

Layerwise fiber volume fraction characterization in composite structures: Method comparison and application to composite pressure vessels

Gerrit Rehs^a, Oliver Dammann^a, Daniela Feldten^b, Nils Meyer^b, Tobias Dickhut^{a,*}

^a Chair of Composite Materials and Engineering Mechanics, University of the Bundeswehr Munich, Neubiberg, 85577, Germany

^b MeFeX GmbH, Egerländer Str. 6, Weiterstadt, 64331, Germany

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ABSTRACT

The Fiber Volume Fraction (FVF) is a key parameter within the mechanical characterization of Carbon Fiber Reinforced Polymers (CFRP) at material level. An accurate methodology for the local determination of the layerwise FVF is required for a reliable and accurate analysis. In this study, a Fiber Volume Gradient Characterization Methodology is developed based on the Fiber Counting Algorithm for the layerwise determination of the FVF for angles up to 45°. For this, acid digestion, loss on ignition and the optical method are compared. While all investigated methods show high consistency, it should be noted that the optical FVF determination allows for localized FVF assessments while being subject to operator dependent image acquisition. Based on this comparison, an automated methodology is validated using optical micrographs. The developed methodology allows for a fast and accurate characterization of the FVF in layered structures and can therefore be applied to various structures in CFRP engineering. As an application example, the layerwise FVF of a Composite Pressure Vessel (CPV) is analyzed by the optical assessment and compared against the loss on ignition method, resulting in deviations below 1.05%.

1. Introduction

Carbon Fiber Reinforced Polymers (CFRPs) are widely applied among different applications like automotive or aerospace because their performance can be tailored by fiber and matrix architecture, layer stacking and fiber orientation, among others [1]. Within the constituent parameters, the fiber volume fraction (FVF) is one critical characterization parameter for CFRPs, to determine material properties such as strength and stiffness using micro-scale equations or the rule of mixtures [1]. A precise knowledge of the local FVF in CFRP parts is therefore of high importance for commercial introduction and performance analysis of CFRP composite at material level which will later be used for an accurate analysis model that leads to a reliable design of the part.

This aspect is particularly relevant for layered filament-wound structures, such as composite pressure vessels (CPVs) which are employed in different Types (Type III-V) in automotive, aviation or space applications [2,3]. During filament winding, the mechanical performance of CPVs is influenced by several manufacturing process parameters, such as winding tension, stacking sequence and winding pattern, compaction, tow-placing quality and curing conditions [4–7]. Consequently, the FVF can vary depending on these process parameters through the laminate, as observed on CPV level by Nebe [8]

and Christ [9]. Since CPV performance is linked to the FVF, precise knowledge of the FVF per layer is relevant to an efficient mechanical design and possible design optimizations [10].

Several methods can be applied to determine the FVF of CFRP structures, each with specific advantages and limitations. One widely established approach is to dissolve the matrix using acid digestion. This process is described in several standards like DIN EN 2564 [11], ISO 14127 [12] and ASTM D3171 [13]. The matrix is completely dissolved and the remaining fiber mass is used to calculate the FVF using the density of the specimen before matrix removal. Another established destructive method for FVF determination is loss on ignition (LOI). The LOI removes the matrix material by thermal decomposition in a furnace, as described in ASTM D3171 [13]. Depending on process parameters such as the applied temperature and dwell time, the matrix and a substantial fraction of the organic fiber sizing are thermally removed [14,15]. At elevated temperatures, the fibers undergo thermal degradation. Therefore, careful selection and control of the process parameters is required. Bleier [16] proposes a process of 20 min at 560 °C, under which the fiber experiences a mass loss due to the high temperature. Accordingly, a correction based on the measured fiber mass loss is recommended. Meyer [17] suggests a reduced temperature of 470 °C with an extended dwell time of 80 min. Owing to the lower

* Corresponding author.

E-mail address: tobias.dickhut@unibw.de (T. Dickhut).

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temperature, the fiber is not as affected by thermal degradation which represents an advantage of this process. The FVF is calculated based on the remaining fiber mass and the resin and fiber densities. Both methods, acid-digestion and LOI are destructive methods. They provide bulk FVF results, while no local characterization of the FVF is possible.

For localized FVF measurements, optical assessment based on polished CFRP cross-sections can be applied. Early applications of quality inspection using optical assessment were reported by Purslow [18]. For optical FVF characterization, two evaluation approaches are commonly used. The first relies on pixel-based area fraction analysis. Based on a threshold, fiber and matrix material is separated into light and dark pixels and the areas are quantified for the determination of the FVF. However, this approach needs to be used with caution whenever high accuracy is required, as the results depend on the threshold and spatial resolution of the microscopes, as reported by Cann [19] and Bleier [16]. The second approach is based on fiber counting algorithms. Fibers within a defined area are counted and the FVF is calculated based on the fiber count using the cross section of the fibers. A key challenge for optical assessments is the preparation of cross-section micrographs with sufficient surface quality and minimal artifacts, as such artifacts compromise the evaluation [16,19]. Additionally, careful treatment is required for fibers intersecting with the boundary of the area of interest (AOI) [16,19]. Optical assessment procedures are described in standards such as ISO 14127 [12]. Donhauser [20] proposed an optical analysis methodology based on neural networks, achieving a relative error of 3.3 % with respect to the manufacturer-provided information.

Alternative approaches for the FVF characterization include thermogravimetric analysis (TGA), reducing hazards and environmental impact compared to acid digestion [21,22]. Within this analysis technique, small samples can be analyzed automatically, showing low deviations of less than 1% to LOI or acid digestion [21]. Similar to LOI, the fibers may undergo thermal degradation [21]. Improved TGA procedures are proposed by Bücheler et al. using temperatures of 430 °C [23] or Wang et al. [24] applying a novel carbonization-in-nitrogen procedure. μ CT represents a nondestructive method, eliminating the need for traditional destructive cross-sections for local measurements. Dilonardo et al. [25] compared FVF characterization by μ CT with acid digestion and concluded that acid digestion characterization is less accurate compared to the μ CT method because of the need of the precise knowledge of the component volume. While three-dimensional information on the CFRP structure can be obtained, limitations arise from the need for sufficient contrast for segmentation and an appropriate balance between sample size and resolution [26–28]. Additionally, μ CT measurements are generally more cost-intensive, limiting the availability of this method. A comprehensive review of the mentioned and additional methods is presented by Yao et al. [29].

Several studies have applied different methods for FVF characterization to CFRP structures, highlighting their applicability and limitations. Cann et al. [19] compared the microscopy-based optical areal method and fiber counting method with the acid digestion method to characterize FVF gradients in CFRP plates. The fiber counting method showed mean differences below 1 %, while being less sensitive to image imperfections compared to the areal method. Christ [9] investigated the layerwise FVF of a CPV applying an area-based optical approach. A threshold for the optical assessment was selected and subsequently the resulting values were calibrated by the mean value derived from TGA. Analysis of a CPV laminate indicated a decrease in FVF towards the outer layers of the laminate, consistent with observations reported by Cohen et al. [10,30]. Nebe [8] analyzed the laminate FVF in CPVs using acid digestion. The study evaluated both mean values as well as values aggregated for layers with the same winding orientation. Although this approach does not provide layer resolved FVF determination, Nebe [8] demonstrated that the stacking sequence influences both FVF and porosity. High compaction forces through circumferential wound layers increased the FVF in underlying layers, whereas helical layers exhibited only limited compaction. Mertiny et al. showed that increased fiber

tension during the winding process can increase the FVF per layer, whereas the resin can be accumulated at the outer surface, resulting in similar mean FVF values [7].

Based on the above mentioned literature review, several established methods, such as acid digestion and loss on ignition, provide bulk FVF values, however they do not resolve local through-thickness FVF variations within layered filament-wound structures. Previous optical approaches have demonstrated the potential for local FVF assessment, but their application to angled plies in towpreg-wound CFRP structures and CPV laminates remains limited, especially based on real micrographs and not employing artificial micrographs. In particular, a systematic comparison of destructive bulk methods and optical fiber counting for layer-resolved FVF characterization in filament-wound reference specimens is still lacking, especially applied to different materials within one study, necessary to assess the optical accuracy of FVF measurements depending on resin and fiber material.

In this context, the present work aims to develop and assess an optical methodology for characterizing through-thickness FVF gradients in angled plies up to 45°, using towpreg-wound CFRP tubes as reference specimens. Finally, the workflow is applied to a filament-wound CPV laminate, and the resulting layerwise FVF distribution is discussed. To support the interpretation of the optical results, a comprehensive comparison with LOI and acid digestion is performed for three different towpreg material systems.

2. Material and methods

To establish a reliable methodology for the layerwise FVF determination in composite structures, the first step is to characterize the experimental methods available for measuring the FVF. Therefore, acid digestion, loss on ignition and optical analysis of micrographs are applied to CFRP specimens manufactured by towpreg winding.

2.1. Material and sample manufacturing

One key challenge for developing a methodology for the characterization of the layerwise FVF of a composite is to identify an appropriate CFRP sample. The most important requirement for the sample is a constant FVF, independent of the location of the structure, as more samples need to be cut from the structure to manufacture different samples which can be subsequently analyzed by the different methods for the FVF characterization.

Initial samples were extracted from an unidirectional (UD) CFRP plate. The manufacturing was performed by prepreg hand layup as well as towpreg robotic winding. The UD plates were cured in an autoclave using a vacuum bag process. For a uniform thickness of the CFRP plates, aluminum caul plates were applied during curing to reduce geometrical scatter within the samples. However, the extracted CFRP samples were found unsuitable for the intended method comparison within this study, as the FVF was not sufficiently homogeneous within the plate. The center of the plates showed lower FVF compared to the edges of the plate by about 3 % even though a trimming edge was foreseen.

Since the objective of this study is a method comparison of different FVF characterization for the through-thickness FVF characterization per layer of a CPV, the approach of using CFRP samples manufactured by plates was discarded. Instead, towpreg-wound tubes were selected as reference specimens for this study. This was done because the towpreg-wound specimens showed low FVF deviations and because their manufacturing process was similar to CPV manufacturing. The tubes were manufactured by a robotic winding machine shown in Fig. 1(a). In total, six different tubes were manufactured. Three materials were used to manufacture tubes with a layup of $\pm 89.6^\circ$ layer orientation. For simplification, this winding angle will be denoted with $\pm 90^\circ$ from this point on. For one material, additional tubes with layups of $\pm 75^\circ$, $\pm 60^\circ$ and $\pm 45^\circ$ were manufactured for analyzing different layer angles, which is necessary for the intended CPV FVF analysis.

