# Evaluation of a Testpiece for Porosity in Carbon Fibre Reinforced Polymers

Johann KASTNER [1], Bernhard PLANK [1], Guruprasad RAO [1]
[1]University of Applied Sciences Upper Austria, Wels, Austria
Phone: +43 50804 44440, Fax +43 50804 944440

Contact e-mail: johann.kastner@fh-ooe.at

**Abstract.** Because of their outstanding high density specific strength carbon fiber-reinforced polymers (CFRP) are the favourite material for many lightweight applications, especially in the aeronautic and automotive industry. However, defects like pores in CFRP cannot be completely avoided during manufacturing. Therefore, reliable and reproducible methods for porosity and defect detection are necessary. Common methods are ultrasonic testing and destructive methods like acid digestion and serial sectioning. All these methods lead to an uncertainty of ±1 vol.% in porosity or even worse. X-ray computed tomography (CT) is another promising method for porosity detection, because it is non-destructive, it is a 3D-method giving also the morphology of all pores and it has a very good reproducibility of around 0.04 vol%.

However, up to now there are no CFRP test parts available with a well-defined porosity which can be used as reference parts for CT. Therefore, we propose a new CFRP-porosity test part for X-ray CT. CFRP-reference plates were made by drilling 300 - 1000 holes with a diameter between 200 to 300 µm in plates with a thickness of 1 mm. The diameters were evaluated by optical microscopy and CT to get reference porosity values and the corresponding uncertainty taking into account also machining induced diameter variations especially near the surface of the drilling hole. These hole-plates were mounted and pressed with porosity free 1 mm CFRP plates at the top and at the bottom. 3 samples with porosity values of 0.96 ± 0.02 vol. %, 2.58 ± 0.03 vol. %, and 4.81 ± 0.12 vol. % were prepared. These test parts were scanned by X-ray computed tomography and different data processing methods were applied to get accurate porosity values. These methods are evaluated and discussed within this contribution.

## 1. Introduction

To reduce transportation costs and weight, many industries, such as the aviation- and automotive industries, develop and optimize new kinds of material systems in the field of composites. Nowadays, many safety-critical structures are manufactured from carbon fibre reinforced polymers (CFRP) [1-3]. A main problem here is that porosity can critically weaken the material strength. The voids in CFRP have been the major reasons for damage in high stress environment. The effects of voids (pores / defects) on structural properties have been studied extensively and concluded that manufacturing defects can severely deteriorate matrix dominated properties of composites, resulting in degraded strength and fatigue of structural performance [2,3].

The porosity in CFRP-materials is mainly evaluated using two kinds of methods; namely destructive methods and non-destructive methods. Methods such as acid digestion, materialography in combination with optical microscopy are destructive methods whereas active thermography, ultrasonic testing and X-ray computed tomography (CT) are non-destructive methods [1-3]. However, the determination of the accurate porosity value requires a careful measurement and data evaluation procedure. For porosity determination in CFRP by CT there were two methods published, one by the Upper Austrian University of Applied Sciences in 2010 [1] and another one by Airbus in 2011 [2]. Both methods give accurate porosity values for a certain material combination if carefully applied which correspond well to the values measured by ultrasonic testing and other standard methods. However, up to now there are no CFRP test parts available with a well-defined porosity which can be used as reference parts for destructive and non-destructive porosity measurement methods [1-6].

Therefore, we propose a new CFRP-porosity test part for X-ray CT and other methods for porosity measurement. CFRP-reference plates were made by drilling micro holes. The diameters were carefully measured by optical microscopy and CT to get reference porosity values. These hole-plates were mounted and pressed with porosity free CFRP plates at the top and at bottom. These test parts were scanned by X-ray computed and porosity values were determined from the CT-data.

## 2. Materials and Methods

### *2.1 Investigated materials*

For reference porosity samples we used plates with 5 ply of PREPREG C 970/PWC T300 3K UT (TY) in plain weave style. In the case of a non-porous material it consists of 60 wt. % carbon fibres and 40 wt. % epoxy resin. The prepreg ply thickness was 0.216 mm. A schematic picture showing the geometry of the samples is shown in Fig. 1 (a) together with a photographic picture of a typical sample (Fig.1 (b)). The drilling process was carried out by Apex Engineering in Vienna by using drillers with a diameter of 200 µm and 300 µm as shown on the left picture in Fig. 2.

