

# Defect and Porosity Determination of Fibre Reinforced Polymers by X-ray Computed Tomography

Johann KASTNER<sup>\*</sup>, Bernhard PLANK<sup>\*</sup>, Dietmar SALABERGER<sup>\*</sup> and Jakov SEKELJA<sup>\*\*</sup>

<sup>\*</sup> Upper Austrian University of Applied Sciences, Stelzhamerstrasse 23, 4600 Wels, Austria

<sup>\*\*</sup> FACC AG, Fischerstraße 9, 4910 Ried im Innkreis, Austria

**Abstract.** The porosity of carbon fibre-reinforced polymers is a very important topic for the practical applications of this material since there is a direct correlation between porosity and mechanical properties, such as shear strength. Therefore the porosity values of carbon fibre-reinforced composites for practical use in aircraft and cars must be lower than a certain level, usually 2.5-5 %. The most common non-destructive method for measuring 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. This can lead to significant errors in the determination of the porosity. Acid digestion and materialography are also used for porosity measurement, but both methods are destructive. This paper deals with the application of X-ray computed tomography for the characterization of carbon fibre-reinforced composites, in particular for the quantitative determination of porosity with a reasonable degree of accuracy. In order to attain this, different segmentation methods were applied, systematic computed tomography investigations on a broad variety of composite samples with different measurement parameters were performed and the results are compared with results obtained by standard porosity measurement methods, such as ultrasonic testing and acid digestion. In addition, the possibility of characterizing the size, shape and position of all individual pores and other material inhomogeneities in three dimensions are demonstrated.

## 1. Introduction

Carbon fibre-reinforced polymers (CFRP) have an increased stiffness and increased strength-to-weight ratio compared to “traditional” materials like metals. Therefore these materials are finding more and more use and becoming more and more important for modern industry, especially in the automotive and aviation industries [1]. Low porosity levels are essential for ensuring the performance of composite parts [2, 3]. There is a direct correlation between porosity and mechanical properties like compressive and interlaminar shear strength as well the elasticity modulus of the material. It has been found that the interlaminar shear strength decreases by 7 % per 1 % of porosity [2]. Usually, porosity values of composite components below 2.5 % and at certain circumstances up to 5 % are acceptable in the aviation industry.

In the literature there are a considerable number of studies of non-destructive and destructive methods for the determination of porosity values of composites [2-12]. These methods are:



- Ultrasonic attenuation [2-6]
- Acid digestion [2,7]
- Materialography – analysis by microscopy [5,6]
- Other methods like active thermography and microwave testing[2,8]
- X-ray computed tomography [3, 9-12]

The most important method among these is ultrasonic (US) attenuation, since there is a rather good and more or less linear correlation between porosity and the attenuation coefficient, if the voids are spherical and homogeneously distributed. Ultrasonic testing is a non-destructive (NDT) method, which can be easily used for quality control [2-4]. Thus, ultrasonic testing has a wide acceptance in research, manufacturing and maintenance applications for the determination of porosity and defect characterisation in composites. However, the ultrasonic attenuation coefficient is also affected by the size, shape and distribution of the voids. This can lead to considerable measurement errors in the range of  $\pm 25\%$  and higher [2,5,6].

Another method for porosity determination is acid digestion [2,7], which is destructive. It is based on gravimetry measuring volume and mass of the whole sample and the mass of the fibre content. For accurate results the material properties like resin and fibre density must be well known. Acid digestion is usually used as a reference method and not for quality control of real parts since it is destructive.

Another destructive method is materialography in combination with (microscopic) analysis.. In this case, slices of the sample are ground and analysed by two-dimensional image processing methods to determine the porosity. According to statistical theory, the volume porosity can be determined from a series of obtained area porosities. To get sufficiently accurate results, at least 30 optical areas have to be analysed. Therefore, this method is time-consuming and rather expensive [5,6]. In addition, technical difficulties, which reduce the accuracy, arise in practise. Due to an inhomogeneous distribution of pores, a limited number of analysed slices or smearing of small pores during the grinding process, the obtained porosity can be inaccurate [5,6].

Additional methods are active thermography and microwave testing [2,8], but these non-destructive methods are still topics of research and the feasibility for practical applications in industry is not yet clear.

Another very promising method for the non-destructive determination of porosity of composites is X-ray computed tomography (CT). Computed tomography is a radiographic NDT method of locating and obtaining the size and volumetric details in three dimensions [9-12]. Due to measurement speed and quality, CT systems with cone beam geometry and matrix detectors have gained general acceptance in materials science research as well as in the market for industrial CT systems. Using CT, a specimen is placed on a rotary plate between the X-ray source and the detector. The specimen is rotated step by step, taking a projection image at each angular position. A computer cluster reconstructs the radiographic images to a volume dataset. At each position of the resulting dataset a grey-value is calculated, which corresponds mainly to the spatial X-ray attenuation coefficient. The usual resolution range of cone beam CT systems goes down to  $1\ \mu\text{m}$  [10], which is enough to characterise pores larger than a few microns. However, the determination of real and accurate values for the porosity of CFRP parts is still a matter of research, since the accurate and reproducible determination of the surface between material and air has not yet been solved. Since porous samples have a rather large and very complex inner surface, the determined porosity values are strongly dependent on the segmentation method and parameters used. There have been a lot of studies [11, 13 and references therein] of different global and local segmentation methods, but no detailed investigations on the influence of the

segmentation method and parameters on the achieved porosity of CFRP materials have been published [3,11].