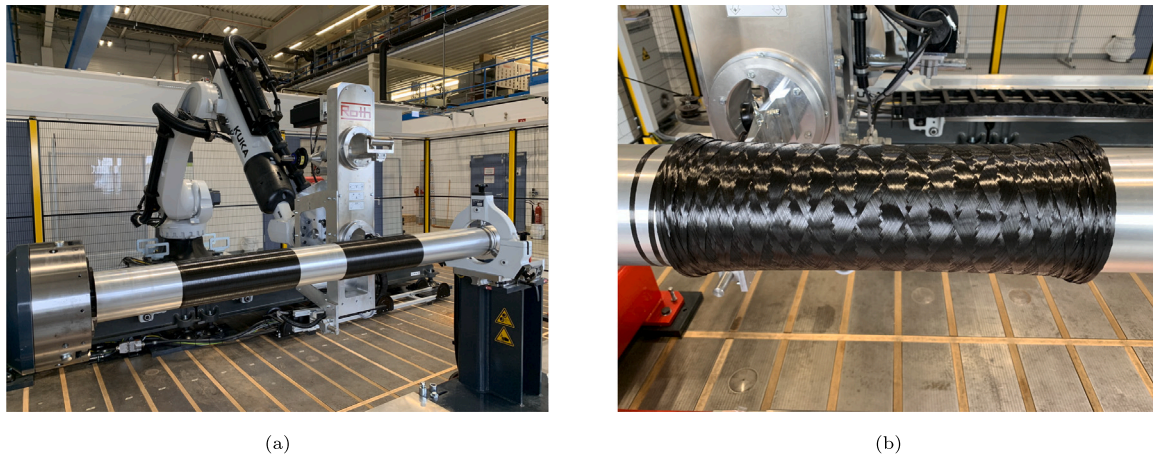


Fig. 1. Towpreg winding processes for different tube configurations and winding angles: (a) Towpreg winding of the ITS50 $\pm 90^\circ$ tube (left) and the IMS65 $\pm 90^\circ$ tube (right) on the winding tooling inside the robotic winding machine, (b) Towpreg winding of the ITS50 $\pm 75^\circ$ tube with transition from hoop layers to the target fiber angle using friction-based stirring.

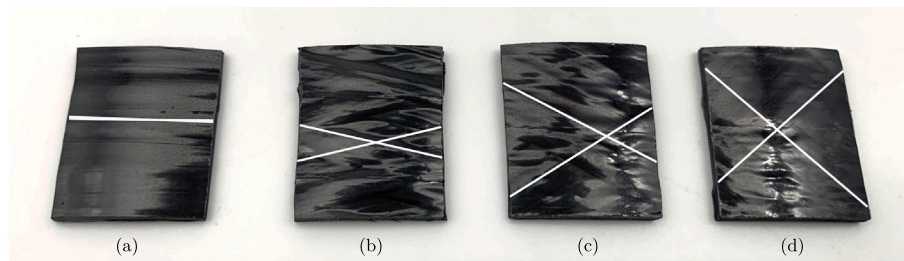


Fig. 2. Representative samples used for the FVF characterization with indicated winding angles. The winding angle is defined with respect to the cut edge at the left or right boundary of the specimen. The indicated angles are always defined with respect to a winding ply $\pm \alpha$ and therefore correspond to twice the ply angle. The specimens were cut to approximate dimensions of $25 \text{ mm} \times 25 \text{ mm} \times 2 \text{ mm}$, resulting in a mass of $m \approx 2 \text{ g}$. The increased surface waviness observed in (b)–(d) is caused by intersecting tows during the winding process. Resin-rich regions are present on the outer surface of all samples, resulting in a glossy surface appearance.

By considering winding angles up to 45° , all possible ply angles can be covered by preparing two cross-sections that are rotated by 90° relative to each other during the optical evaluation. If only a single cross-section is evaluated, fibers oriented at angles close to 90° relative to the section normal appear as highly elliptical cross-sections, which is not desirable. Fig. 1(b) shows the winding process of a $\pm 75^\circ$ tube. For all tubes deviating from a $\pm 90^\circ$ winding angle, the samples were cut from the center section of the tube where a constant winding angle is present.

All tubes were manufactured on an aluminum tool with an inner diameter of $D = 135 \text{ mm}$ and an approximate wall thickness of $t \approx 2 \text{ mm}$ each. After winding, the tubes were cured in an autoclave using a vacuum bag. For smooth surfaces, shrink tape was applied on the upper CFRP layer before vacuum bagging and curing. Caused by increased temperatures in the autoclave, the tape shrinks and adds compaction to the CFRP. During curing, the resin is trapped underneath this tape, which can result in resin-rich areas at the surface of the CFRP tube.

After curing, the tubes were cut into rings of $l \approx 25 \text{ mm}$ length on a Steinadler A600 diamond cutting machine. Consequently, these rings were cut into 15 individual samples. For the optical specimens, this cut was carried out using an automated diamond cutting QATM Brillant 265 machine. The remaining cuts were done by hand using a rotary power tool. Fig. 2 shows the specimens for the ITS50 material. These samples formed the basis for the comparison of the different FVF characterization methods. For each ring, five different methods of FVF characterization were applied within this study. For each method, three samples were used at an angular distance of 120° . In total, 90 samples were manufactured and analyzed.

Table 1

Overview of the used fiber and resin systems with corresponding fiber diameters.

Fiber	Filament diameter	Resin system	Resin density
T700SC	7 μm	K-Preg 002-002-70-25	1.2 g/cm^3
IMS65	5 μm	LME 11725/LY 3512	1.13 g/cm^3
ITS50	7 μm	LME 11725/LY 3512	1.13 g/cm^3

Table 1 provides an overview on the used material systems. For subsequent analyses, the fiber and resin densities are taken from the corresponding datasheets [31–34]. For the proposed workflow in this study, the fiber diameters provided in the material datasheets were assumed to be representative input values, while acknowledging that the actual fiber diameters are subject to a certain statistical distribution. Similarly, the resin density was assumed to be accurate for the FVF calculation. These assumptions are considered later in the discussion of the results.

2.2. Processes of fiber volume fraction characterization using different methods

Three different methods were applied to analyze the FVF of the samples. The methods are acid digestion, loss of ignition and optical assessment using optical micrographs.

2.2.1. Acid digestion

For determining the FVF using acid digestion, the density was measured using a Sartorius MSA225S-100-DI precision balance by

Archimedes' principle. Subsequently, the samples were put into boiling flasks. 75 ml sulfuric acid (96 %) was added and the boiling flask was mounted on a magnetic stirrer. The sample in sulfuric acid was heated to boiling and remained after boiling for more than one hour on the magnetic stirrer. A dimroth cooler was used during this process. Hydrogen peroxide H_2O_2 was used to bind the dissolved polymer. This was done until no reaction was visible anymore. Subsequently, the boiling flask was cooled down. The fibers were washed with water until a pH value of 7 was reached. After this, the fibers were cleaned with distilled water and subsequently with acetone (>99.8 %). Then, the fibers were dried in a furnace at $T = 120^\circ\text{C}$ for more than one hour. Subsequently, the fibers were cooled down in a desiccator for 20 min. The mass of the sample before and after this process was measured using the precision balance.

The FVF $\varphi_{\text{Laminate}}$ can be calculated according to DIN EN 2564 [11] or ISO 14127 [12] by:

$$\varphi_{\text{Laminate}} = \psi \cdot \frac{\rho_{\text{Laminate}}}{\rho_{\text{Fiber}}} \quad (1)$$

using the fiber mass fraction:

$$\psi = \frac{m_{\text{Fiber}}}{m_{\text{Laminate}}} \quad (2)$$

This workflow is executed based on standard [11].

2.2.2. Loss on ignition

For determining the FVF using loss on ignition, the weight of the sample was measured using the precision balance as a first step. The samples were then placed into a fire-resistant crucible.

For burning the polymer, two different muffle furnaces were used. The first furnace is operated at the *University of the Bundeswehr München*. A temperature measurement was performed to determine the ideal position of the crucible based on the temperature distribution within the furnace. In order to be able to insert the thermocouples, the supply air vent was opened and the thermocouples were introduced into the furnace. Each measurement point is marked by an $\times$ in Fig. 3, the location of the crucible is indicated by a gray circle. The coordinate center is located at $(x, y) = (0\text{ mm}, 0\text{ mm})$. The temperature towards the walls of the furnace is calculated based on a linear extrapolation. The temperature profile for a desired temperature of 400°C is shown. For the *Nabertherm L9/11/SKM* muffle furnace the ideal conditions are identified in the back corners left and right. The locations are marked in Fig. 3(c) by a cross. The influence of the setting of the supply air vent is discussed in the results section. A similar measurement was performed for the *Heraeus KR 260 E* furnace, which is operated at the *MeFeX GmbH*. The temperature profile is shown in Fig. 3(d). Again, the optimal position for the fire-resistant crucibles is indicated by a cross.

For the burning process, two different methods were applied.

- $T = 560^\circ\text{C}$ for $t = 20\text{ min}$ as suggested by Bleier [16]
- $T = 470^\circ\text{C}$ for $t = 80\text{ min}$ as suggested by Meyer [17]

After burning, the samples were cooled down in a desiccator for 20 min. Right after, the mass of the remaining fibers was measured using a precision balance. LOI methods are standardized in DIN EN ISO 1172 [35] for glass fiber reinforced polymers (GFRP) and in ASTM D2584 [36] for GFRP and CFRP.

The FVF can be calculated as proposed in [1]:

$$\varphi_{\text{Laminate}} = \frac{1}{1 + \frac{1-\psi}{\psi} \cdot \frac{\rho_{\text{Fiber}}}{\rho_{\text{Matrix}}}} \quad (3)$$

using Eq. (2) for the fiber mass fraction. Another approach would be the application of Eq. (1).

For the evaluation of the FVF using the LOI process, the thermal degradation of the fibers should be considered. To assess the influence of this effect, pure fibers are burned using both process temperatures, 470°C and 560°C . By this, the linear fiber degradation rate can be measured after the loss of the sizing of the fibers. Based on a starting

point during the LOI process, the thermal degradation can be calculated and the FVF result can be corrected [16,17].

Assuming that the fiber mass at the end of the LOI process ($m_{f,1}$) is reduced by a linear fiber mass degradation rate $\dot{m}_f$ starting at t_k until the process end t_1 , the corrected initial fiber mass $m_{f,\text{corrected}}$ can be calculated according to Eq. (4). The sizing is treated as matrix material.

$$m_{f,\text{corrected}} = \frac{m_{f,1}}{1 - \left(1 - \frac{\chi}{100}\right) \cdot \dot{m}_f \cdot (t_1 - t_k)} \quad (4)$$

where χ is the fiber sizing in %.

Based on the corrected fiber mass due to the thermal degradation, the corrected FVF can be calculated using Eqs. (2) and (3). The effect of the thermal degradation on the FVF results is discussed in Section 3.2.

2.2.3. Optical assessment

For determining the FVF using optical assessment by optical micrographs, the samples were embedded in cold curing resin *IP* from *Presi*. The curing was carried out at room temperature, following the instructions of the supplier.