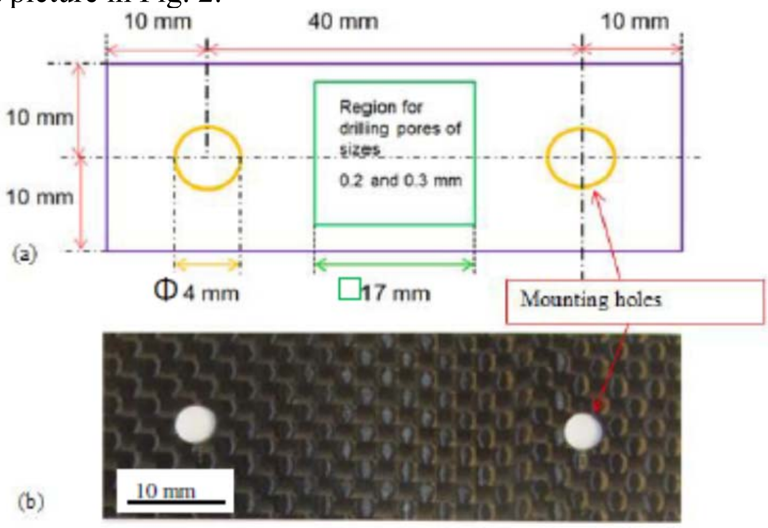


**Figure 1**. Schematic of dimensions of the drilled CFRP plates (a) and photographic picture of a typical CFRP-plate (b). The thicknesses of the CFRP plates are around 1 mm. The drilled CFRP-plates were mounted together by screwing from the front and back side through the mounting holes.

### *2.2 Measurement methods*

The individual plate thickness and the thickness of each combination were measured at five different points using a screw gauge (Preisser Messtechnik, Germany). Drill hole diameters were measured by optical microscopy (LOM) at a magnification of 50x with an Olympus microscope (BX51). Each drill hole was measured from one side of the plates by using manual 3-point circle fitting. Measurements and other functions are performed using PicEd-software which has tools to capture images and measure the objects by fitting regular shapes.

In addition to microscopy, CT scans on the centred region of the plates were performed with (5 µm)$^3$ voxel size for measuring the diameters by fitting a cylinder. CT scans were performed on a Nanotom 180 NF device manufactured by GE phoenix|x-ray. The device uses a 180 keV nano-focus tube and a full digital 2304$^2$ pixel flat panel detector (Hamamatsu). Molybdenum was used as target material. Details of the CT-systems can be found in [7]. No pre- or post-filters were used for the scans. Applied voltage on the X-ray tube was 60 kV at a voxel size between (2.75 µm)$^3$ and (120 µm)$^3$. Voxel sizes were calibrated by a 3.9796 ±0.0020 mm ball bar manufactured by GE. CT data processing and evaluation was done in VGStudio Max 2.2 by Volume Graphics. Thresholds were calculated in our in-house tool iAnalyse developed by the University of Applied Sciences Upper Austria [1,3] and by the Airbus method [2].

## 3. Results and Discussion

### *3.1 Fabrication of the CFRP-reference porosity samples*

To create a porosity reference sample with closed holes different combinations of the drilled plates (D1 to D3) and undrilled plates (N0) were joined as shown in the right picture of Fig. 2.

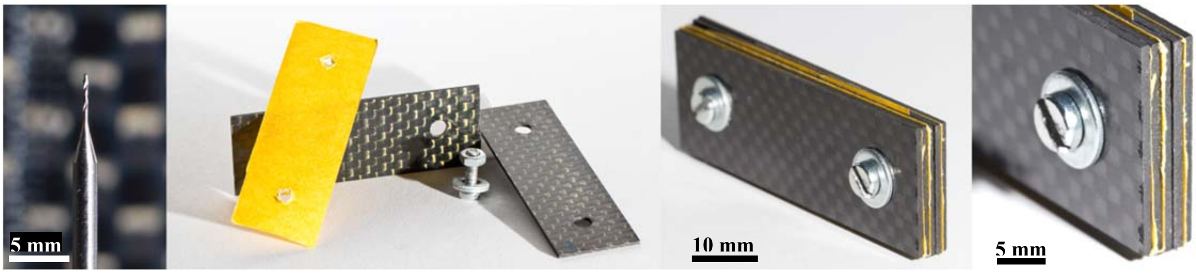


**Figure 2.** Overview of fabrication the reference porosity samples. Artificial holes were fabricated by micro-drilling (left picture). Thee drilled CFRP-plates were mounted together by screwing from the front and back side (right picture).