This paper deals with the application of CT for the characterization of CFRP and in particular with the determination of the porosity with a reasonable degree of accuracy and repeatability. In order to identify the most suitable segmentation method for obtaining accurate porosity values the following aspects are discussed:

1. The segmentation method should be as simple as possible.
2. The results must be feasible in a sense that the obtained air-material interfaces correspond to the unprocessed CT data.
3. The repeatability of the porosity values must be high.
4. The obtained porosity values must be consistent with values obtained using standard methods like ultrasonic testing and acid digestion.

## **2. Experimental**

### *2.1. Samples*

The samples were prepared by 20 layers of PREPREG C 970/PWC T300 3K UT (TY), which consists of carbon fibers and 40 % epoxy resin. The prepreg ply thickness was 0.21 mm and the curing temperature 177°C. In this way plates with about 170 x 200 mm and thicknesses between 4 and 5 mm were fabricated. The exact thickness strongly depends on the amount of porosity within the samples. By varying the vacuum pressure during the heating stage, porosity levels in the range between 0 and around 10 % were obtained. All in all, 8 different plates were manufactured. Out of these plates 9 different samples with a size of 17 x 20 x 4.5 mm<sup>3</sup> were investigated.

### *2.2. Porosity measurement methods*

#### *Ultrasonic testing*

For ultrasonic testing a commercial system was used. The measurements were performed using the pulse-echo (reflection) technique with a frequency of 5 MHz. The equipment consists of GE USM35 combined with Krautkrämer K5MN transducer. The transducer had a diameter of 6.35 mm and a delay line of 9 mm plexiglass. The coupling agent used was Exosen 30. As reference a part with the same thickness and 0 % porosity was used. Attenuations on the porosity parts were measured against the signal level on the reference part.

#### *Acid digestion*

The porosity measurement by acid digestion was performed in accordance with the procedure as described in [7]. The difference in the mass of the specimens before and after extraction of the resin by sulphuric acid digestion was measured. By using a physical fibre density of 1.75 g/cm<sup>3</sup> and a resin density of 1.26 g/cm<sup>3</sup>, the porosity values for the various samples were determined.