After curing of the resin, the samples were polished before acquiring the microscope images using a digital microscope. The polishing process involved two grinding steps, followed by three polishing steps, performed on a *Presi Mecatech 334* machine. The grinding steps used grinding plates of $75\text{ }\mu\text{m}$ and $30\text{ }\mu\text{m}$ grain size. Subsequently, the polishing was carried out with polishing plates of $9\text{ }\mu\text{m}$, $3\text{ }\mu\text{m}$ and $1\text{ }\mu\text{m}$ grain size. During polishing, lubricant as well as diamond suspension was applied. Special attention needs to be taken during these steps to avoid contamination of the polishing plates with coarser diamonds of the preceding polishing steps. Therefore, all samples were cleaned using an ultrasonic bath after each step.

The optical micrographs were taken using a *Keyence VHX-6000* microscope. Different magnifications can be used. The magnification should be chosen with regard to the possible resolution of the microscope. It must be ensured that each fiber is represented by a sufficient number of pixels to allow reliable evaluation. For this study, a magnification of $500\times$ and $1000\times$ proved to provide reliable datasets for the optical assessment workflow. For image acquisition, 30 micrographs were recorded per sample at $500\times$ magnification, while 45 micrographs were captured at $1000\times$ magnification. The selected number of micrographs was chosen as a compromise between acquiring a sufficient number of images to allow a meaningful evaluation and limiting the overall measurement effort. This was particularly important because the study was intended to provide conclusions that remain applicable to image acquisition without fully automated microscopy. Referring to a cross-sectional area of 4600 mm^2 for one sample, the acquired micrographs represent approximately 1 % of the area at $500\times$ magnification and less than 1 % at $1000\times$ magnification. Automated image acquisition, provided that high-quality polishing results are achieved, offers a straightforward approach to increase the number of evaluated micrographs as required.

The main workflow of the optical assessment was implemented in *Matlab* using the open-source software *MIJ 1.3.9* [37]. This allows fast analysis of multiple pictures and provides reproducible data. Additionally, parameter studies can be performed to gain more insight into the required settings for robust FVF analysis. Similar functionalities are also provided for *Python* [38].

First, the micrograph, shown as an example in Fig. 4(a) is opened using the *ImageJ/Fiji* interface in *Matlab*. Next, the image is converted to an 8-bit-image. The contrast is enhanced using the microscope before the analysis software. If necessary, the image can be cropped to a specific size, allowing for analysis of larger or smaller areas of the micrographs. Consequently, the quality of the picture is enhanced by the "smooth" and "sharpen" algorithm. Following, a close operation followed by an open operation is performed. The close operation fills in little defects within the picture. The open operation removes small

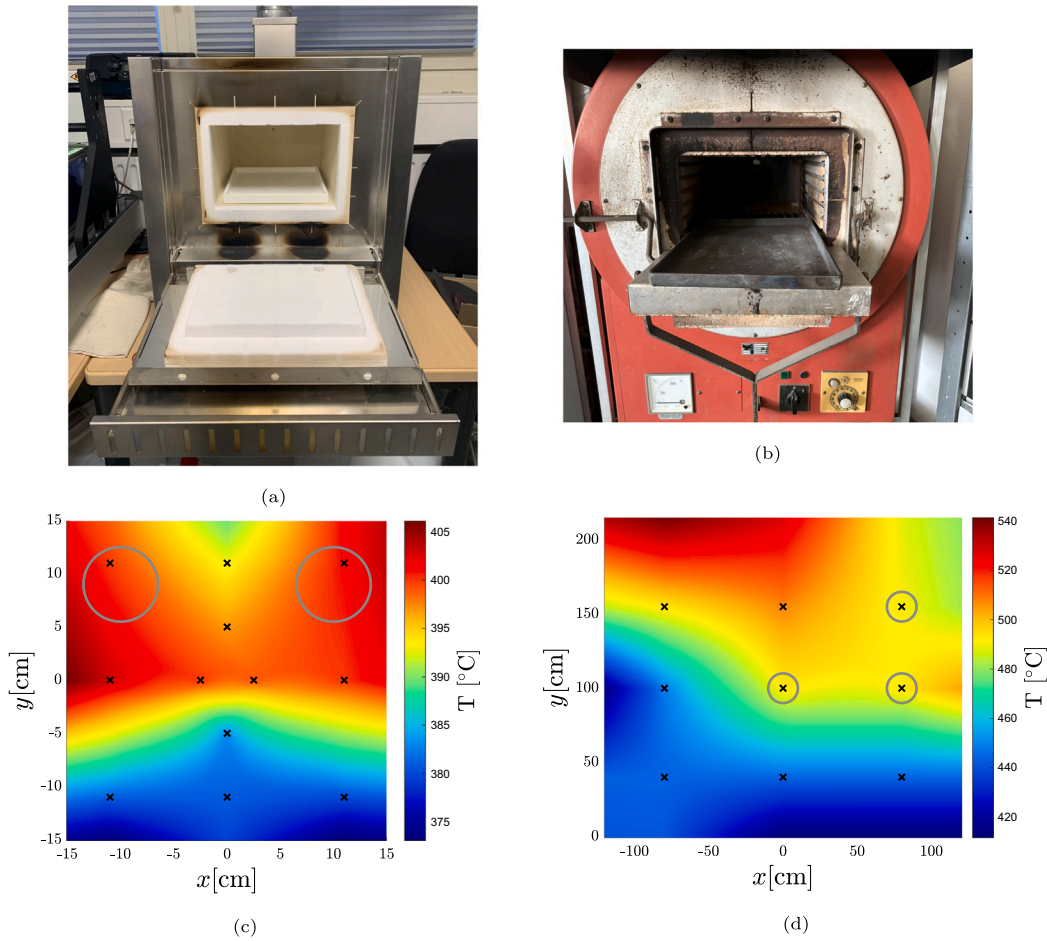


Fig. 3. Comparison of the two furnaces: (a) Nabertherm L9/11/SKM furnace and (b) Heraeus KR 260 E furnace used for LOI measurements. Their individual temperature distributions are shown in (c) for the Nabertherm L9/11/SKM furnace at 400 °C using an open vent setting and in (d) for the Heraeus KR 260 E furnace at 470 °C shown for the upper half ($y > 0$). Crucible position indicated by a gray circle. Temperature was measured 1 cm above the baseplate.

isolated pixels. This workflow prepares for the final evaluation step through the application of the *watershed* algorithm, which separates contiguous areas. The micrograph after this workflow is shown in Fig. 4(b).

Based on a lower threshold $A_{\min}$, individual particles present in the picture are counted automatically as fibers. With this particle count n , the FVF can be calculated using Eq. (5):

$$\varphi_{\text{Laminate}} = \frac{\pi \cdot r_f^2 \cdot K \cdot n}{A \cdot B} \quad (5)$$

where r_f is the fiber radius (see Table 1), K is a factor for accounting for the fiber section angle, n is the fiber count and $A \cdot B$ is the area of the picture.

Based on trigonometric considerations K is defined as:

$$K = \frac{1}{\cos(\alpha)} \quad (6)$$

where α is the angle of the section cut. For 90° fiber orientation and a cut perpendicular to the fiber, the section cut angle is defined as $\alpha = 0$, resulting in round fiber cross-sections.

For the counting process of n a minimum area $A_{\min}$ needs to be defined which is used in the software as a lower threshold for counting a particle as a fiber.

For determination of this minimum area $A_{\min}$, the proposed counting algorithm in *ImageJ* is calibrated using the Circular Hough Transformation implemented in the *imfindcircles* function within *Matlab* for detecting circles. Therefore, 30 pictures of each sample of the 90° materials are analyzed with both workflows. The calibration process was performed with an area of interest (AOI) of 1000 × 1000 px.

A comparison of the two workflows is shown in Fig. 4(b) and (c). For visualization, the AOI is set to only 300 × 300 px. Fig. 4(b) shows the image after the *ImageJ* workflow. Fig. 4(c) shows the results of the *imfindcircles* workflow. The AOI is represented by a yellow rectangle. Detected fibers within the AOI are shown green, fibers intersecting with the AOI boundary are shown red and fibers outside of the AOI are shown in blue. It has to be noted that the *imfindcircles* function is limited to circle detection. The function is not applicable to the samples showing other section cut angles deviating from 0°. This is an advantage of the *ImageJ* workflow, necessary for the CPV analysis. Nevertheless, the *imfindcircles* function allows for a visually inspectable and manually correctable calibration of the *ImageJ* workflow, as the counted fibers can be easily visualized and obvious detection errors can be identified, as shown in Fig. 4(c). This is the reason why this workflow was chosen for the calibration of Eq. (5) based on $A_{\min}$.

For calibrating $A_{\min}$, different effects need to be considered. A methodology needs to be defined for the fibers intersecting with the AOI, which are partially inside and outside of the AOI. Based on an area evaluation of all taken images ($n > 540$), approximately 50 % of the area of the boundary-intersecting fibers lies inside the AOI, consistent with [19]. Accordingly, every second boundary-intersecting fiber detected by *imfindcircles* can be counted as a full fiber and $A_{\min}$ could be defined as $A_{\min} = 1/2 \cdot A_f$. Unfortunately, this neglects the fact that micrographs are not free from imperfections such as scratches or artifacts of the polishing process. This results in detection errors for both workflows. Depending on the polishing quality the *imfindcircles* function sometimes does not detect all fibers, as shown in Fig. 5.

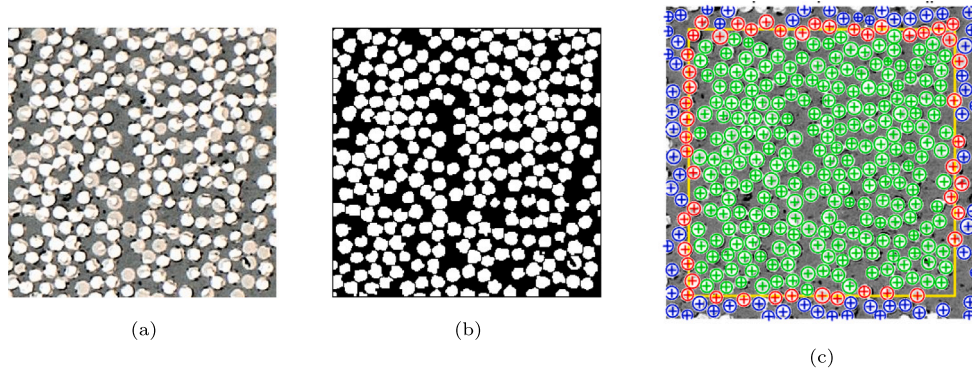


Fig. 4. Visualization of the fiber counting process. (a) shows the raw picture after image acquisition, (b) enhanced picture after fiber segmentation in *ImageJ*, (c) shows the fiber counting process implemented using *imfindcircles* in *Matlab* for the raw picture. The area of interest (AOI) is shown by a yellow box. All fibers which are completely inside the AOI are shown in green, all fibers which are intersecting with the AOI are highlighted red, all fibers outside the AOI are shown blue. For the red fibers, the area inside and outside the AOI can be calculated.

