

High resolution X-ray CT of fibre and particle filled polymers

Johann Kastner, Bernhard Plank, Dietmar Salaberger, Christoph Heinzl

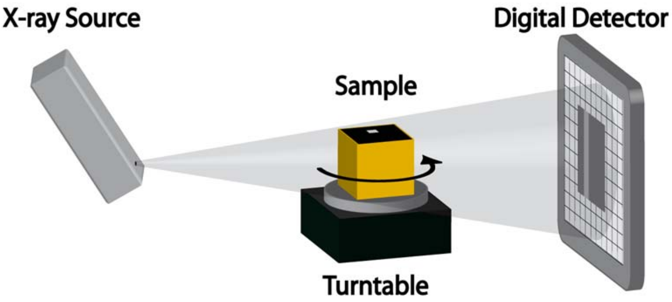
University of Applied Sciences Upper Austria, Wels, Austria.

iCT Conference 2014
February 25 - 28, Wels

Agenda

- Introduction to X-ray computed tomography
- CT of glass, basalt, carbon, and cellulose fibre reinforced polymers – Extraction of fibre properties (orientation, length and diameter distribution)
- Propagation based phase contrast CT of CFRP
- CT of particle filled polymers – Extraction of particle properties

Cone beam X-ray computed tomography



$I(d) = I_0 e^{-\mu d}$
$\rightarrow \mu(x,y,z)$

- Non-destructive testing and materials characterization
- 3D-Geometry measurement

CT-Systems

Type	Beam Geometry	Abbreviation	X-ray source	Voxel size
Medical XCT-systems	Fan beam	med-XCT	140 kV rotating anode tube	> (0.3 mm)³
Industrial XCT: v|tome|x s 240 Rayscan 250E	Cone beam	µXCT	225 kV µ-focus tube	> (3 µm)³
Industrial XCT: Nanotom 180 NF	Cone beam	sub-µXCT	180 kV nanofocus tube	> (0.4 µm)³
Synchrotron XCT: Grenoble, ESRF-XCT ID19	Parallel beam	sXCT	6 – 80 keV	> (0.2 µm)³

Resolutions below 1 µm are possible!

CTs of FH Wels: RayScan 250E und Nanotom

<u>Tubes:</u>
- 225 kV-Microfocus
- 450 kV-Minifocus

<u>Voxelsize:</u> till (3 µm)3

<u>Tube:</u> 180kV high-power nanofocus™

<u>Voxelsize:</u> till (0.4 µm)3

Experimental – Samples

<u>**Materials science:**</u>

- Interested in inhomogeneities like pores, cracks, inclusions, …
- Fibre/Particle distribution and orientation, fibre length and fibre diameter

<u>**Investigated samples:**</u>

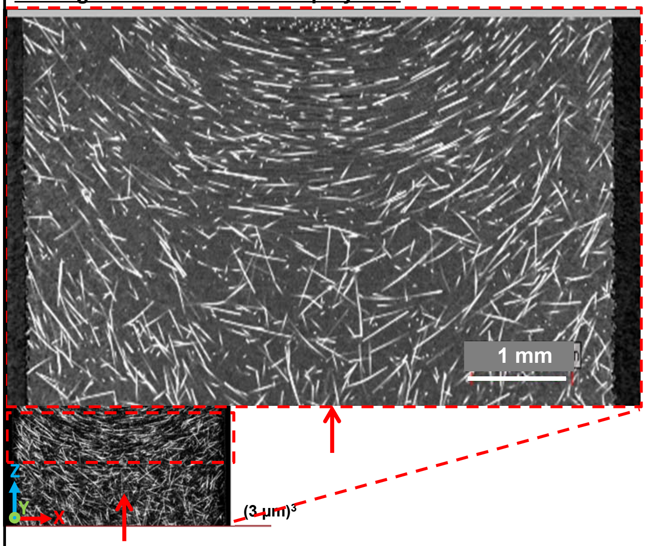
Matrix	Filler	Density of Filler	Voltage	Voxel Size
PP-polypropylene	Glass fibres Cellulose fibres	2.6 g/cm^3 1.5 g/cm^3	60 40-60	(2 – 6 µm)^3 (2.5 – 5.25 µm)^3
PA66-polyamide **Resin**	Carbon fibres Carbon fibres	1.8 g/cm^3	60	(1.25 µm)^3 (1 µm)^3
Resin	Basalt fibres	2.7-3.0 g/cm^3	80	(0.78 µm)^3
PP	Mica particles	2.8 g/cm^3	50	(1.2 - 6 µm)^3