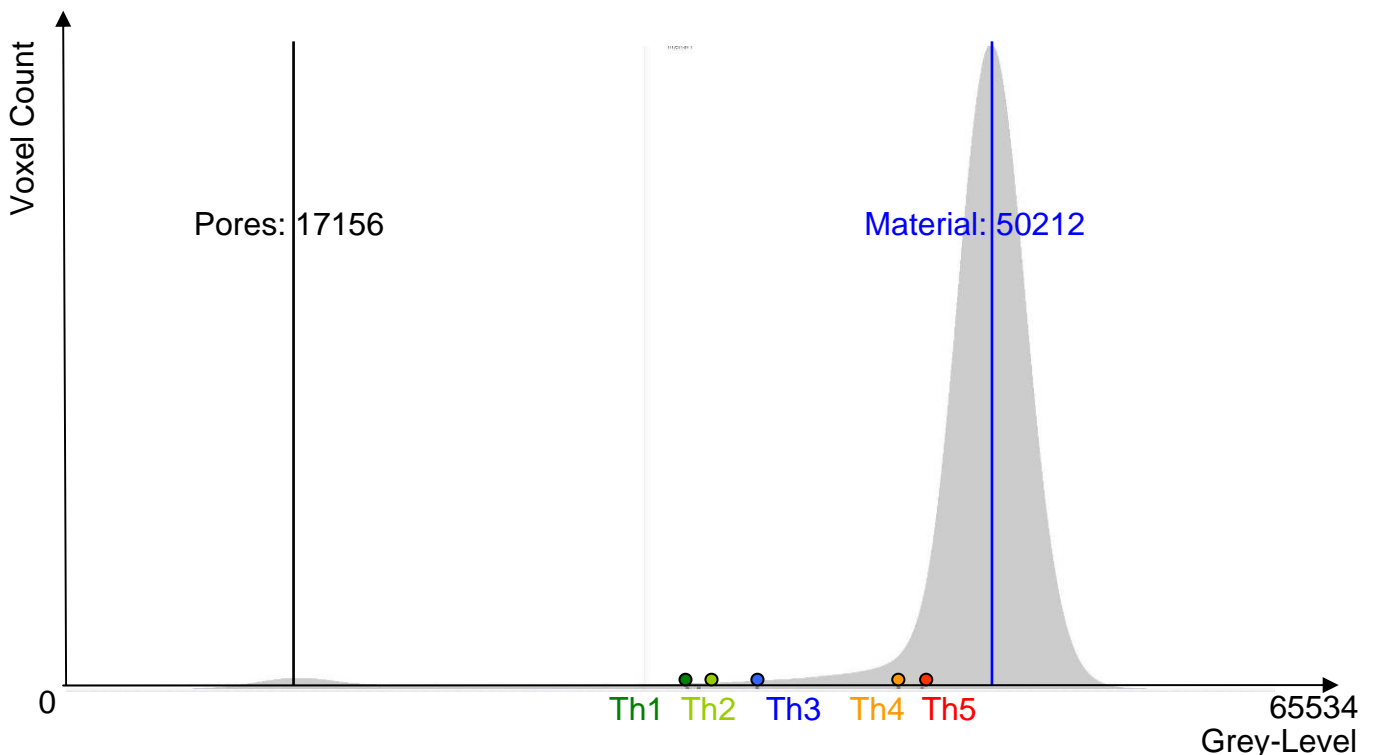
#### *X-ray computed tomography*

The X-ray tomograms were scanned using a Nanotom 180NF CT device constructed by GE phoenix/x-ray with a 180 kV nano focus tube and a 2300x2300 pixel Hamamatsu detector. The target used was made of molybdenum. Further details on the CT device can be found in [10]. The resolutions (voxel sizes) used were between 2.75 and 20 µm, the voltage at the

nano focus tube was 60 kV and the measurement current between 160 and 300  $\mu\text{A}$ . The CT data was reconstructed by using beam-hardening correction implemented in the Nanotom reconstruction software.

### 3. Evaluation of computed tomography data

For preprocessing an edge-preserving anisotropic diffusion filter [11] for reducing noise without blurring edges in the dataset was applied. In the next step a volume of interest without the ambient air was defined. To keep the evaluation procedure as simple as possible for segmentation only global thresholding methods, which are based on an evaluation of the grey value histogram, were applied. The grey value histogram together with the position of the different thresholds for a typical composite sample with a porosity value around 4-5% is shown in Fig. 1. There is a bimodal distribution of the grey values showing two clearly distinguishable nearly Gaussian peaks one for the air/pores and one for the material. The lowest threshold investigated was calculated by VG Studio Max, 2.0 [14] within the calibration procedure. Using a method described by Otsu [13] the calculated threshold revealed a slightly higher value. Threshold 4 was obtained by drawing a line between the air peak and material peak in the histogram and calculating the maximum distance between this line and the grey values. Threshold 5 was determined by calculating the tangent at the turning point of the material peak. Threshold 3 was obtained by a balanced combination of threshold 2 and threshold 4 (73 % threshold 2 and 27 % threshold 4). A summary of the different threshold values can be found in Table 1.



**Fig 1.** Grey value histogram and corresponding threshold values of a typical composite sample with a porosity around 4.5 %. CT measurement parameters: 60 kV, 160  $\mu\text{A}$ ,  $(10 \mu\text{m})^3$ .

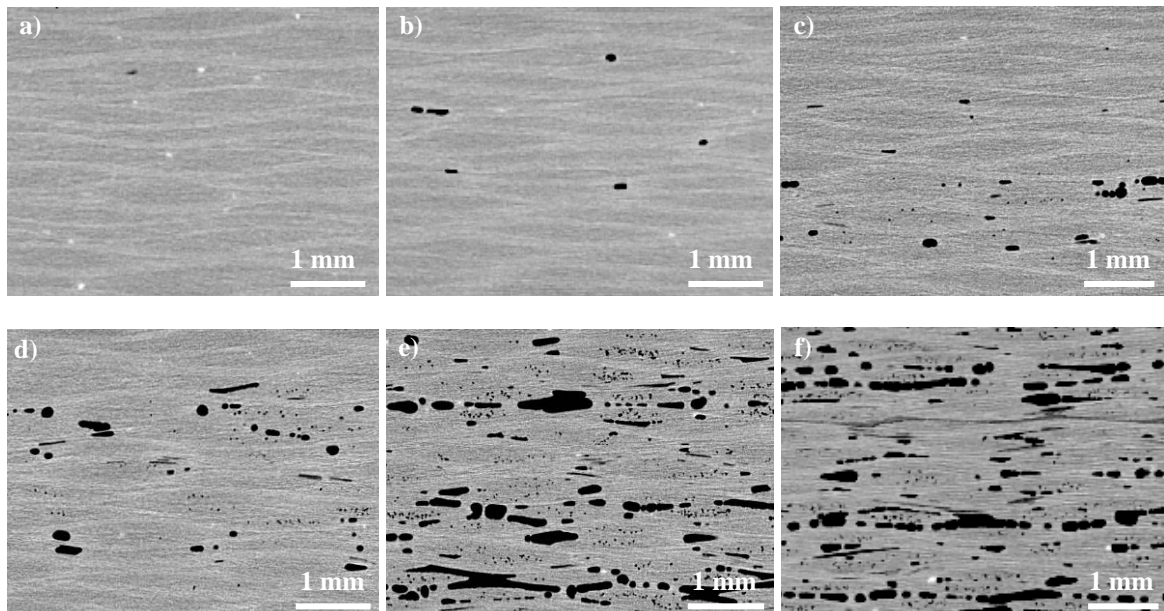
**Table 1.** Description of different thresholding methods for porosity determination together with the corresponding threshold values and porosity numbers for the sample from Fig.2.

