Advanced X-Ray Tomographic Methods for Quantitative Characterisation of Carbon Fibre Reinforced Polymers

Johann KASTNER, Bernhard PLANK, Andreas REH, Dietmar SALABERGER, Christoph HEINZL
Upper Austria University of Applied Sciences, Stelzhamerstrasse 23, 4600 Wels, Austria

Abstract. In recent years, a strongly growing demand of aeronautic industry for light weight materials like carbon fibre reinforced polymers (CFRP) motivated by economic and environmental issues is observed. The main interest of using CFRP composites is based on weight reduction, and on the high fatigue resistance capabilities. The next generation aircrafts, as the Airbus A350 XWB aim to be made of more than 50 % of composite materials. However with the increase of the composite share the efforts for non-destructive testing are exploding. X-ray computed tomography (CT) is to date a very promising method for non-destructive and fully three dimensional characterizations of composite aeronautic components. CT provides a volumetric map of a specimen including internal and external 3D-structures, generated from a set of radiographs. This paper covers the quantitative characterization of CFRP parts by advanced X-ray computed tomography and data analysis methods. The discussed topics are: High resolution CT, phase contrast CT, quantitative evaluation of fibre length and orientation distribution of fibres, quantitative determination of porosity and interactive exploration and visual analysis of porosity.

1. Introduction

X-ray computed tomography (CT) provides a volumetric map of a specimen in three dimensions, generated from a set of radiographs. Due to the speed and quality of measurement, CT systems with cone beam geometry and matrix detectors have become widely used [1,2]. Continuous improvements in the quality and performance of X-ray tubes, detectors and devices have led to cone beam CT systems which can now achieve spatial resolutions down to 1 µm and even below. Applications of high-resolution X-ray computed tomography for the 3D-characterization of carbon fibre reinforced polymers (CFRP) are reported. The main topics are:

1. High resolution CT: State of the art cone beam CT-systems can reach voxel sizes below 500 nm and resolutions below 1 µm. Selected high resolution applications are presented.
2. Phase contrast CT: X-ray imaging methods based on the phase shift of the X-ray beam allow a significant increase of the contrast enhancing the interface. We compare a propagation based phase contrast CT-method with an interferometric CT method based on the Talbot-Lau interferometer and discuss the possibilities.
3. Quantitative evaluation of fibre length and orientation distribution of fibres: CT-data are processed by applying several consecutive steps: pre-processing, segmentation, medial axis extraction, individual fibre extraction including cluster
analysis and final fibre characterisation. In this way quantitative data of every individual fibre are extracted.

4. Quantitative determination of porosity: The porosity of CFRP composites is a very important topic for the practical application since there is a direct correlation between porosity and mechanical properties, such as shear strength. A segmentation method to evaluate the CT data and to obtain a measure of porosity with a high level of repeatability and a high correspondence to values obtained by ultrasonic testing and acid digestion is presented.

5. Interactive exploration and visual analysis of porosity: A visualization pipeline for the interactive exploration and visual analysis of CFRP specimens is presented. Porosity maps are introduced to allow a fast porosity evaluation and to calculate local pore properties, i.e., volume, surface, dimensions and shape factors.

2. Experimental – CT Scanning

X-ray tomograms were scanned using a “nanotom 180NF” CT desktop device, developed and manufactured by GE Sensing & Inspection Technologies: phoenix|x-ray with a 180 keV high-power nano-focus tube with transmitting target and a 2300x2300 pixel Hamamatsu detector. The X-ray tube of the “nanotom” is equipped with an external liquid cooling system to ensure stable measurement conditions and to minimise thermal influences during scans of longer duration. The cone-beam CT data were reconstructed by a filtered-back projection Feldkamp algorithm [2]. The reconstructed data were processed and visualised with the commercial software VGStudio MAX 2.2 and an in-house developed software called iAnalyse. Bigger components from aeronautic industry are usually scanned by using a Rayscan 250E CT-device. The Rayscan 250E system is equipped with a 225 keV-µ-focus tube and 450 keV mini-focus tube. As detector a 2048x2048 a-Si flat panel matrix-detector by Perkin Elmer is used.