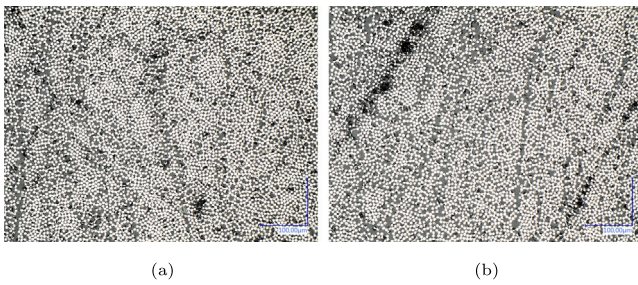


Fig. 5. Visualization of typical quality issues with regard to micrographs. (a) shows a picture with smearing (black areas), which makes the detection of the fibers more difficult. (b) shows the presence of global scratches (black lines), which also lead to a misdetection of fibers.

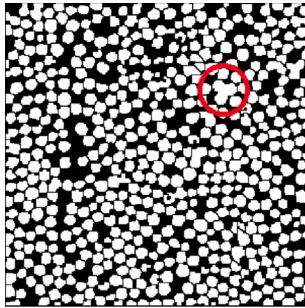


Fig. 6. Misdetection of fiber count because of a failed *watershed* segmentation, resulting in a lower fiber count.

Misdetections can be manually corrected for the calibration process, increasing the detected fiber count.

For the *ImageJ* workflow, there are also limitations present. One source of misdetection of the correct fiber count is the presence of connected fibers. Here, the applied *watershed* algorithm does not separate two fibers into single fibers. This leads to undercounting of the true fiber number, as shown in Fig. 6. The dependence of this error source is discussed with regard to the resolution in the results section. Furthermore, the $A_{\min}$ is based on a pixel detection, which is dependent on the threshold and the resolution. To overcome this drawback, the $A_{\min}$ value is calibrated using the fiber counting workflow using the *imfindcircles* function. For cross sections deviating from 0° , K also needs to be considered. The results and the definition of the $A_{\min}$ value is discussed in the results section.

3. Results and discussion

For the results section, the optical calibration process for the fiber counting algorithm is discussed first. Based on these results, the method comparison is performed. Besides the method comparison, two additional process parameters within the LOI process are reviewed in detail. Finally, the results of the FVF characterization of a CPV layup is presented.

3.1. Effect of image calibration processes

For an accurate implementation of the *ImageJ* workflow following the observations made in Section 2.2.3, $A_{\min}$ can be defined according to Eq. (7).

$$A_{\min} = k \cdot K \cdot A_f \quad (7)$$

where k is the fraction of A_f at which a particle is counted as a full fiber within the fiber counting algorithm. For fiber cross section cut angles of 0° (perpendicular to the fiber) $K = 1$, resulting in $A_{\min} = k \cdot A_f$. The fiber surface A_f can be calculated by the fiber radius and the conversion from micrometer to pixels based on the resolution.

$$A_f = \pi \cdot r_f^2 \cdot s \quad (8)$$

where r_f is the fiber radius in μm and s is the conversion of μm in px.

For calibration, a parameter study for k was performed in an interval $0.05 \leq k \leq 0.6$ for all samples with winding angle $\pm 90^\circ$.

Based on the variation of k , a mismatch in the FVF difference $\Delta\phi = \phi_{\text{ImageJ}} - \phi_{\text{imfindcircles}}$ between *ImageJ* and the *imfindcircles* algorithm is present. The optimal k factor is associated with the minimum value in the absolute difference between *ImageJ* and *imfindcircles*. The results of this procedure for different materials as well as different magnifications are shown in Fig. 7. Fig. 7(a) shows $\Delta\phi$ for different materials and magnifications for different k values. Towards lower k values, the FVF derived in *ImageJ* is overpredicted. Towards values of 0.5–0.6, the FVF is underpredicted in reference to the FVF derived by the *imfindcircles* workflow. The optimal values for each material and magnification are shown in Fig. 7(b).

As discussed in Section 2.2.3, $A_{\min}$ and therefore k is a function of different parameters. In addition to the polishing quality, there is also a dependence on the fiber diameter and the chosen magnification. Based on the magnification and the available resolution of the microscope, the fibers of the different materials are represented by different numbers of pixels. This phenomenon is visualized in Fig. 7(b), where k is shown as a function of fiber diameter d_f in px. The results show that the k values slightly increase with the increase of d_f . The more pixels are used to represent a fiber, the higher the k value. As an upper threshold, $k = 0.5$

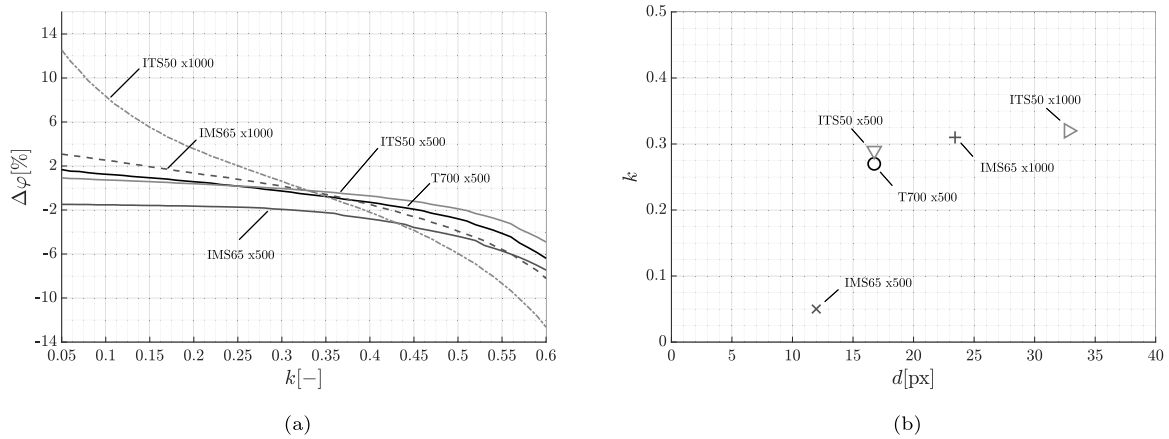


Fig. 7. Results of the calibration process using *ImageJ* and *imfindcircles* for winding angles of $\pm 90^\circ$: (a) shows the FVF difference ($\Delta\phi$) between *ImageJ* and *imfindcircles* based on a given k factor. (b) shows the optimal k -factor for each material and magnification based on the diameter of the fibers in pixels. A total number of 540 pictures were evaluated for this dataset.

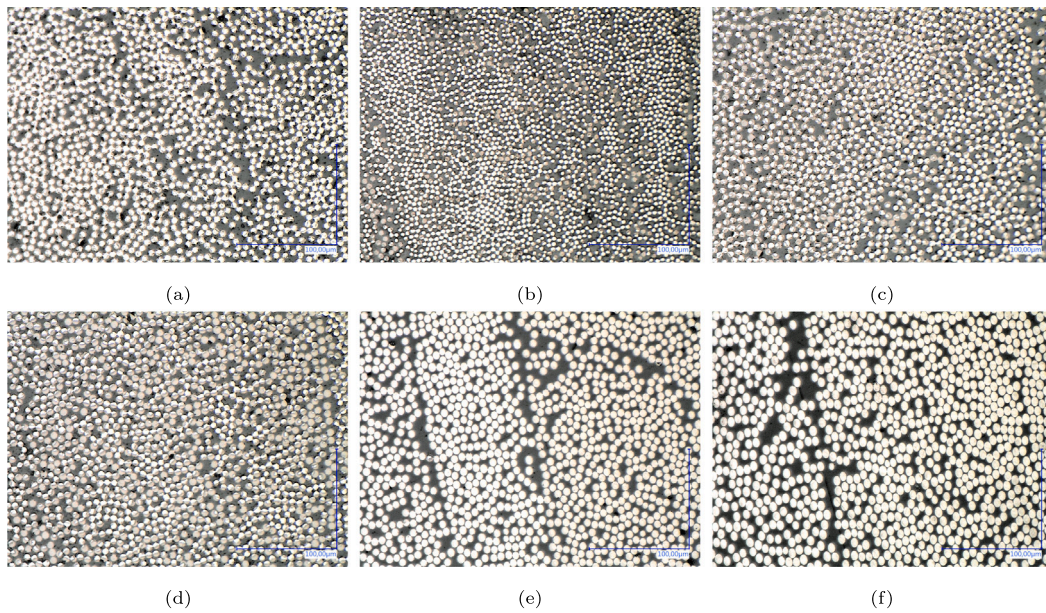


Fig. 8. Representation of the fiber appearance for all materials at a magnification of 1000 $\times$: (a) T700SC $\pm 90^\circ$, (b) IMS65 $\pm 90^\circ$, (c) ITS50 $\pm 90^\circ$, (d) ITS50 $\pm 75^\circ$, (e) ITS50 $\pm 60^\circ$, (f) ITS50 $\pm 45^\circ$.

can be set due to the counting approach of the edge fibers. It can be seen, that the values for all materials and magnifications vary within 0.27 to 0.32, except for the IMS65 500 $\times$ analysis. Here, the number of pixels is not sufficient for the proposed workflow. The close operation joins individual fibers, so that the k value needs to be chosen close to zero for a realistic FVF. Therefore, it can be stated that the fiber diameter in pixels should be at least 16 px for a robust workflow.

For winding angles deviating from $\pm 90^\circ$, the fiber edges are less frayed due to the polishing process. This is shown in Fig. 8. For all winding angles deviating from $\pm 90^\circ$, the fiber edges appear smoother. Here, it is advised to increase the k -Factor to a value of $k = 0.5$.

The following results can be summarized for the calibration process of k . These results are based on five data points obtained from three different materials and two magnification levels. Additional investigations using different materials and magnification levels are required to validate the present results more comprehensively.

- k is dependent on the polishing quality: low polishing quality results in lower k values.

- k is dependent on the pixel count to represent a fiber. Magnifications resulting in the representation of a fiber with less than 16 px are not advised.
- Optical calibration processes, as proposed in this study, are advised to improve the accuracy of the optical evaluation.

3.2. Effect of thermal fiber degradation on loss on ignition results

As discussed in Section 2.2.2, carbon fibers are affected by thermal degradation during the LOI process. Therefore, the mass loss $\dot{m}_f$ is measured. Fig. 9 presents the result on the thermal degradation of the different fibers used in this study for both process temperatures, 470 $^\circ\text{C}$ and 560 $^\circ\text{C}$.