Method	Description	Threshold values from Fig.2	Porosity of sample from Fig. 2 (%)
Th1-VG	Calibrated threshold by VG2.0	35583	3.81
Th2-Otsu	Threshold obtained by Otsu-method	36433	3.97
Th3-Final	Final Threshold (73% Th2 and 27% Th4)	38996	4.59
Th4-Histo	Threshold based on a maximum distance of the bimodal histogram	45925	8.36
Th5-Tangent	Tangent threshold	47086	10.42

## 4. Results

### 4.1 Computed tomography results of samples with different porosity values

Fig. 2 shows cross-sectional CT-images of carbon reinforced composite samples with porosity values between 0 and 10 vol-%. The size, shape and position of the pores can be observed in all pictures. In the low-porosity samples the pores are mostly spherical, whereas in the samples with porosity higher than 3 % the pores become flat and larger. In addition, the pores are not distributed homogeneously. At certain layers there are many pores and at certain layers there are almost no pores. This can be clearly seen and is demonstrated by the CT-results.

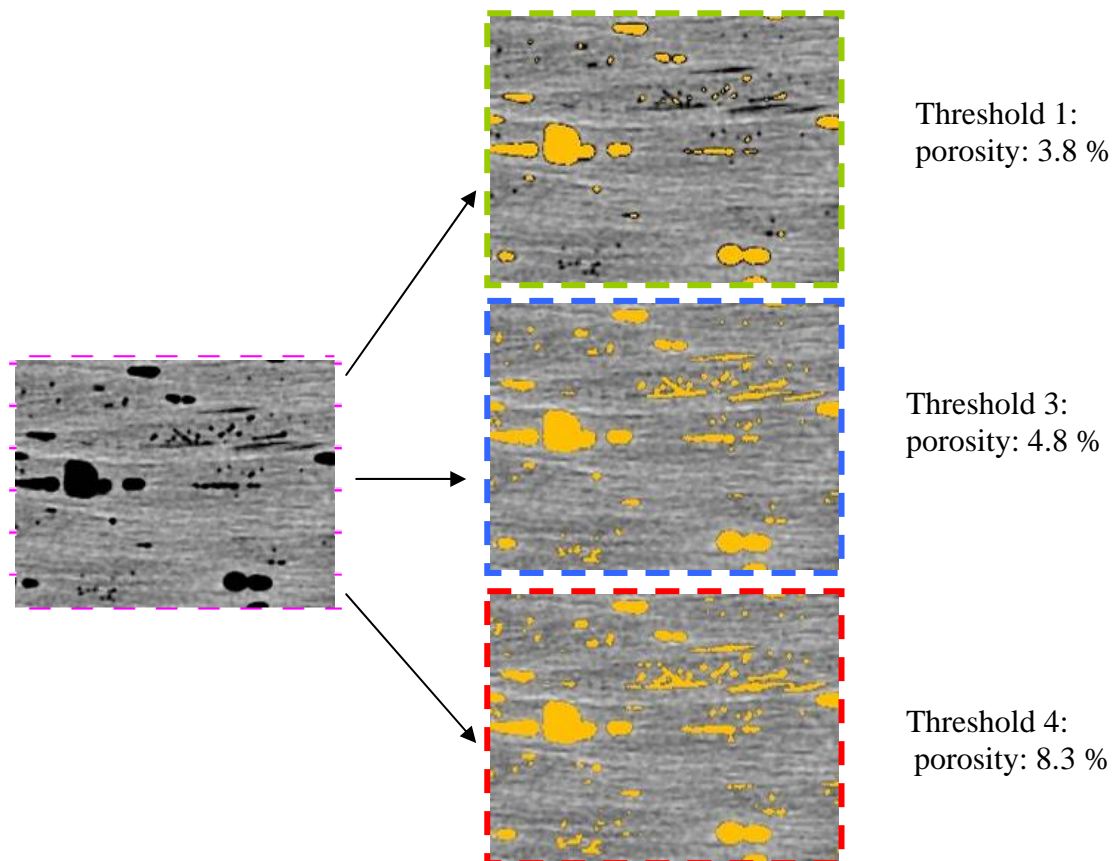


**Fig 2.** CT-cross-sectional images of typical carbon reinforced composite samples (a: ~0 vol-%, b: ~0,15%, c: ~0.35 %, d: ~0.9 %, e: ~4.50 %, f: ~10 %). The CT-measurements were performed with 60 kV, 160  $\mu$ A and voxel size  $(10 \mu\text{m})^3$ .