3. Results and Discussion

3.1 High Resolution X-ray Computed Tomography

As described cone beam CT-systems can reach voxel sizes below (500 nm)$^3$ and resolutions below 1 µm. Figure 1 shows a cross-sectional CT-picture (left) and a three-dimensional visualization (right) of a short carbon-fibre reinforced polymer (polyamide PA66) produced by injection moulding. The diameters of the carbon fibres are around 7-8 µm.
Fig. 1. Cross-section of CT-data of carbon-fibre reinforced polyamide manufactured by injection molding and a 3D representation of the individual fibres in the matrix. Voxel size was (1.25 μm)³.

Figure 2 shows a detailed view of a cross section (left) and a three-dimensional visualization (right) of the CT-data of a carbon-fibre reinforced polymer (CFRP) manufactured by PrePreg technology. Individual carbon fibres, resin, macro and micro-pores can be clearly distinguished.

Although the density differences between the carbon fibres (density ~1.75 g/cm³), the polymer matrix PA66 (density ~1.14 g/cm³) and the epoxy matrix (density ~1.26 g/mm³) are rather low, the individual fibres can be clearly seen in both examples.
3.2 Phase Contrast Tomography

When X-ray attenuation coefficients at the interface between two different materials are too close, conventional absorption-based methods can fail to show a significant difference in the resulting grey-values. In such cases phase-imaging methods can increase the contrast significantly and the edges can be enhanced [3]. X-ray computed tomography phase imaging methods can be classified into (1) interferometric methods, (2) techniques using an analyser (or diffraction enhanced imaging) and (3) free space propagation based methods (also called in-line based methods) [4]. The influence of propagation based phase contrast on the resulting CT-data and the detectability of details are shown in Fig. 3. Here the object-detector distance ODD is increased from 189 to 486 mm, which results in an increase in the phase contrast and detail detectability.

![Fig. 3. Cross-sectional CT-data of a carbon fibre reinforced polymer sample at different object-detector distances: 189 mm at the left picture and 486 mm at the right picture. Voxel size was constant at (2.75 μm)³.](image)

The increase in phase contrast with increasing distance between the object and the detector can be seen also in the grey value profiles along a certain pore. This is shown in the left graphic of Fig. 4. The corresponding phase contrast values for the various distances between the object and the detector are shown in the right graphic of Fig. 4. The phase contrast values were calculated from the upward and downward overshooting of the grey value profiles across the edges of a pore within the specimen.

![Fig. 4. Grey-value profile along a pore from Fig.3 within the CFRP with different object-detector-distances (left picture) and phase contrast values versus object-detector-distance (right picture).](image)
In the next section we present results of an interferometric CT method based on the Talbot-Lau interferometer [4]. A CFRP sample with about 4% porosity was investigated with the Talbot-Lau interferometer setup. Fig. 5 displays the CT axial slices of the linear absorption coefficient $\mu$, the refraction coefficient $\delta$ and the linear scattering coefficient $\varepsilon$. Here, only the larger macro pores appear, whereas the resolution does not allow seeing the small micro pores within the fibre bundles. In the absorption CT slices, the fibre bundles themselves cannot be distinguished from the epoxy matrix and they appear with limited contrast in the phase contrast CT pictures. However, the fibre bundles are by nature heterogeneous and thus lead to a strong scattering signal. Consequently, the bundles parallel to the section plane appear clearly in the cross-sectional picture of the scattering coefficient in the form of white stripes (right picture).

**Fig. 5.** Cross-sectional CT pictures of the CFRP sample with absorption contrast (left picture), phase contrast (picture in the centre) and dark field scatter contrast (right picture). The CT-data were acquired with a Talbot-Lau interferometer by CSEM (Switzerland). Voxel size was $(44 \, \mu m)^3$. Adapted from [4].

### 3.3 Quantitative Evaluation of Fibre Length and Orientation Distribution of Fibres

From the CT-data quantitative data can be extracted. For example, the CT-data of glass fibre reinforced polymer can be processed to extract fibre orientation and fibre length distribution [5]. The CT-data is processed by several consecutive steps: pre-processing, segmentation, medial axis extraction, individual fibre extraction including cluster analysis and final fibre characterisation [5]. Figure 6 shows fibre length distribution functions of polypropylene granules with 10%, 30 %, 40 %, 50 % and 60 % of glass fibres (PPGF). It is clearly recognisable that the fibre length distribution function is shifted to lower values with increasing fibre content. The higher the fibre content the more fibres will be broken during the manufacturing process.
Fig. 6. Fibre length distribution function of granules with 10 %, 30 %, 40 %, 50 % and 60 % glass fibres (PPGF). The fibre length distribution was determined from CT-data with (2 µm)³ voxel size.