Results 1

- Glass (GFRP), basalt, carbon (CFRP), and cellulose fibre reinforced polymers
- Quantitative fibre extraction – Determination of orientation (orientation tensor), fibre length and diameter distribution

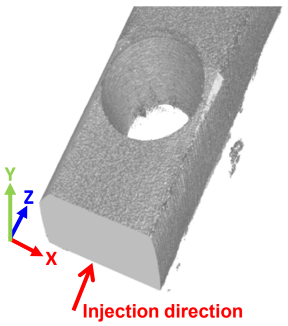
CT of glass fibre reinforced PP

Short glass fibres reinforced polymer:



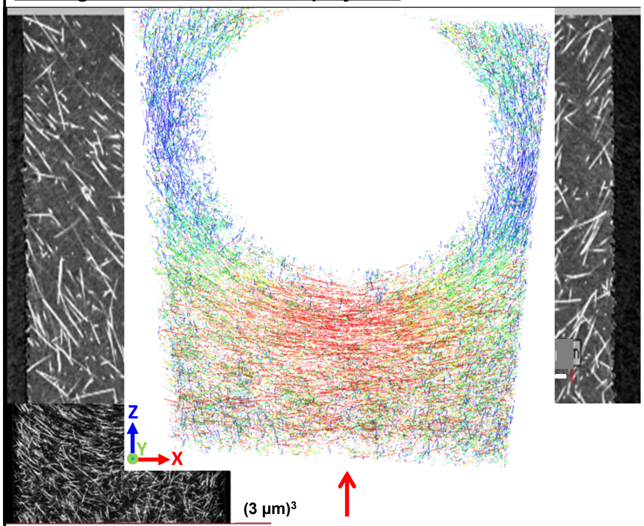
Properties:

- Fibre diameter ~ 13 µm
- Density:
  PP matrix: ~0.9 g/cm³
  Glass fibres: ~2.6 g/cm³



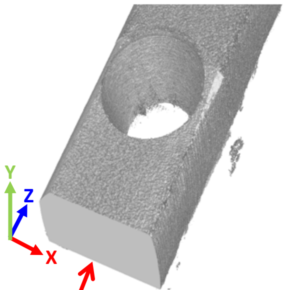
CT of glass fibre reinforced PP

Short glass fibres reinforced polymer:

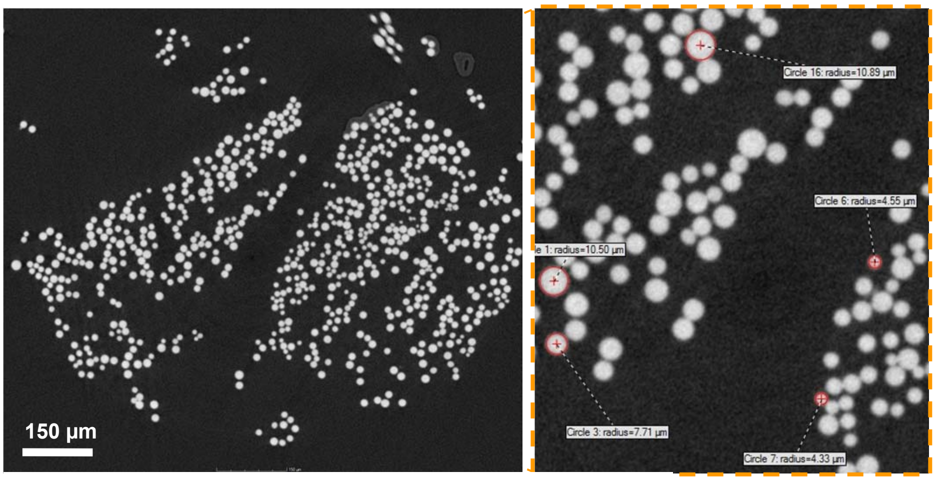


Properties:

- Fibre diameter ~ 13 µm
- Density:
  PP matrix: ~0.9 g/cm³
  Glass fibres: ~2.6 g/cm³



CT of basalt fibre reinforced polymer



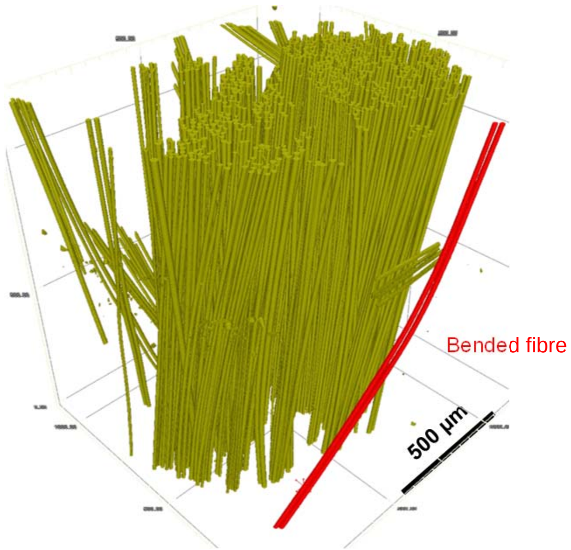
(0,78 µm)³

Properties:

- Fibre diameter: 4-11 µm
- Density: matrix: ~1.1 g/cm³, basaltfibres: ~2.75 g/cm³

CT of basalt fibre reinforced polymer

OBERÖSTERREICH
University of Applied Sciences

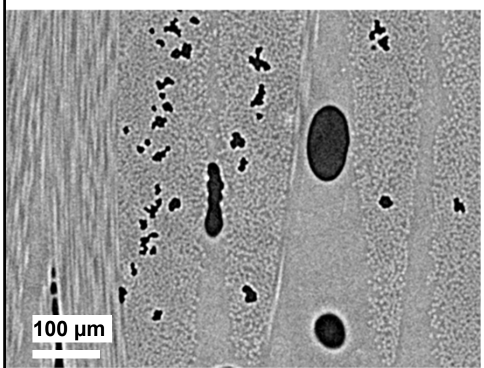


(0,78 µm)³ VS

CT of carbon fibre reinforced polymer

OBERÖSTERREICH
University of Applied Sciences

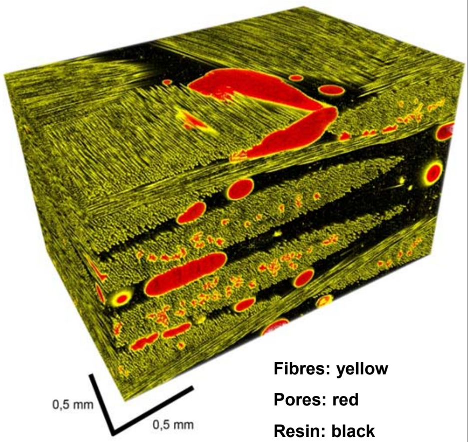
CT-cross sectional picture



(1 µm)³

Individual fibres, resin and pores are visible!