At the beginning of the measurements, there are nonlinear phenomena present. This can be attributed to the sizing removal. Starting from a measurement point of $t = 50$ min, the fiber mass reduction is linear. A linear regression is used to derive the fiber degradation rate in Table 2(a).

Based on a correction time t_k using the measured fiber degradation rate $\dot{m}_f$, the corrected $\varphi_{\text{corrected}}$ can be calculated using Eq. (4). The

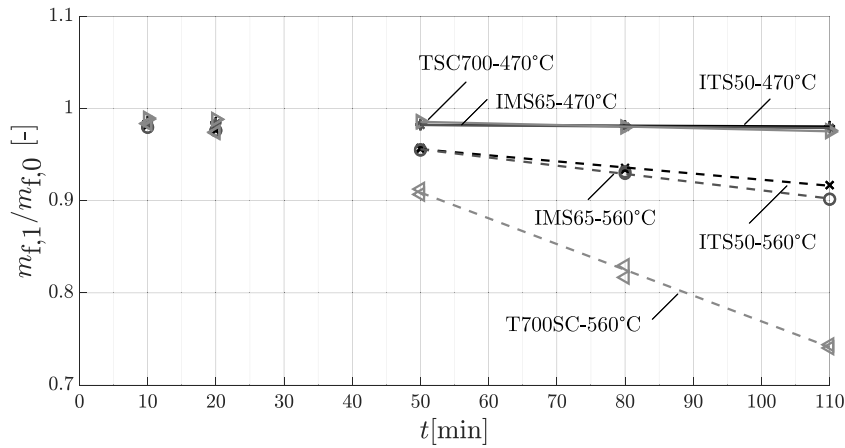


Fig. 9. Measurement results for the thermal fiber degradation based on the different fiber materials for 470 °C and 560 °C. The linear regression shown in the figure was performed from $t = 50$ min to $t = 110$ min.

Table 2

Results of the thermal degradation.

(a) Results of thermal degradation of the different fibers using different LOI process temperatures.			(b) Effect of FVF correction for the different material and process temperatures. The minimal value is 0% for $t_k = t_1$, and the maximum value is calculated on the assumption $t_k = 0$ min.		
$\dot{m}_f$ in %/min			Material	470 °C	560 °C
Fiber	470 °C	560 °C		$t = 80$ min	$t = 20$ min
ITS50	0.004	0.066	ITS50	0%–0.19%	0%–0.82%
IMS65	0.006	0.089	IMS65	0%–0.34%	0%–1.26%
T700SC	0.017	0.280	T700SC	0%–0.86%	0%–3.80%

choice of $\dot{m}_f$ is highly complex, as several nonlinear processes take place simultaneously. The resin is burned starting at the surface of the sample after a time delay following the insertion of the sample into the furnace. Subsequently, the fiber sizing is removed and consequently, the fibers start to degrade linearly. This process is also expected to be dependent on the surface-to-volume ratio of the sample due to the involved combustion processes. Summarizing, t_k determination is a highly complex nonlinear task. Precise knowledge of the thermal degradation is only possible considering all of the above-mentioned phenomena.

The LOI correction method proposed by Bleier [16] was applied to the measured data. The resulting values for t_k were all located near the end of the process window, or, in some cases, even close to the time at which the specimens are removed from the furnace. The corrected values for $\varphi_{\text{corrected}}$ resulted in unrealistically high results of $\varphi_{\text{corrected}} > 80\%$. Therefore, this correction could not be applied within this study. Additionally, the proposed correction method is based on a FVF-calibration value, which makes the result circularly dependent on this calibration method. As this study aims for an independent comparison, this approach was not chosen.

Therefore, in this study, a correction is performed on Eq. (4). For t_k , two limit values were considered using $t_k = 0$ min and $t_k = t_1$. Assuming $t_k = 0$, the FVF will be corrected by its maximum value based on $\dot{m}_f$. Assuming $t_k = t_1$, results in no correction at all. The correct value for $\varphi_{\text{corrected}}$ will lie between these two values. By this approach, the accuracy of the LOI process will be assessable. The results for this approach are shown in Table 2(b).

From Table 2 it can be concluded that the 470 °C LOI process generally results in lower φ increases, with regard to t_k variations. Due to the complexity in determining the precise t_k value, this process is preferred due to its lower dependency on the correction. Due to the nonlinear effects occurring during the LOI process, the uncertainty resulting from the correction approach is expressed as a function of

t_k . The accurate and material-specific determination of t_k for different specimens remains a topic for future studies.

In the following, the LOI results are presented without a correction. As a result, the FVF results tend to show lower values, compared to corrected values with $t_k > 0$. Regarding strength prediction, this can be seen as conservative, while for FVF-dependent stiffness determination of CFRP structure, this fact needs to be considered carefully.

3.3. Comparison of FVF characterization methods

Following, the FVF results for all materials are evaluated applying the methods described in Section 2.2.

First, the FVF results for the three $\pm 90^\circ$ materials are visualized in a box plot in Fig. 10. For the ITS50 material, the FVF derived from the LOI process shows lower values compared to the acid digestion value. The LOI process with shorter process time and higher temperature results in lower values. The optical assessment results in higher FVF values compared to the acid digestion. The standard deviations are very low for this material, which is in accordance with good manufacturing quality during the winding process. For the IMS65 material similar results can be observed. Both LOI values, for 470 °C as well as 560 °C, result in lower values compared to the acid digestion. The optical FVF is slightly higher compared to the acid digestion. Nevertheless, the deviation is less compared to the ITS50 values. Here the deviations within the methods are higher, which can be explained by lower winding quality due to towpreg width deviations. For the T700 results, the trend of higher acid digestion values is reversed. Here, the acid digestion values are lower compared to the LOI values. The trend that the 470 °C results in higher FVF remains. Considering the measured thermal fiber degradation, the LOI FVF values would need to be corrected within the limits of Table 2(b). This results in smaller deviations compared to acid digestion in case of ITS50 and IMS65 material, however the deviations for the T700 material increase.

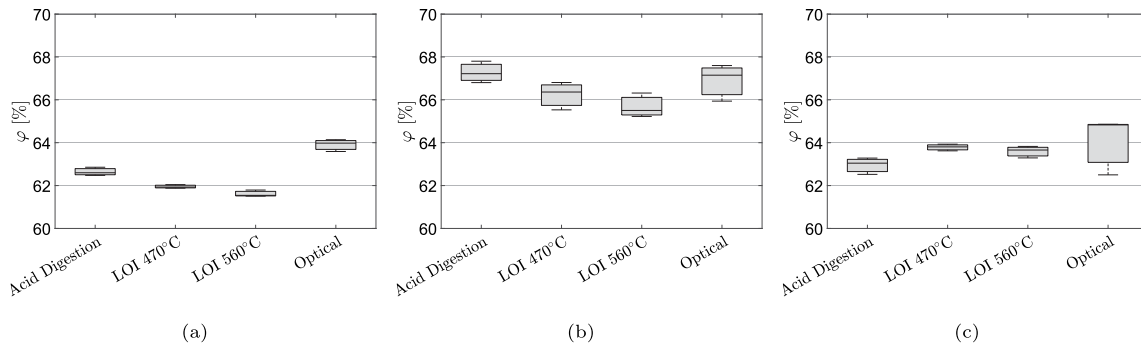


Fig. 10. FVF characterization results for $\pm 90^\circ$ materials, shown as boxplots: (a) ITS50 $\pm 90^\circ$, (b) IMS65 $\pm 90^\circ$, (c) T700SC $\pm 90^\circ$.

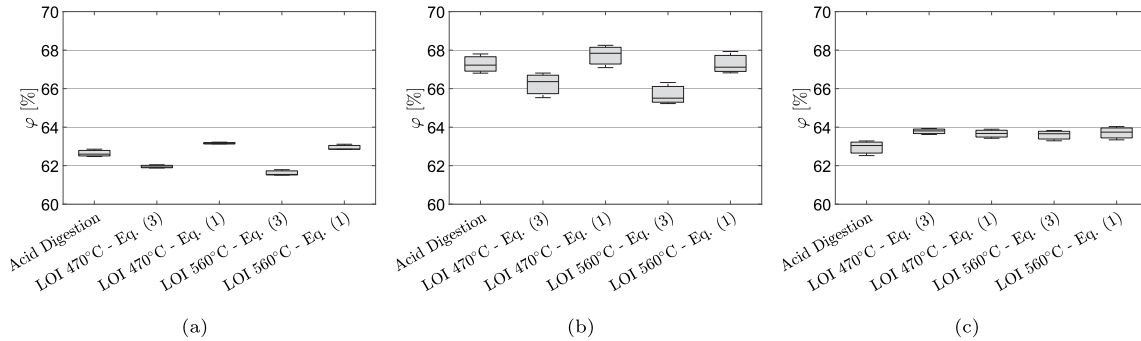


Fig. 11. FVF characterization results for $\pm 90^\circ$ materials, shown as boxplots applying Eqs. (1) & (3) for LOI FVF calculation: (a) ITS50 $\pm 90^\circ$, (b) IMS65 $\pm 90^\circ$, (c) T700SC $\pm 90^\circ$.

Overall, the FVF characterization methods show good agreement, with deviations of less than 1.59% in reference to the acid digestion result. An intraclass correlation coefficient according to McGraw and Wong [39], applying a two-way mixed-effects model for single measurements and absolute agreement, resulted in a value of 0.88, also indicating good agreement [40]. For the ITS50 and IMS65, the LOI process results in lower FVF values than for the T700, compared to the acid digestion. One reason for this could be that the LOI characterization strongly depends on the resin density. Depending on the resin system and the accuracy of supplier-provided density data, LOI-based results may be affected. The ITS50 and IMS65 material systems use the same resin system, whereas the T700 system employs a different resin. The acid digestion method is only dependent on the fiber density but not on the resin density (see Eq. (1)), because the FVF calculation is based on the sample density, which can be determined independently of the supplier data sheet. In contrast, application of Eq. (3) for the LOI evaluation requires both the resin and fiber densities. Alternatively, Eq. (1) can also be used to determine the FVF from LOI measurements. When this approach is applied, the ITS50 and IMS65 FVF values increase by about 1.3%, whereas the T700 values remain unchanged, as shown in Fig. 11. As a result, a consistent trend is observed across all materials, with LOI yielding slightly higher FVF than acid digestion, which is in agreement with experimental findings of an inter-laboratory trial organized in 1998 as reported in ISO 14127 [12]. It can be concluded that LOI-based FVF results calculated using Eq. (3) [1] exhibit a stronger dependence on supplier-provided material data than values obtained using Eq. (1). Precise knowledge of the fiber and matrix densities is therefore required when applying Eq. (3). Whenever possible, the matrix density should be experimentally verified. Regarding the thermal degradation process, the tendency of LOI measurements to result in higher FVF values than acid digestion when applying Eq. (1) indicates that the degradation onset time t_k is likely closer to the end of the LOI process.