Similar results can be expected for carbon fibre reinforced polymers, even though the fibres are usually much thinner and the contrast is significantly lower. The left picture of Fig. 7 shows a 3D-representation of the CT-data of a CFRP-sample. The right picture shows the extracted fibres. The orientation of the fibres is visualized in green and red.

Fig. 7. CT-data a CFRP volume of (570x356x374) µm³. The voxel size was (1 µm)³. The orientation of the fibres is visualized by colour coding.

3.4 Quantitative Determination of Porosity

The porosity of carbon fibre reinforced polymers is a very important topic for the practical applications since there is a direct correlation between porosity and mechanical properties, such as shear strength. Therefore the porosity values of CFRP-samples for practical use must be below a certain level, usually 2.5 - 5 %. The most common non-destructive method for determining the porosity is ultrasonic testing, since there is a mostly linear correlation
between ultrasonic attenuation and porosity. However, the ultrasonic attenuation coefficient depends not only on the porosity, but also on the shape and distribution of the pores and the presence of other material inhomogeneities, which can result in significant errors.

Another very promising method for the non-destructive determination of porosity of composites is X-ray computed tomography. One of the main problems regarding porosity determination using CT is found in the accurate and reproducible determination of the surface between material and air. The left picture in Fig.8 shows cross-sectional CT pictures of CFRP samples with increasing porosity 0, 0.9 % to 4.5 % porosity. For determination of the exact porosity values we have developed a thresholding method based on the grey value histogram [6]. The accuracy of our quantitative porosity determination has been shown using real world CFRP parts. The results have been compared to the reference methods ultrasonic testing and acid digestion. The right picture of Fig. 8 shows, that there is good correlation of the porosity obtained by CT versus the porosity obtained by ultrasonic testing. The correspondence is very good, since the correlation factor is 0.984.

3.5 Interactive Exploration and Visual Analysis of Porosity

Besides quantitative porosity determination the visualization and the calculation of local pore properties, i.e., volume, surface, dimensions and shape factors is also important for a non-destructive testing practitioner or a materials scientist. Since CT gives a complete 3D-representation of a CFRP-specimen including inner and outer surface CT-data are a good basis for visualization of material properties and geometry. We have developed an approach for interactive exploration and visual analysis of CFRP specimens to enhance the evaluation of such components and materials [7]. Our solution includes the calculation of local pore properties for pore classification and their visualization. Beside the porosity map view, the introduced pipeline (shown in Fig. 9) integrates region of interest and parallel coordinates interaction in a two stage drill-down approach. The best viewpoint widget allows to see the quality of the viewpoints on a sphere.
Fig. 9. Overview of the porosity visualization pipeline for interactive exploration and visual analysis of porosity in CFRP specimens. Adapted from [7].

An example for the application of the interactive exploration and visual analysis of a certain volume of a CFRP-sample is shown in Fig. 10. In the left image, a porosity map view of the specimen is depicted. The use of parallel coordinates allows the user to filter pores with specific pore properties. In the central picture of Fig. 10 the biggest pore in the selected region of interest is highlighted. The right picture of Fig. 10 shows long and thin micro pores in the fibre bundles. For further filtering of pores in a specific direction, the dimensions bounds can be set in the parallel coordinates view additionally to the shape factor. Selected pores are highlighted in colour. Not selected pores are given in black where white and gray illustrate the typical twill weave pattern of the fibre bundles.

Fig. 10. Porosity map view of a CFRP specimen (left), where low porosity are colored in blue and higher porosity in green and red. Porosity evaluation of a region of interest (right) showing different pore classifications using parallel coordinates interaction. The biggest pore in the region is selected and highlighted in the central picture. Only the small micro pores between the fibres are highlighted on the right picture.
4. Conclusion and Summary

X-ray computed tomography is a powerful non-destructive testing tool for characterization and visualization of different properties of carbon fibre reinforced composites. Different aspects of CT applied to CFRP like high resolution CT, propagation based phase contrast CT and interferometric phase contrast CT were discussed. The quantitative evaluation of fibre length and orientation distribution of individual fibres was presented. Additionally a threshold based method for quantitative determination of porosity in CFRP specimens by CT was introduced and compared with results obtained by ultrasonic testing. Furthermore, interactive exploration and visual analysis of porosity on different samples were applied.

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References