3D-visualization

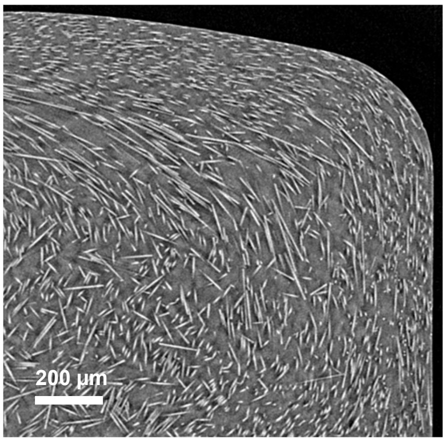


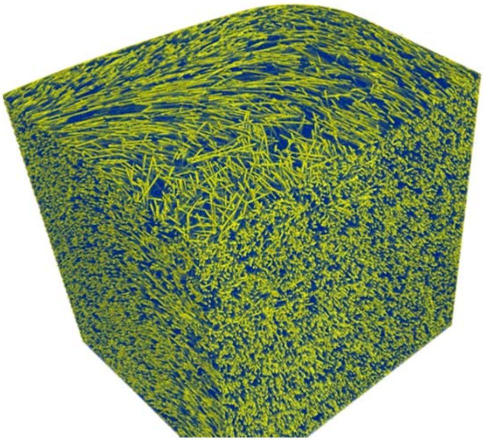
Fibres: yellow
Pores: red
Resin: black

CT of injection molded carbon-fibre reinforced polyamide

CT-cross sectional picture

3D-visualization



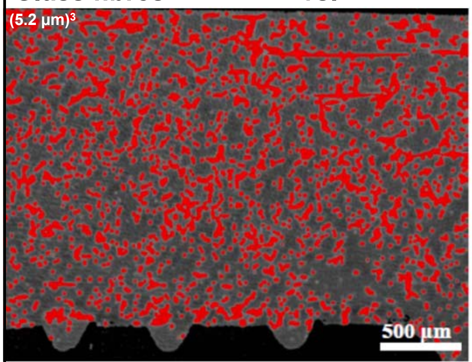


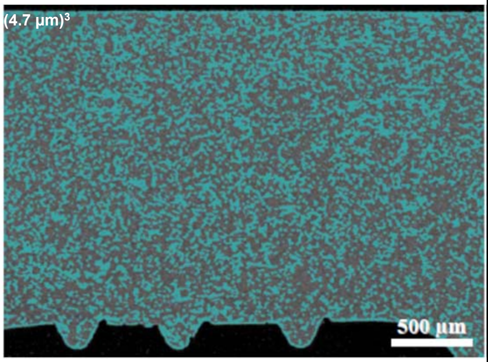
D_{fibre} ~ 7.5 µm

(1.25 µm)³

CT of glass and cellulose fibre reinforced polymer

Glass fibres vs. Cellulose fibres





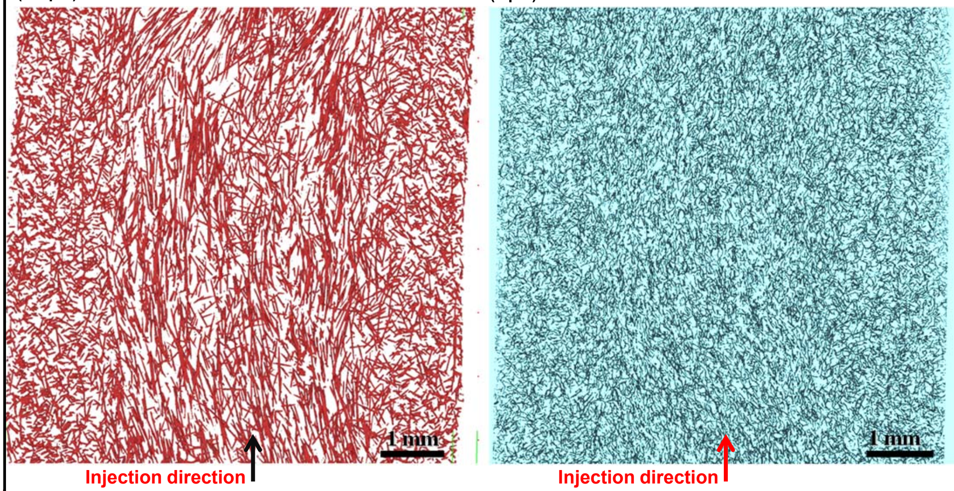
Properties:

- PP matrix: ~0.9 g/cm³
- Cellulose fibres: ~1.5 g/cm³: Ø ~ 7 µm
- Glass fibres: ~ 2.6 g/cm³; Ø ~ 13 µm

CT of glass and cellulose fibre reinforced polymer

Glass fibres vs. **Cellulose fibres**

(5.2 µm)³ (4 µm)³



Quantitative Results

- Quantitative fibre extraction – Determination of orientation, fibre length and diameter distribution