#### 4.2 Investigations using different threshold values

In Fig. 3 the influence of the various threshold values on the segmentation and on the obtained porosity numbers is demonstrated. For this reason a CT-data set for a sample with a porosity around 5 % is processed by different threshold values. The segmented pores are depicted in yellow in Fig. 3. With threshold 1 (and also 2) an under-segmentation can be observed, especially of small pores which are not segmented. Threshold 4 (and also 5) leads to an over-segmentation; also some areas of the resin are segmented. The result of threshold 3 seems to be most plausible and most accurate.



**Fig 3.** Influence of various thresholds on the porosity evaluation. The left picture is the original cross-sectional CT picture. The three pictures in the centre are CT slices together with segmented pores, which are depicted in yellow.

#### 4.3 Investigations of repeatability

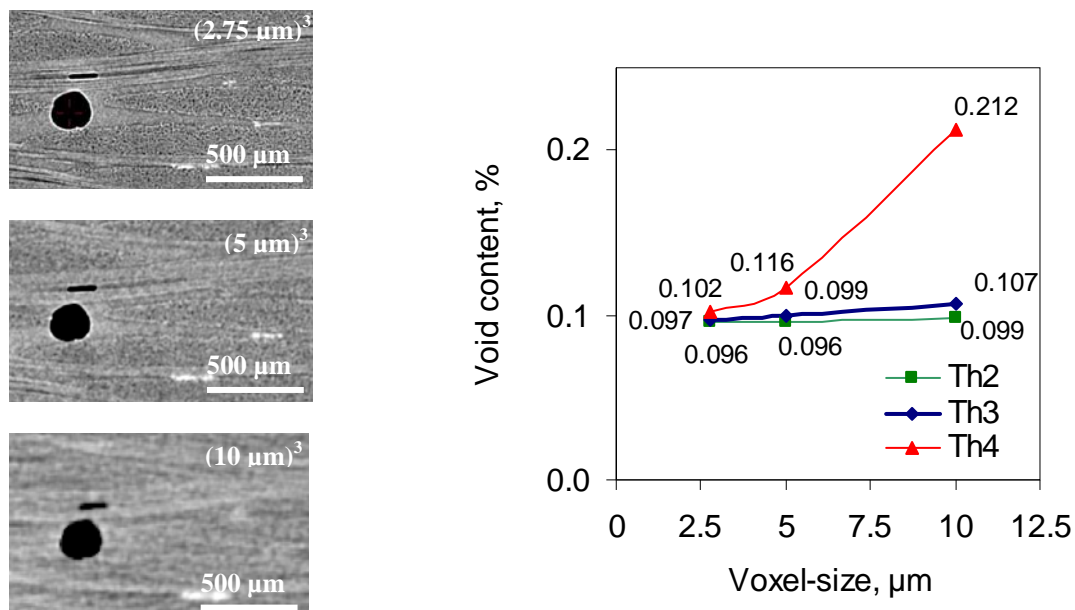
In order to make the results as reproducible as possible 4 CT measurements were performed on a sample with the same measurement parameters (tube voltage=60 kV, tube current=160  $\mu$ A, integration-time=1000 ms, voxelsize=10  $\mu$ m, number of projections=1500). The absolute standard deviations of the porosity numbers obtained by various segmentation methods are summarized in Table 2. As shown, the repeatability is quite good using the methods Th1-VG, Th2-Otsu and Th3-Final, whereas the repeatability using Th4-Histo and Th5-Tangent is much worse. All in all, the repeatability for the computed tomography method seems to be much better when compared to ultrasonic testing, where reported values are in the area of around 0.5 % [2,4-6].

**Table 2.** Repeatability of porosity values determined by different thresholds applied to the same CT data.

Method	Mean Porosity [%]	Absolute standard deviation of the porosity (percentage point)
Th1-VG	3.81	0.028
Th2-Otsu	3.97	0.031
Th3-Final	4.6	0.043
Th4-Histo	8.4	0.283
Th5-Tangent	10.42	0.459