A further observation was the tendency of the optical method to yield slightly higher FVF values. The reason for this can be found in the presence of resin accumulations towards the outer surface of the samples. Due to a matching manufacturing process compared to the CPV, shrink tape was applied on the outer layer before vacuum bagging. These resin accumulations are shown in Fig. 12 for the $\pm 90^\circ$ materials.

Using threshold-based determination of the resin pockets, the volumetric proportion of the resin pockets can be estimated. Therefore, the micrograph is transformed into a grayscale picture (bw), which is shown in Fig. 13. The resin is shown in white, the CFRP in black. Table 3 shows the trend that towards lower winding angles, the amount of resin pockets is increasing. The IMS65 shows the least resin pockets, which is in accordance with Fig. 10, resulting in no overprediction of the optical method compared to the ITS50 and T700 results. Due to the localized measurement of the optical method, the mean FVF is dependent on the micrograph location. As the micrographs are not captured automatically, a human effect is present within this process. Analyzing the data, a gradient in the thickness direction of the CFRP sample is present. Towards the tooling, at the right side of the images in Fig. 12, a higher FVF is present compared to the sample surface, facing away from the tooling. Therefore, a similar number of pictures were taken on the inside and outside of the samples. Nevertheless, the resin pockets on the far outside of the sample seem not to be captured in the correct statistical order by this human AOI selection process.

To demonstrate the influence of this, a sample was recorded in its entirety to show the effect of the image selection process by humans. The 138 pictures of this sample were grouped in inside, middle and outside location. Inside is referring to the tooling side. It can be observed from Fig. 14 that there is a gradient in FVF from inside to outside. Especially on the outside, the outliers are more pronounced, resulting in high deviations. Missing some of these low outliers results in higher mean FVF. This can be seen by the results in Table 4 when comparing the complete and partial scan. The non-automated recording results in

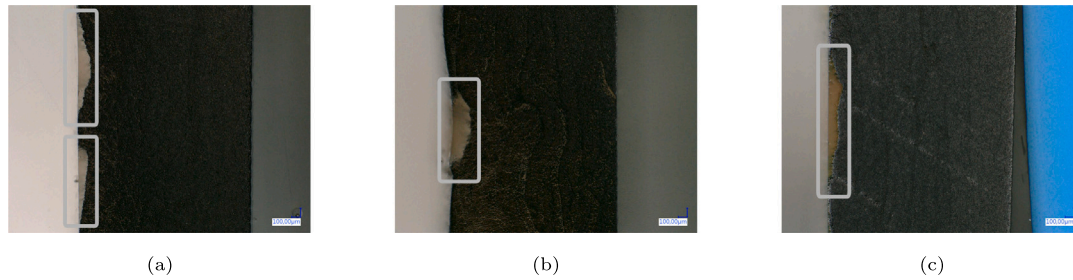


Fig. 12. Micrographs for the materials (a) ITS50 $\pm 90^\circ$, (b) IMS65 $\pm 90^\circ$ and (c) T700SC $\pm 90^\circ$. The different pictures show locations within the micrographs where resin accumulates at the laminate outer surface as a consequence of the curing process in the autoclave using shrink tape as surface finish. The location of the resin accumulation is highlighted by a gray box.

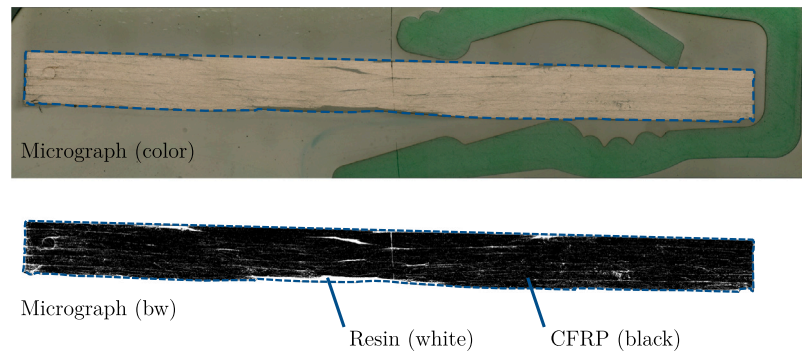


Fig. 13. Threshold based determination of the pure resin amount within a $\pm 45^\circ$ -ITS50-sample. For this sample, a pure resin content of 5.35 % is determined.

Table 3

Threshold-based determination of volumetric resin pocket percentage. For ITS50 75° , this workflow could not be applied because of insufficient contrast between $+75^\circ$ and -75° . Nevertheless, from optical evaluation, a similar value compared to ITS50 90° and ITS50 60° can be assumed.

Material	T700SC $\pm 90^\circ$	IMS65 $\pm 90^\circ$	ITS50 $\pm 90^\circ$	ITS50 $\pm 75^\circ$	ITS50 $\pm 60^\circ$	ITS50 $\pm 45^\circ$
Resin amount	2.6%	0.8%	2.2%	(2.55%)	2.9%	5.0%

Table 4

Results of the optical FVF compared to acid digestion for a complete and partial recording of the sample using the microscope.

Method	φ_{optical} in %	$\Delta\varphi_{\text{optical-acid digestion}}$ in %
Complete recording (n = 138)	63.11	0.47
Partial recording (n = 30)	63.98	1.34

slightly higher FVF values. Due to the chosen recording workflow of taking pictures for the optical method on the inside and the outside of the sample, this effect results in only slight overpredictions compared to the acid digestion method. However, this should not be considered as a disadvantage of this method. The optical method allows for local measurements while acid digestion and LOI only give insight into the mean bulk FVF. For the mechanical analysis of a CFRP structure, this fact needs to be considered. Resin pockets on the outside will lead to lower mean FVF values. The CFRP inside the sample shows higher FVF values compared to mean values. From a design point of view, the performance could be improved considering this local higher FVF. Therefore, the optical method provides more detailed and valuable insight into the CFRP structure and its performance capabilities.

The results of the samples deviating from a winding angle of $\pm 90^\circ$ are presented as boxplots in Fig. 15.

The trends are similar to the ones observed for material ITS50 $\pm 90^\circ$, shown in Fig. 10. The LOI processes tend to show lower FVF results compared to the acid digestion results. Again, the 560°C process results in lower values compared to the 470°C process, resulting in a higher difference with regard to the acid digestion. The optical FVF values are significantly higher compared to the mean values of acid digestion and LOI, resulting in a lower ICC value of 0.28. Referring to Fig. 15 and Table 3, increased resin pockets can be the reason for this. Caused by the winding process, intersections of tows are present in the tube in contrast to the $\pm 90^\circ$ tubes. At each intersection point, resin accumulations are likely to occur. As the optical method is only capable of acquiring 2D data, these locations may not be represented in the analysis sufficiently. Multiple section cuts along the axial direction of the tube are advised. In addition, this effect is added to the human-induced effect within the optical method, both effects resulting in an overprediction. After correcting the resin content based on the 2D data, the mismatch compared with the other methods is reduced. This result is indicated by “Optical (Resin)” in Fig. 15, increasing the ICC value to 0.66. This value is lower compared to the $\pm 90^\circ$ -ICC value, which may be due to the limitation of 2D measurements.

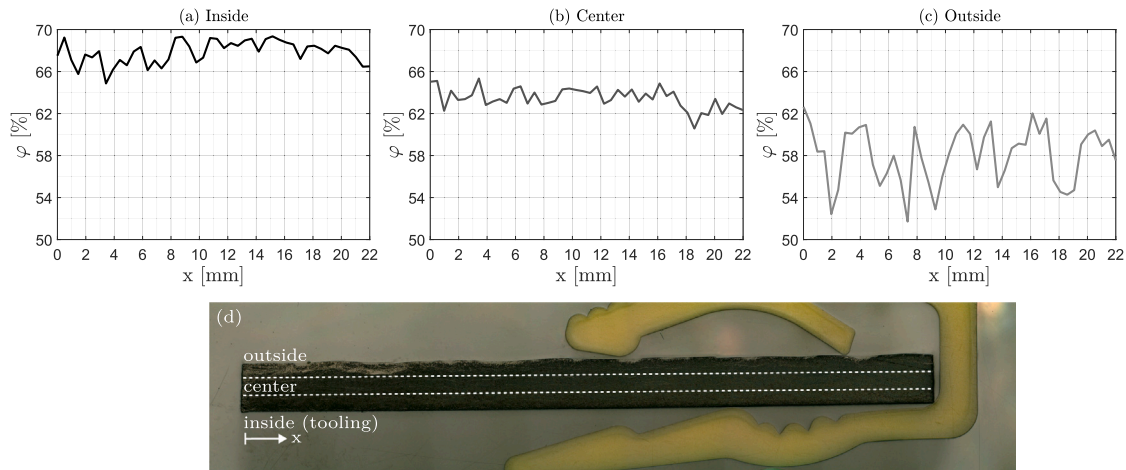


Fig. 14. Results of the optical FVF through the thickness of an ITS50 sample for the complete recording of the sample: (a) Results for the inside location of the sample (tooling side), (b) center location of the sample, (c) outside location of the sample as shown for the sample in (d). Inside is referring to the tooling side, outside to the surface of the sample. For each location, 46 pictures are acquired over the length x of the sample.

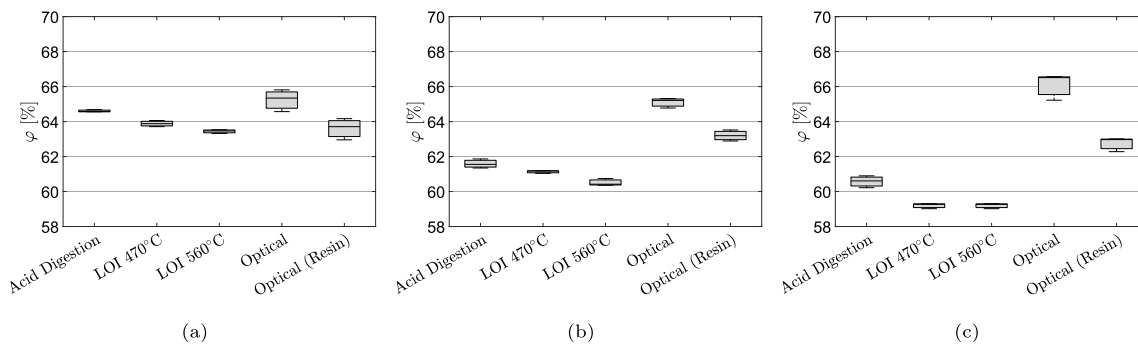


Fig. 15. FVF characterization results for all ITS50 samples showing lower winding angles than $\pm 90^\circ$ shown as boxplots: (a) ITS50 $\pm 75^\circ$, (b) ITS50 $\pm 60^\circ$, (c) ITS50 $\pm 45^\circ$.