Fibre extraction – Quantitative results

FHW iAnalyse: Individual fibre exctraction pipeline

Original data ↓ **Preprocessing** ↓ **Segmentation** ↓ **Medial axis extraction** ↓ **Individual fibre extraction** ↓ **Fibre characterisation**

Fibre characterisation:

- Orientation …
- Fibre length … … of each individual fibre!
- Diameter …

Global fibre orientation (orientation tensor):

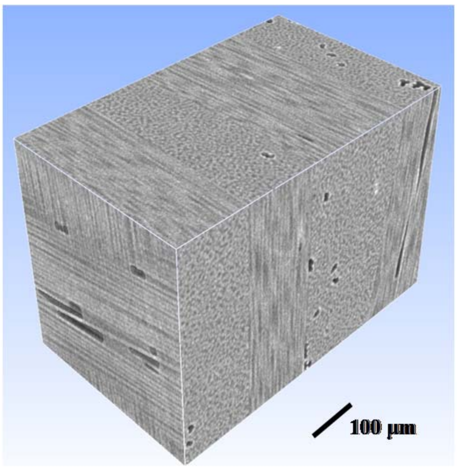
$$a_{ij} = \frac{1}{n}\sum_{k=1}^{n} a_{ij}^{k} = \frac{1}{n}\left(\sum_{k=1}^{n} p_i^k p_j^k\right) = \begin{pmatrix} a_{xx} & a_{xy} & a_{xz} \\ a_{yx} & a_{yy} & a_{yz} \\ a_{zx} & a_{zy} & a_{zz} \end{pmatrix}$$

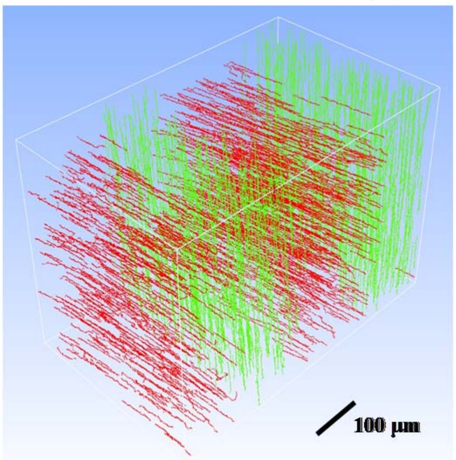
Advantages of XCT fibre characterisation:

- Nondestructive method with high reproducibility
- Many fibres (up to 90.000) per sample
- Only one method is needed for:
  fibre length, diameter and orientation