Before combining samples in various combinations CT-scans were performed. If an air gap was observed there was a need to eliminate the same by using filler materials as interlayers. Typical CT-scans are presented in Figure 3. In Fig. 3 (a) no filler material is used which leads to significant air gaps, whereas in Fig. 3 (b) a plastic interlayer is used, which reduces the air gaps strongly.

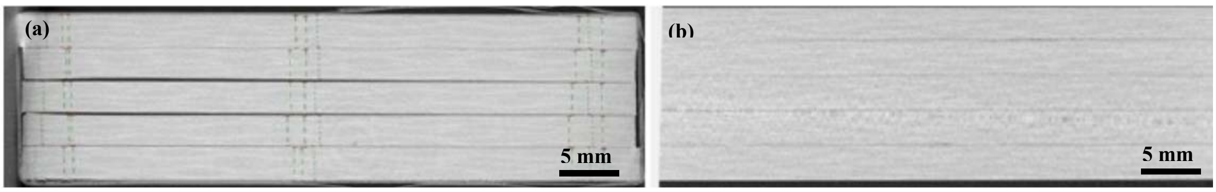


**Figure 3**. CT-slices of mounted CFRP-plates: In (a) air gaps are visible, in (b) air gaps are almost completely removed by using a plastic interlayer and applying a proper mounting.

Fig. 4 shows an overview of one undrilled plate (bottom left) and three samples containing different drill holes: Sample D2 with 0.3 mm drill holes (green), sample D1 with 0.2 mm drill holes (red) and samples D3 with 0.2 mm and 0.3 mm drill holes (yellow). The number and the arrangement of drill holes for the samples D1, D2 and D3 are summarized in Table 1.

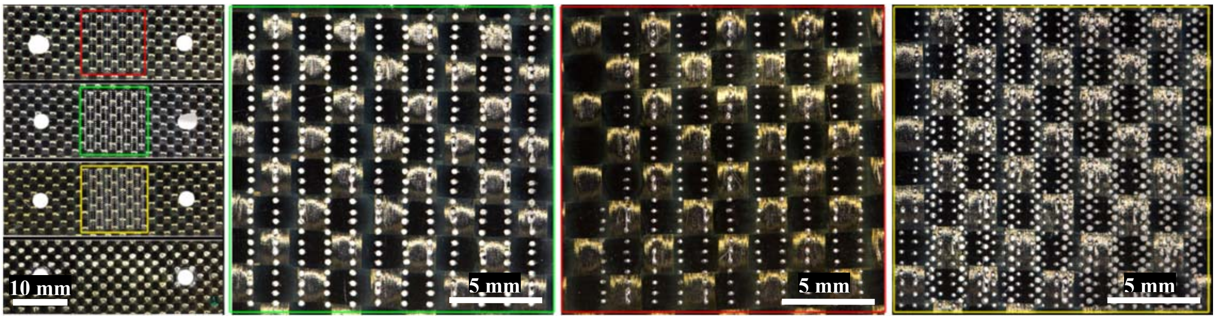


**Figure 4.** Overview of drilled and undrilled plates (left) and three samples containing different number of drill holes with different diameters. Detail of the sample D2 with 0.3 mm drill holes (green), detail of the sample D1 with 0.2 mm drill holes (red) and detail of samples D3 with 0.2 mm and 0.3 mm drill holes (yellow).

**Table 1.** Number of drilled holes and drill diameters for analysis

| **Sample** | **Arrangement (columns × rows)** | **Total** | **Drill diameter (µm)** |
|---|---|---|---|
| Sample D1 | 29× 10 + 10× 1 | 300 | 200 |
| Sample D2 | 29× 12 + 20× 1 | 350 | 300 |
| Sample D3 big holes | 29× 10 + 20× 1 | 310 | 300 |
| small holes | 30× 23 + 10× 1 | 710 | 200 |

### *3.2 Determination of Reference Porosity Values*

To assess the void volume correctly it is requisite to evaluate diameters of the drilled holes accurately. The diameters of the drilled holes were measured by using light optical microscopy and X-ray computed tomography.

(1) Light optical microscopy (LOM)
The measurement of the diameters of the drill holes by light optical microscopy was not accurate enough because there were too many breakouts and differences between the front and back sides of the plates as can be seen in Figure 5. Maximum deviation between front and back were in a range of ±8.24 µm for the 200 µm drill holes. Deviation of individual drill holes measured on the same side was between ±7 µm and ±13 µm for both diameter types.