#### 4.4 Investigations using different resolutions/voxel sizes

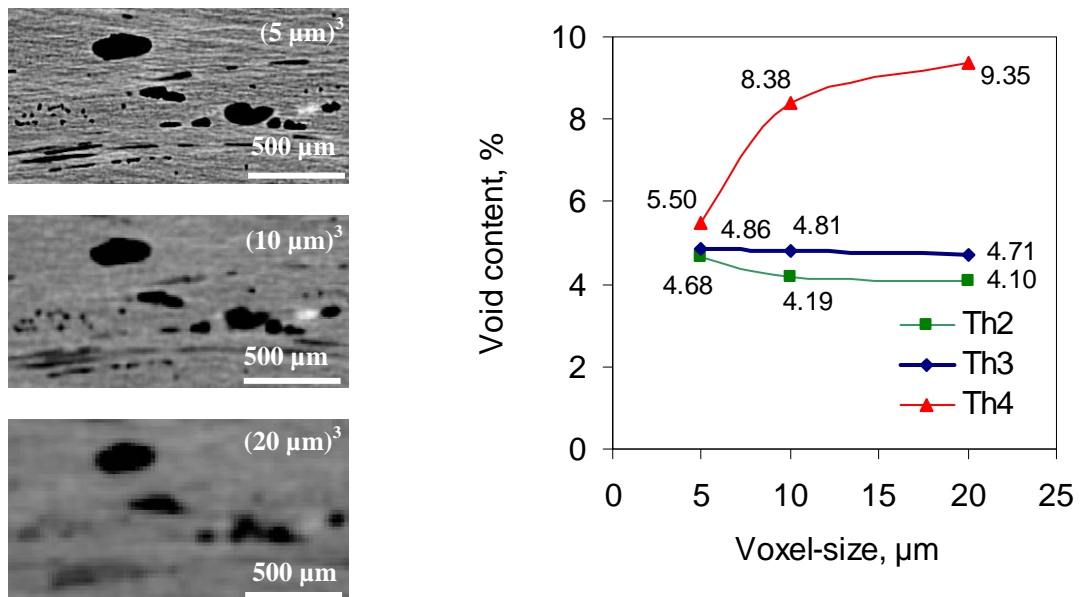
The influence of the CT measurement parameters, such as resolution/voxel size, on the obtained porosity should be as low as possible. Therefore CT measurements with different resolutions on a sample with a rather low porosity (around 0.1 %) and on a sample with a rather high porosity (around 5 %) were performed and analysed. In Fig. 4 & 5 on the left side cross-sectional CT pictures measured with voxel sizes between 2.75 and 20  $\mu\text{m}$  are shown.



**Fig. 4.** Left pictures: CT cross-sectional images of a CFRP sample measured with voxel-sizes between 2.75 and 10  $\mu\text{m}$ . Other measurement parameters were kept constant (voltage 60 kV and number of projections 1500). Right picture: Influence of CT voxel size on the determined porosity evaluated by different segmentation methods. The minimum defect volume was kept constant.

By improving the voxel size, the sharpness of the CT data improves and more and more details in the sample can be recognised. In the right picture of Fig. 4 & 5 the porosity obtained by the segmentation methods Th2, Th3 and Th4 versus the voxel size is shown. It can be seen, that the porosity obtained by Th2 and Th4 strongly depends on the voxel size,

whereas the porosity values obtained by method Th3 are almost independent of the voxel size. These results indicate that the method Th3 seems to give the most accurate porosity value.



**Fig. 5.** Left pictures: CT cross-sectional images of a CFRP sample measured with voxel-sizes between 5 and 20  $\mu\text{m}$ . Other measurement parameters were kept constant (voltage 60 kV, number of projections 1500). Right picture: Influence of CT voxel size on the determined porosity evaluated by different segmentation methods. The minimum defect volume was kept constant.

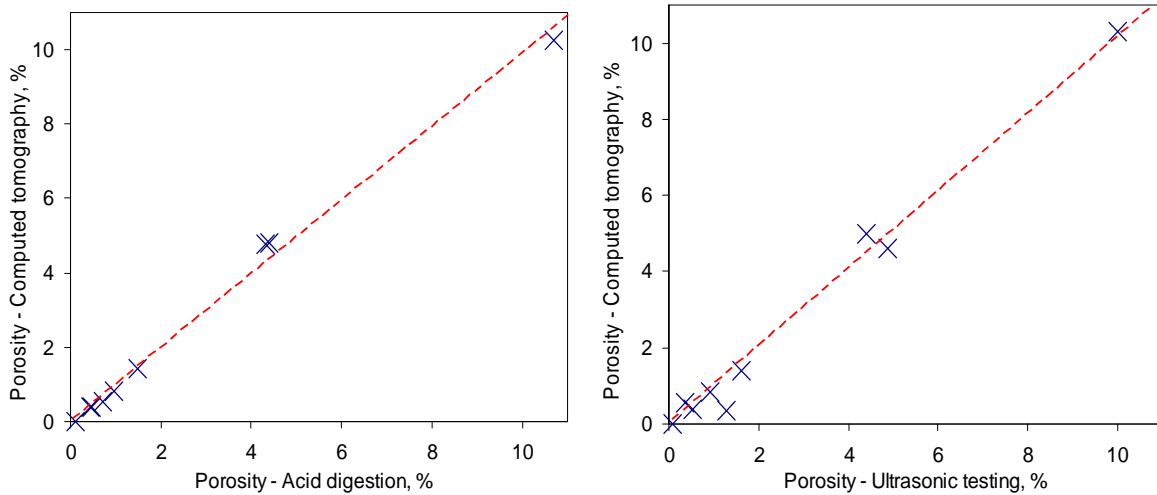
In addition, CT investigations with slightly varying currents and integration times on the detector were also performed. No significant influence on the obtained porosity values was detectable.