Following the discussion of the individual results, the complete dataset is summarized in Table 5. In addition, for better visualization, Bland-Altman plots are presented in Fig. 16, using acid digestion as the reference method for comparison. Aligning with the previous discussion, the optical values result in higher values, especially for the angled layers, whereas the LOI methods show lower FVF values. The results obtained using the LOI process at 470°C are closer to those from acid digestion, indicating improved thermal stability compared with the 560°C process. Correction of resin content improves the accuracy compared to the acid digestion values. Nevertheless, it should be mentioned that the local FVF, derived by the optical method is expected to be correct. Therefore, the non-uniformity of FVF within the CFRP tubes should be considered in future analyses of CFRP-wound structures, especially in helical layers.

3.4. Effects on loss on ignition characterization using different process settings

In this section, the effect of different furnaces for the same LOI procedure is discussed as well as the vent settings of the furnace. As presented in the previous section, the LOI process using 470°C showed

accurate results compared to acid digestion. Therefore, this method could be employed as a fast method for an accurate FVF result.

3.4.1. Effect of different furnaces

As shown in Section 2.2.2, each furnace for the LOI process has a unique temperature profile. This leads to deviations within the FVF characterization.

For each material with $\pm 90^\circ$ winding angle, measurements were performed in the *Nabertherm* furnace as well as in the *Heraeus* furnace. Table 6 shows the comparison for the LOI process using 470°C . The results of this investigation indicate that the measurement using the *Nabertherm* furnace consistently results in higher FVF values compared to *Heraeus*. The increase of FVF is between 0.49–0.99%. One reason may be associated with the different temperature profiles. To minimize this effect as much as possible, the samples were already located at appropriate locations within the temperature profile in the furnace for this investigation. Another effect can be found in different atmospheres within the furnace. Comparing the LOI results to the acid digestion values, it can be concluded that the *Nabertherm* furnace shows smaller deviations.

Table 5

FVF characterization method results for all materials. The LOI results are obtained using Eq.(3). The optical values are determined using 500× magnification, except for the IMS65 (1000×). For 500× magnification, 30 pictures were evaluated per sample, for 1000× magnification, 45 pictures were analyzed. The analyzed volume based on these pictures represents ≈1 % for 500× and <1 % for the 1000× magnification. The average value through-thickness is presented. The effect of resin accumulations, observed in the following detailed analysis for the angled materials using the optical evaluation is presented in column Optical (Resin). The results in the table contain measurements on 90 samples. The area of evaluation is presented in reference to one ring, cut from the tube. The minimum sample count n was based on the requirements of the applicable standards.

Material	φ in %				
	Acid digestion	LOI 470 °C	LOI 560 °C	Optical	Optical (Resin)
Volume of evaluation sample count	20 % $n = 3$	20 % $n = 3$	20 % $n = 3$	≤1 % $n = 3$	≤1 % $n = 3$
T700SC ±90°	62.95 ± 0.32	63.79 ± 0.13	63.59 ± 0.22	64.06 ± 1.11	–
IMS65 ±90°	67.28 ± 0.41	66.24 ± 0.53	65.68 ± 0.46	66.90 ± 0.70	–
ITS50 ±90°	62.64 ± 0.16	61.95 ± 0.07	61.61 ± 0.13	63.90 ± 0.23	–
ITS50 ±75°	64.60 ± 0.06	63.88 ± 0.15	63.44 ± 0.09	65.24 ± 0.51	63.61 ± 0.50
ITS50 ±60°	61.59 ± 0.21	61.13 ± 0.07	60.51 ± 0.17	65.10 ± 0.23	63.20 ± 0.26
ITS50 ±45°	60.58 ± 0.28	59.51 ± 0.16	59.20 ± 0.13	66.10 ± 0.62	62.75 ± 0.33

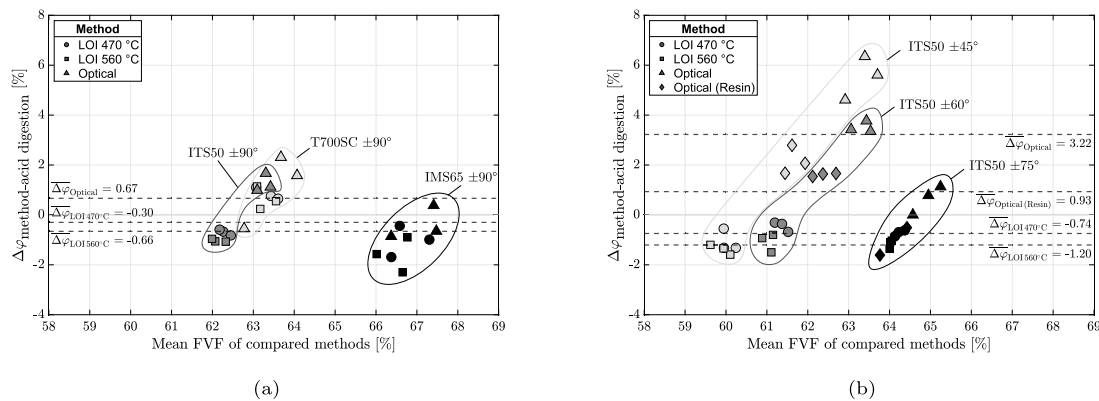


Fig. 16. Bland–Altman analysis of the FVF characterization methods for (a) ±90° materials and (b) angled ITS50 materials with winding angles of ±75°, ±60°, and ±45°. The mean FVF of the compared method and the acid digestion value is plotted against the deviation from acid digestion. Dashed horizontal lines indicate the mean deviations for LOI at 470 °C, LOI at 560 °C, optical evaluation, and resin-corrected optical evaluation. The angled materials show increased deviations for the optical method, whereas the resin correction reduces the mismatch with respect to acid digestion.

Table 6

Results of FVF characterization by LOI using the 470 °C cycle for two different furnaces. Each sample set consists of three measurements ($n = 3$).

Material	φ in %		
	Nabertherm furnace	Heraeus furnace	$\Delta\varphi_{\text{Nabertherm-Heraeus}}$
T700 ±90°	63.71 ± 0.13	62.94 ± 0.06	0.77
IMS65 ±90°	65.95 ± 0.53	64.96 ± 0.21	0.99
ITS50 ±90°	61.99 ± 0.07	61.53 ± 0.12	0.46

Table 7

Results of FVF characterization by LOI using the 470 °C cycle for different supply air settings of the door of the furnace. Each sample set consists of three measurements ($n = 3$).

Material	φ in %		
	Supply air closed	Supply air open	$\Delta\varphi_{\text{open-closed}}$
ITS50 ±45°	59.63 ± 0.07	59.78 ± 0.18	+0.15
ITS50 ±60°	61.11 ± 0.07	61.10 ± 0.18	−0.01
ITS50 ±75°	63.80 ± 0.15	63.66 ± 0.06	−0.13

3.4.2. Effect of vent setting

The second parameter for the furnace, which can be varied within the LOI process is the vent setting. Therefore, comparative measurements using the *Nabertherm* furnace have been carried out on the ITS50 samples with ±45°, ±60° and ±75° winding angles. The results are summarized in Table 7. The results show that there is no clear trend present for the vent setting. The maximum absolute difference in FVF is 0.23 % for the ±45° samples, which is slightly higher compared to the standard deviation. Based on these results, a closed vent setting is preferred for the operator, as less combustion gases enter into the laboratory through the open vent. The closed vent was used for all other measurements presented above.

3.5. Layerwise FVF characterization of a composite pressure vessel

Based on the results presented above, the optical FVF characterization methodology is applied to a CPV laminate. Therefore, a CPV with seven layers was manufactured using the ITS50 material system. The CPV consists of three hoop layers and four helical layers, resulting in a wall thickness of approximately 4.9 mm.

The cross sections for the CPV laminate are shown in Fig. 17 for two different cut sections. These cross-sections were taken from a tested CPV, which is why Fig. 17 shows the presence of inter-fiber failure (IFF) within the laminate. The pictures acquired for evaluation for each layer were captured in areas without the presence of IFF. For each layer, at least 15 pictures were taken for analysis.

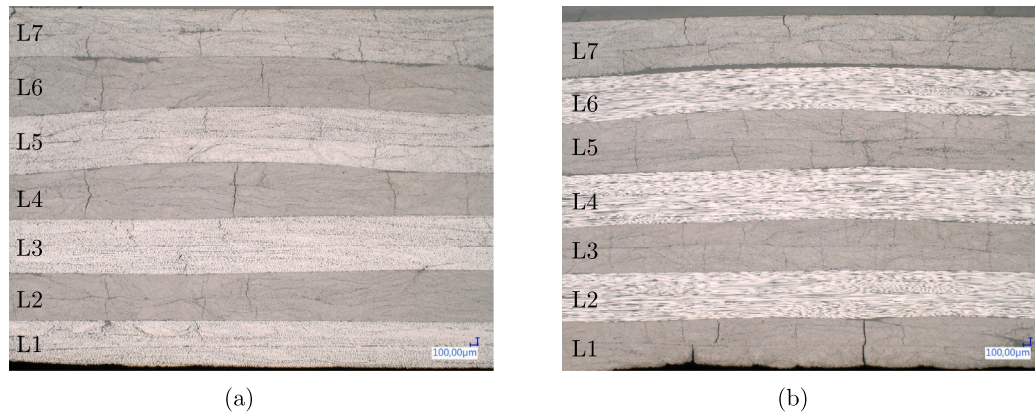


Fig. 17. Cross sections of the CPV laminate from two different section cut angles: (a) section normal in the circumferential direction, (b) section normal in the meridional direction. The mandrel side of the CPV laminate is located at the bottom of the picture.

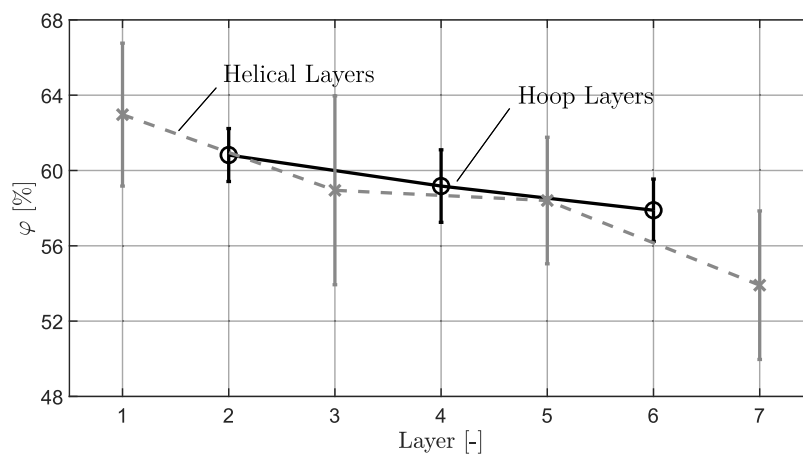


Fig. 18. Layerwise FVF results for the CPV laminate.