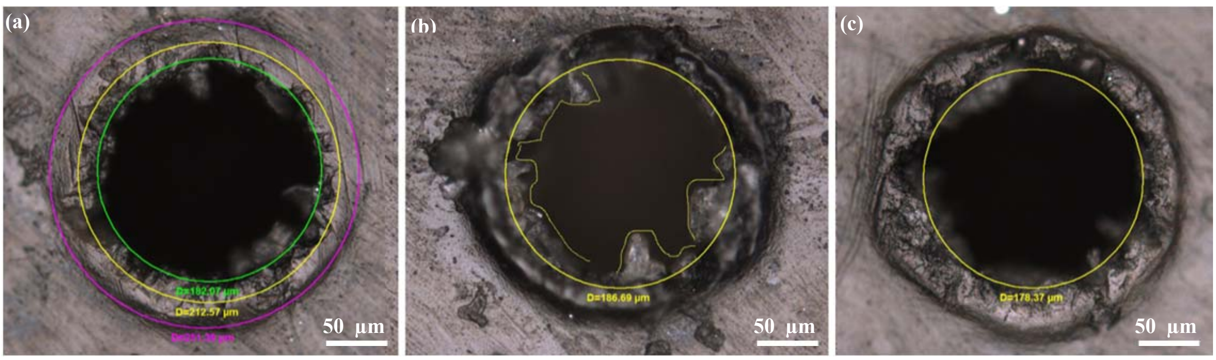


**Figure 5**. Delamination visualised under microscope (a) area of impact (b) ingrowth of CFRP in void region (c) outgrowth of CFRP around drilled region.

(2) X-ray computed tomography (CT) and subsequent cylinder fitting

The 3D-cylinder-fit tool of VG Studio Max was used to fit three cylinders on every drill hole within the CT-data. The investigated regions were defined at the front- and at the back-side of the plates as well the total length of the drill hole as shown in Figure 6. This method leads to much more accurate results than measurement by light optical microscopy.

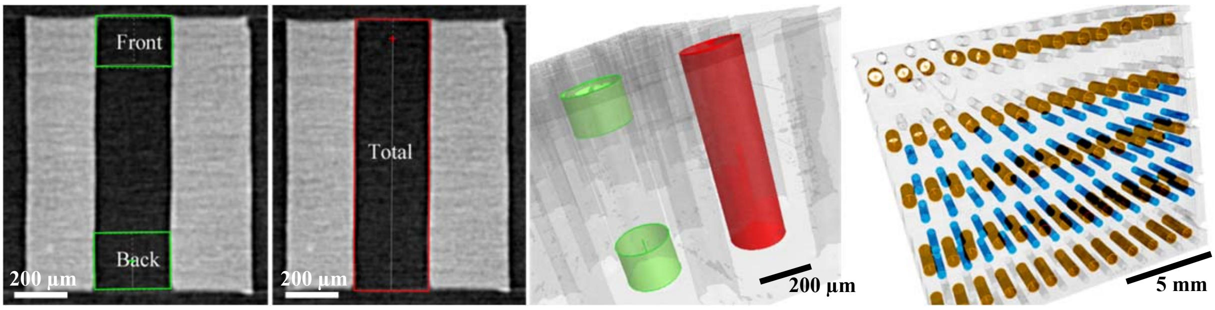


**Figure 6**. Cylinder fit within the CT-data of drill hole, front/ back (green) and total (red) to obtain the diameter of the drill holes accurately. All 65 fitted cylinders of Sample D3 are shown in the right figure, in orange 0.3 mm and in blue 0.2 mm holes. The voxel size of the CT-measurement was $(5\ \mu m)^3$.

Finally high resolution scans at $(5\ \mu m)^3$ were performed and cylinders were fitted for 65 drill holes per sample plate to get a mean diameter and deviation. For comparison of the front and back side results, cylinders with only 0.2 mm in depth were evaluated for each side by CT. In Table 2 the evaluated mean diameters are shown. Highest deviation is obvious for Plate D1 between the front and back side of the plates evaluated by LOM. Evaluation by CT shows only small differences in the range of maximum ±1.45 µm between front and back side. For final porosity estimation the diameters of the entire cylinders measured by CT are used.