#### 4.5 Comparison of porosity values obtained by computed tomography, by acid digestion and ultrasonic testing

For comparison the same samples characterised by CT were analysed by acid digestion and the obtained porosity values compared. The correlation diagram is shown in Fig. 6 (a), where the porosity obtained by CT using threshold 3 and the porosity obtained by acid digestion are plotted. There is a rather good correspondence revealing a correlation factor of 0.993.

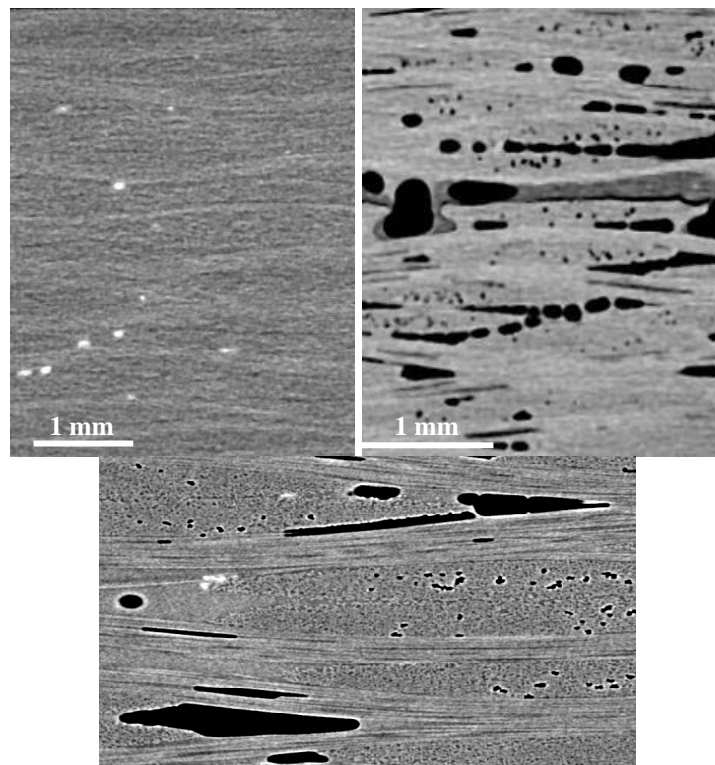
For comparison, samples characterised by CT were also analysed by ultrasonic testing and the obtained porosity values were compared. In Fig. 6 (b) the correlation diagram is plotted, where the porosity obtained by CT versus the porosity obtained by ultrasonic testing are shown. The correspondence is nearly as accurate as for the acid digestion CT diagram, since the correlation factor is 0.984.





**Fig 6.** (a) Comparison of porosity values determined by computed tomography and acid digestion. The CT data was evaluated by using threshold 3 for segmentation. The dashed line show the trend, which corresponds to  $y = 0.990 x$  and the correlation factor is 0.993. (b) Comparison of porosity values determined by computed tomography and ultrasonic testing. The slashed trend line corresponds to  $y = 1.017 x$  and the correlation factor is 0.984.

#### 4.6 Further inhomogeneities and features detectable by computed tomography



**Fig 7.** Left picture: cross-sectional CT picture of particles with a density higher than the matrix. Middle picture: cross-sectional CT picture of voids filled with resin. Right picture: High-resolution cross-sectional CT picture showing individual fibres, fibre bundles and micro-porosities. CT measurement parameters: 60 kV, 160  $\mu\text{A}$ ,  $(10 \mu\text{m})^3$  (left and central picture) and 60 kV, 240  $\mu\text{A}$ ,  $(2.75 \mu\text{m})^3$  (right picture).

CT can be used to not only distinguish between air (pores) and material, but also other inhomogeneities and features within the composite specimen are detectable. Fig. 7 shows cross-sectional CT pictures of selected specimen areas. In the left picture, particles with a higher density than the resin are noticeable. In the middle picture a pore partly filled with resin can be seen. As this example shows, the CT contrast is high enough to distinguish resin regions without carbon fibres and resin regions filled with carbon fibres. In the right picture in Fig. 7 micro pores within the fibre-bundles and larger pores in the resin are visible. The larger pores are mainly located between the crossing points of the fibre bundles. In addition, the spatial resolution of 2.75  $\mu\text{m}$  is high enough, so that even single carbon fibres are detectable by CT. By using a higher CT resolution of around and below 1  $\mu\text{m}$  the carbon fibres and the polymer matrix are even better distinguishable from each other [10]. In addition CT can be also used to detect cracks and delaminations within composite samples [3, 10].