Table 8

FVF results for the CPV laminate derived by optical assessment and comparison with the LOI full-thickness reference value. Layer 1 is located at the mandrel side, while Layer 7 is located at the outer side of the CPV laminate. The LOI value was derived from a full-thickness measurement of the CPV laminate.

(a) Layerwise FVF values derived by optical assessment. $\Delta\varphi$ is calculated by φ_{Optical} layer minus the mean FVF $\bar{\varphi}_{\text{LOI}}$				(b) Mean FVF values and deviation between optical assessment and LOI.	
Layer	Winding angle	φ_{Optical} %	$\Delta\varphi_{\text{Optical-LOI}}$ %	Quantity	Value
1	$\pm 17^\circ$	62.97 %	+5.15 %	$\bar{\varphi}_{\text{Optical}}$	58.87 %
2	$\pm 90^\circ$	60.82 %	+3.00 %	$\bar{\varphi}_{\text{LOI}}$	57.82 %
3	$\pm 17^\circ$	58.95 %	+1.13 %	$\Delta\varphi_{\text{Optical-LOI}}$	+1.05 %
4	$\pm 90^\circ$	59.17 %	+1.35 %		
5	$\pm 29^\circ$	58.40 %	+0.58 %		
6	$\pm 90^\circ$	57.89 %	+0.07 %		
7	$\pm 42^\circ$	53.91 %	-3.91 %		

Based on the winding angles, the parameter K can be calculated and consequently, the optical FVF methodology can be applied. As reference measurement, the mean optical FVF of the CPV analysis is compared to a measurement using the 470 °C LOI process.

The results for each layer, as well as the mean values of the optical and LOI method are shown in Fig. 18. For each layer, depending on its type, circumferential or helical, the FVF is shown as a function of the layer position. In Table 8(a) the results are summarized for each layer. The difference against the mean LOI value is also provided. Table 8(b) compares the mean values of LOI and the optical method (see Fig. 18).

Again, the difference in LOI and optical assessment can be explained with the presence of resin accumulations. When analyzing Fig. 17(b), a resin accumulation is present between layer 6 and 7. Due to the impact of compaction of the last hoop layer, resin is pushed out of the laminate. The thickness of this resin interlayer is approximately 48 µm, which is 0.98 % of the total thickness of the laminate of 4898 µm which aligns well with the 1.05 % over-prediction of the optical assessment. Taking the resin interlayer into account, the optical method results in low deviations compared to the LOI characterization.

It can be noted from Fig. 17 that there is a decrease in FVF from layer 1 to layer 7. The highest FVF is present in layer 1, the lowest FVF

can be found at the outside of the laminate at layer 7. Referring to Table 8(a), deviations from +5.15 % to −3.91 % compared to the mean LOI FVF are present within the layout. Considering micro-mechanical models or the rule of mixtures, this FVF gradient can be expected to influence the mechanical response to inner pressure of the CPV. Therefore, the FVF gradient should be analyzed as accurately as possible and implemented into numerical CPV models for failure prediction [9,10,41].

The hoop layers show overall lower standard deviations compared to the helical layers. For the hoop layers, the decrease in FVF is almost linear. For the helical layers, there is a decrease from layer 1 to 3. The third helical layer (layer 5) shows almost the same FVF compared to the second (layer 3). Due to the presence of no additional hoop layer on the outside of the laminate, there is a strong decrease for the final helical layer, resulting in the lowest FVF of 53.91 %.

These results are in good alignment with other studies [9]. In contrast to the wet-winding results in [9], the decrease of FVF per layer from inside to outside seems to be less for towpreg winding. The reason for this could be found in the fact that during wet winding, the compaction applied by hoop layers within the winding process already causes resin flow from inside (mandrel) to outside during manufacturing. In contrast, during towpreg winding, the resin flow occurs mainly during the autoclave process. This effect should be investigated for different CPV configurations to understand the influence of different layout types and CPV thicknesses.

The application of the optical FVF characterization to a CPV laminate was demonstrated, resulting in good agreement between the optical method and LOI. Therefore, it can be concluded that the proposed method is capable of capturing through-thickness FVF gradients in layered CFRP structures.

4. Conclusion

A comprehensive study on the FVF characterization using different methods such as acid digestion, loss on ignition and optical assessment was carried out. A detailed calibration process of the optical assessment is presented. Different material systems were analyzed to prove the applicability of the workflow for different fiber diameters and different resin systems. The workflow was applied to the FVF analysis of a CPV.

The following conclusions can be drawn based on the results of this study:

- An optical characterization methodology is implemented in the open-source software *ImageJ* which can be used for the FVF characterization of layered CFRP structures or ply angles up to 45°. By preparation of two cross-sections, all fiber angles can be analyzed using this methodology. A calibration process for $A_{\min}$ is advised to account for the individual polishing quality, resulting in the highest accuracy. For the materials analyzed in this study, k ranged from 0.27 to 0.32 when the fiber diameter d was represented by more than 16px. This range can be regarded as a preliminary guideline for the investigated material systems and imaging conditions. However, additional calibration studies are required to further validate the transferability and robustness of this k -range as well as the d limitations.
- The optical FVF characterization enables local resolution of FVF values within CFRP structures. While this represents a key advantage, it also constitutes a limitation, since the evaluated volume is smaller than that considered in acid digestion and LOI, particularly when automated image acquisition is not available. Therefore, optical measurements either require the assumption that the two-dimensional cross-sectional evaluation is representative of the three-dimensional FVF distribution, or additional section cuts at different locations within the CFRP component should be evaluated.

- An automated image acquisition process is advised to capture all regions of the sample in a statistically representative manner. Images taken by hand tend to result in higher FVF due to operator-dependent image acquisition. A systematic comparison of these results would provide valuable insight into the inter-operator and intra-operator repeatability of the optical assessment methodology.
- For best accuracy of the optical FVF characterization, the polishing quality should be as good as possible with only limited artifacts and scratches. In addition, the higher the resolution of the microscope, the more stable the fiber counting algorithm can be implemented.
- The comparison of the different FVF characterization methods shows overall good agreement of the different methods with deviations of less than 1.06 % for the 470 °C LOI process, 1.59 % for the 560 °C LOI process and 2.18 % for the optical process (including resin content correction) compared to acid digestion, especially for the $\pm 90^\circ$ -materials. Thermal degradation of the fibers during the LOI tends to result in lower FVF values derived by LOI compared to acid digestion when applying Eq. (3). This trend is less pronounced when applying Eq. (1). Within the 470 °C process, this effect of thermal degradation is less compared to the 560 °C process temperature. A disadvantage of acid digestion and LOI is the non-localized measurement result. Manufacturing-induced resin pockets can result in overall lower mean FVF values compared to the optical FVF of each layer which is very important to consider within the design of CFRP parts.
- The proposed workflow can be applied to CPVs, enabling through-thickness characterization of local FVF of different winding layers of CPV layout designs, improving the design analysis accuracy. Reference measurements resulted in deviations from the FVF characterized by LOI of less than 1.05 %.

4.1. Recommended future research

Based on the presented results in this study, different future research aspects can be suggested to improve the proposed workflow for characterizing FVF in layered CFRP structures.

One aspect is to add additional data to the calibration process for optical FVF characterization. It is expected that additional data based on different fiber diameters and resolutions will help extend the proposed workflow to a broader range of materials. It is expected that a general equation for the choice of k can be derived as a function of the fiber diameter and the polishing quality.

Another aspect is a detailed study of the effect of the surface finish of the wound tubes on the FVF characterization. It is expected that a peel-ply surface finish will increase the overall FVF of the samples, showing fewer resin pockets on the laminate outside. It should be evaluated whether the bulk FVF is increased and the gap between the optical and bulk results is minimized.

In addition, with the recent developments of artificial intelligence, it is recommended to add a study on the performance of AI-based image detection for layerwise FVF characterization of CPVs based on the derived data from different materials.

CRedit authorship contribution statement

Gerrit Rehs: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Investigation, Conceptualization. **Oliver Dammann:** Software, Methodology, Investigation, Conceptualization. **Daniela Feldten:** Writing – review & editing, Software, Investigation. **Nils Meyer:** Writing – review & editing, Software, Investigation. **Tobias Dickhut:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Prof. Dr. Tobias Dickhut reports financial support was provided by Universität der Bundeswehr München Universitätsbibliothek. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix. Matlab algorithm for optical fiber volume fraction characterization

Listing 1: Matlab Algorithm for optical FVF-characterization based on version R2023a using MIJ

```

1      %% Input parameters
2      r_Fiber_micrometer = 3.5; % r in um
3      angle= 0; % Fiber Angle
4      roiSize=1000; % ROI in px
5      Scaling_px_um = 0.418; %Scaling for
        x500
6      %% Calculation of parameters
7      r_Fiber_pixel = r_Fiber_micrometer/
        Scaling_px_um;
8      A_min = pi*r_Fiber_pixel^2*(1/cos(
        deg2rad(angle)))*(29/100);
9      ROI_micrometer = roiSize*
        Scaling_px_um;
10     %% Start MIJ
11     javaaddpath 'C:\Program Files\
        MATLAB\R2023a\java\mij.jar'
12     javaaddpath 'C:\Program Files\
        MATLAB\R2023a\java\ij.jar'
13     MIJ.start
14     %% FVF Algorithm
15     MIJ.run('Open...', ['path=[C:\...
        path...\jpg]'])
16     MIJ.run("8-bit", "");
17     MIJ.run("Canvas Size...", ['width='
        ,num2str(roiSize),' height='
        ,num2str(roiSize),' position=
        Center']);
18     MIJ.run('Threshold...', '
        setAutoThreshold=Default');
19     MIJ.run("Close");
20     MIJ.run("Smooth", "");
21     MIJ.run("Sharpen", "");
22     MIJ.run("Convert to Mask", "");
23     MIJ.run("Close-", "");
24     MIJ.run("Open", "");
25     MIJ.run("Watershed", "");
26     MIJ.run("Analyze Particles...", ['
        size=',num2str(A_min),'-
        Infinity display clear']);
27     Fiber_count=length(MIJ.
        getResultsTable());

```

```

28     FVF = (Fiber_count*pi*
        r_Fiber_micrometer.^2*(1/cos(
        deg2rad(angle)))/(
        ROI_micrometer*ROI_micrometer)
        *100;
29     MIJ.run('Clear Results')
30     MIJ.closeAllWindows();
31     MIJ.exit
32     %% FVF-Result
33     fprintf('The calculated fiber
        volume fraction is %.2f%%\n',
        FVF);

```

Data availability

Data will be made available on request.

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