**Table 2.** Results of the diameter evaluations of the drill holes of plates D1, D2 and D3 measured by light optical microscopy and X-ray computed tomography.

| **Plate** | **Mean diameter LOM [µm]** | | **Mean diameter CT [µm]** | | |
|---|---|---|---|---|---|
| | **Front** | **Back** | **Front (0.2 mm)** | **Back (0.2 mm)** | **Entire cylinder** |
| D1 | 183.76 ±10.14 | 167.32 ±13.16 | 193.45 ±1.85 | 192.79 ±2.75 | 192.69 ±0.59 |
| D2 | 284.23 ±6.85 | 284.68 ±7.26 | 292.87 ±1.36 | 293.62 ±1.03 | 293.6 ±0.83 |
| D3 small holes | 189.24 ±9.18 | 184.67 ±7.38 | 195.09 ±1.29 | 196.34 ±0.88 | 196.05 ±0.92 |
| D3 big holes | 278.97 ±8.00 | 287.25 ±7.00 | 291.09 ±1.20 | 294.00 ±1.44 | 293.37 ±0.89 |

The overall deviation for the diameters used for porosity calculation was calculated by taking into account the standard deviations from front, back, and the entire cylinder of each plate and drill hole type. Together with the thickness of the individual plates, the mean diameter and amount of drill holes a total void volume for each plate was calculated.

To create a porosity reference sample with closed holes three different combinations (S1, S2 and S3) of the drilled plates (D1 to D3) and undrilled plates (N0) were joined. Since the thicknesses of the plates, the plastic interlayer and diameters of the drill holes are known the porosity for each plate combination can be determined quite accurately. In Figure 7 CT images of one combination with an expected porosity of 4.81 vol. % is shown. The porosity values for various combinations of the drilled CFRP-plates are summarized in Table 3.

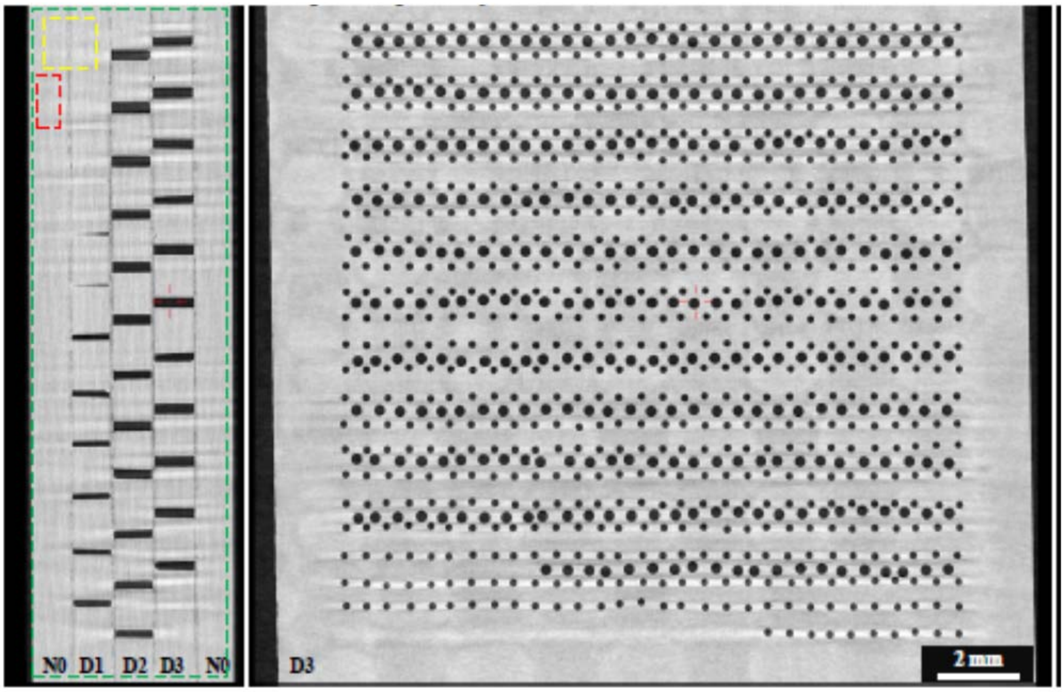

**Figure 7.** CT-slice images of combination S3 of different plates (N0 - D1 - D2 - D3 – N0) to create a porosity reference sample with an expected porosity of 4.81 vol. % (Adapted from ref. [5]).

