of the filament production process is the most important step in order to achieve high quality printed parts.

CT scans can be used to accelerate process optimization. By varying the process parameters in the filament production line and comparing the CT scans of the differently produced filaments, the time for evaluating the best setup of process parameters is substantially reduced. Correlations between changes in process parameters and changes in the morphological structure of the filament can be determined.

Several filaments or parts can be scanned at the same time. The scanned parts can be analyzed and compared to each other directly in one scan. Therefore, it is possible to get an overview of the morphology of the scanned objects. In industry it is possible to compare different commercial filaments and find out which one will fit best to the desired application.

Besides the comparison of filaments to each other, it is also possible to compare parts manufactured by different technologies. Differences in mechanical or thermal properties of components can be linked to differences in morphology due to the characteristics of the used manufacturing technology.

CT scan helps to get qualitative and quantitative information of NFC filaments and printed parts thereof. Therefore, it represents a great quality control tool in advanced composite manufacturing, which can help to accelerate optimization processes.

Acknowledgements
The work was supported by the European Regional Development Fund (EFRE) and the province of Upper Austria through the programs IWB 2014-2020 – Upper Austria (project BioCarb-K) and INTERREG V-A Austria – Germany/Bavaria 2014-2020 (project TFP Hy-Mat AB97) and INTERREG Austria - Czech Republic 2014-2020 (project COM 3D-XCT)
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