## 5. Discussion

Cone beam X-ray computed tomography was used to characterize composite samples and a method for applying CT for the quantitative measurement of porosity developed. In order to determine the porosity of carbon fibre reinforced polymers using CT no material parameters, such as the exact density of the polymer and the fibres or the velocity of sound in the material, are needed. In addition, the determined porosity values do not depend on the shape and distribution of the pores as is common in ultrasonic attenuation [2,4]. However, the biggest challenge for the application of CT for porosity determination is the exact and reproducible detection of the interface between air/pores and the material. Various segmentation methods based on global thresholding and evaluating the grey value histogram were investigated for this paper. The best method proved to be a combination of the well-known Otsu-threshold and a threshold based on a maximum distance of the material peak within the bimodal grey-value histogram, to be precise 73 % Otsu-threshold and 27 % the latter thresholding method. The optimal method was derived in a way so that the following requirements are fulfilled:

1. The segmentation method is simple.
2. The segmented pores correspond well with the pores visible within the unprocessed CT dataset.
3. The repeatability of the porosity value is high. The value found for four measurements of the 4.6 %-porosity sample was  $\pm 0.043$  percentage point (=0.9 % relative value).
4. The dependency of the derived porosity values on the CT measurement parameters is rather low. CT investigations with different resolutions/voxel sizes show a maximum change of 0.15 percentage point (=3.3 % relative change) in the porosity depending on the absolute value of the porosity. Changing other CT measurement parameters revealed an even smaller dependency.
5. The porosity values are consistent with values obtained using standard methods. This was proved by investigating the same samples by CT, ultrasonic testing and acid digestion. The correlation factor of porosity values obtained using the CT method with porosity values obtained by acid digestion is 0.993 and the correlation factor with ultrasonic testing is 0.983.

The results presented indicate that the CT porosity measurement method proposed here has a repeatability around 0.04 percentage point (=0.9 % relative value). These values are much better as published for ultrasonic testing [2,4,5], acid digestion[2,7] and

materialography – analysis by microscopy [5,6]. The repeatability values reported for these methods are in the area of around 0.5 percentage point. The accuracy of the presented method is hard to determine, since there is no accurate reference method. The investigations presented here indicate an accuracy better than 0.5 %-points (=5-10 % relative accuracy) based on the following findings:

- On average the porosity values determined by CT don't differ more than 0.3 %-points from the porosity values obtained by ultrasonic testing and acid digestion.
- The method presented here does not strongly depend on the CT-measurement parameters. The changes of the porosity values with CT-measurement parameters (like resolution) are below 0.15 percentage point.

Although this is only a rough estimation and further investigations for supporting the conclusions presented here are necessary the advantages of computed tomography for porosity determination are evident and clear.

In addition, computed tomography has some further advantages. The volume of every pore and of every inhomogeneity can be characterised by its size, shape and position, as well as defects, such as inclusions with a higher density and fibres, can be observed and analysed by computed tomography. Table 3 shows a comparison of the most important methods for the determination of the porosity of carbon reinforced composites.

**Table 3.** Comparison of methods (ultrasonic testing, acid digestion, materialography and computed tomography) used for porosity determination of composites.

Method	Measurement principle	Accuracy in percentage points	Replicability	Time	Costs	Limitations	
Ultrasonic testing	Absorption of ultrasonic waves	$\pm 1$ % (if the material is well known $\pm 0.5$ %)	o	+	+	Influence of pore-geometry and orientation, sample-geometry	non-destructive
Acid digestion	Gravimetry	$\pm 1$ % (if the material is well known $\pm 0.5$ %)	destructive	-	o	Influence of analysed volume and material properties	destructive
Materialography - microscopy	Analysis of optical pictures	$\pm 1$ %	destructive	+ ... - (Depending on number of micro sections)	+ ... - (Depending on number of micro sections)	Influence of grinding, small analyse volume	destructive
Computed Tomography	Absorption of X-Rays	<0.5 %	+	o	-	Samples must be small for a good resolution	non-destructive

## 6. Conclusion

X-ray computed tomography is a powerful non-destructive testing tool for characterizing and, in particular, for measuring the volumetric porosity of carbon-fibre reinforced composites. We have presented a segmentation method based on global thresholding 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. Moreover, we have demonstrated that CT is not limited to measuring the porosity. Using

CT, the size, shape and position of the pores as well as other defects, such as inclusions with a higher density or filled pores, can also be observed. These results show, that CT can be used in science and industry as:

- an alternative method for the non-destructive measurement of porosity in composites,
- a method which gives additional information about the inner structure of the porous composite material like size, shape and position of the pores as well as impurities and other inhomogeneities
- a reference method for established methods

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