**Table 3.** Porosity values for various combinations of the drilled CFRP-plates.

| **Sample** | **Plates** | **Porosity values in %** |
|---|---|---|
| S1 | N0-D1-N0 | 0.96 ± 0.02 |
| S2 | N0-D2-N0 | 2.58 ± 0.03 |
| S3 | N0-D1-D2-D3-N0 | 4.81 ± 0.12 |

### *3.2 Measurement of porosity by X-ray computed tomography*

The fabricated reference samples S1, S2 and S3 for porosity were scanned by X-ray computed tomography with a voxel size of (11 µm)$^3$. This voxel value was chosen since the scanned volume is maximized and the porosity measurement by CT gives accurate values [3]. The Airbus adapted threshold was used for evaluation the porosity of the artificial porosity samples using the same principle as Airbus threshold but the region of interest for evaluating the threshold was adapted and includes CFRP resin, fibre, and filler material between the air-gap [2, 5]. The results are summarized in Table 4. The agreement between the porosity values determined by measuring the diameters (volumes) of the drill holes multiplied by number of holes and the porosity values determined from the CT-data of the whole samples is very high. The mean deviation is 0.05 %-points and 2.5 %, which is rather low.

**Table 4.** Porosity values for various combinations of the drilled CFRP-plates.

| **Sample** | **Porosity in % determined by hole diameter measurement** | **Porosity in % measured by CT** | **Deviation in %-points** | **Deviation in relative %** |
|---|---|---|---|---|
| S1 | 0.96 ± 0.02 | 0.92 | -0.04 | -4 |
| S2 | 2.58 ± 0.03 | 2.63 | +0.05 | 1.9 |
| S3 | 4.81 ± 0.12 | 4.88 | +0.07 | 1.5 |
| Mean deviation | | | 0.05 | 2.5 |

## 4. Conclusion and Summary

Up to now there are no CFRP test parts available with a well-defined porosity which can be used as reference parts for destructive and non-destructive porosity measurement methods. Therefore, we have introduced a new way to create reference samples for a quantitative porosity determination by means of CT and evaluated the reference parts. CFRP-reference plates were made by drilling several hundred of micro-holes (diameter 200 and 300 µm) in CFRP-plates. The diameters were evaluated by optical microscopy and X-ray CT to get reference porosity values and the corresponding uncertainty taking into account also machining induced diameter variations near the surface of the drilling hole. These hole-plates were mounted and pressed with porosity free 1 mm CFRP plates at the top and at the bottom. These test parts were scanned by X-ray computed with a voxel size of $(11\ \mu m)^3$ and porosity values were determined by proper processing the CT-data. A very good agreement between the expected and measured values was obtained; the mean deviation of the porosity values was just 0.05 %-points.

**Acknowledgements**
The project was supported by COMET programme of FFG and by the federal governments of Upper Austria and Styria as well as by the program IWB2020 financed by the European Regional Development Fund and the government of Upper Austria (project MICI). We thank Mr. J. Sekelja from FACC for supporting the project.

**References**

[1] J. Kastner, B. Plank, D Salaberger and J. Sekelja. Defect and Porosity Determination of Fibre Reinforced Polymers by X-ray Computed Tomography, 2th International Symposium on NDT in Aerospace, 2010, Hamburg/Germany.
[2] R. Stoessel, D. Kiefel, R. Oster, B. Diewel, L. Llopart Prieto. µ-Computed Tomography for 3D Porosity Evaluation in Carbon Fiber Reinforced Plastics (CFRP), International Symposium on Digital Industrial Radiology and Computed Tomography, 2011.
[3] G. Rao. Evaluation of porosity in Carbon fiber reinforced polymer samples using industrial computed tomography by applying various segmentation methods and measurement parameters, Master thesis, Anhalt University of Applied Sciences Köthen and Martin Luther University Halle-Wittenberg, 2015.
[4] Y. Nikishkov, L. Airoldi, A. Makeev, Measurement of voids in composites by X-ray Computed Tomography, Composites Science and Technology 89 (2013), pp. 89–97.
[5] B. Plank, G. Rao and J. Kastner. Evaluation of CFRP reference samples for pososity made by drilling and comparison with industrial porosity samples by means of quantitative XCT. Proceedings 7th International Symposium on NDT in Aerospace, 2015, Bremen/Germany
[6] J. Kastner (Editor), Proceedings Industrial Computed Tomography Conference 2012, 19.-20. September 2012, ISBN 978-3-8322-9418-2, Shaker Verlag, 2012 and Proceedings Industrial CT-Conference iCT2014, Wels, Austria, 25.-28. February 2014, ISBN-13: 978-3844025576, Shaker Verlag, Aachen (2014).
[7] J. Kastner, B. Harrer, G. Requena and O. Brunke, `A comparative study of high resolution cone beam X-ray tomography and synchrotron tomography applied to Fe- and Al-alloys´, NDT & E International 43 (2010), pp. 599-605.