D. Salaberger, J. Kastner et al, International polymer processing 3, 283-291 (2011)

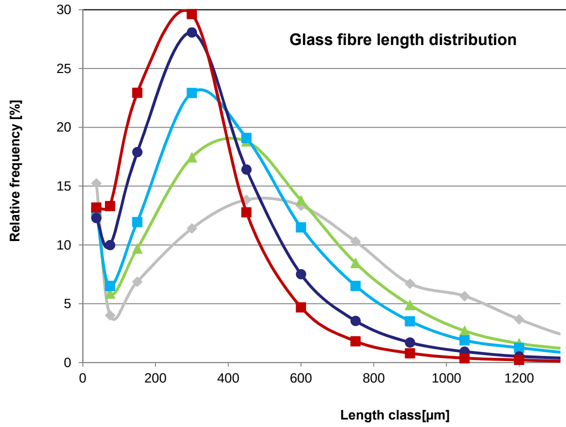
Fibre orientation of CFRP



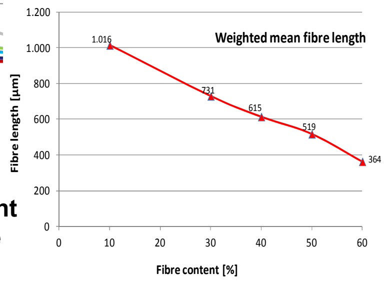


(1 µm)³

Fibre length distribution function of GFRP

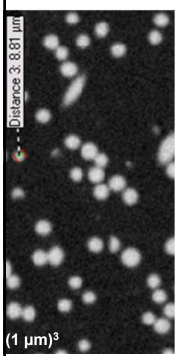


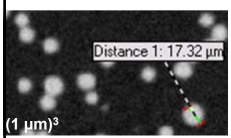
(2 µm)³



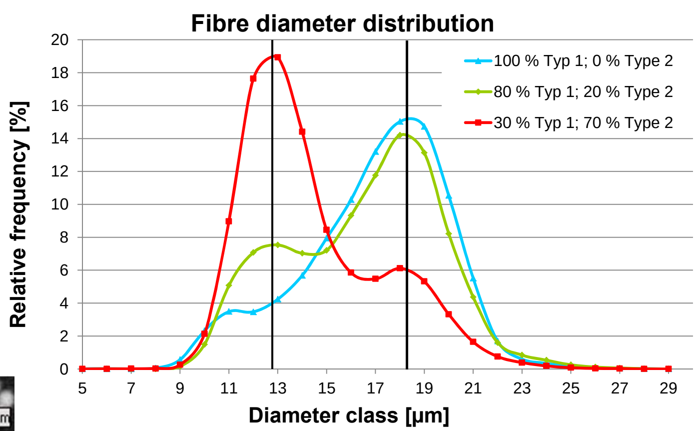
➔ **Fibre length reduction with higher fibre content due to higher fibre-fibre interaction during the compounding process**

Fibre diameter distribution function of GFRP





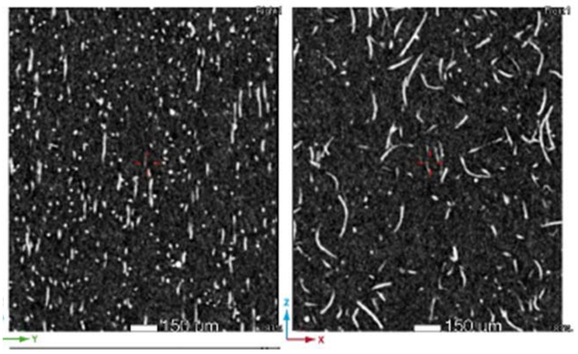
(2 µm)³



Type 1: Ø ~ 18 µm → Short fibres 250 µm initial length
Type 2: Ø ~ 13 µm → Long fibres 4500 µm initial length

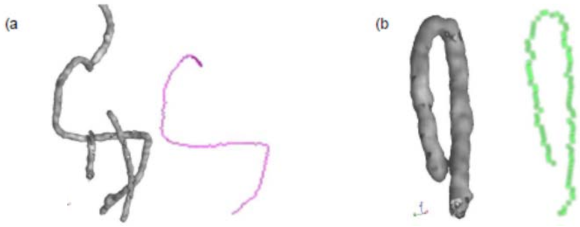
Fibre length distribution of cellulose fibre reinforced polymer

OBERÖSTERREICH
University of Applied Sciences



Cross sectional CT images

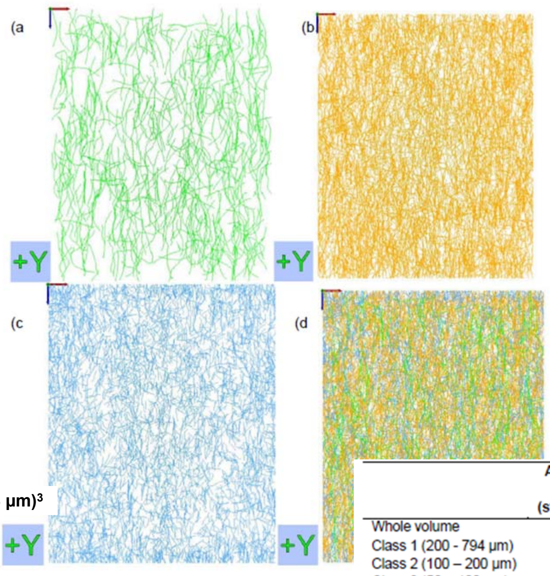
(3 µm)³



3D visualization of two single fibres showing the gray value data on the left and the extracted medial axis in colour on the right

Fibre length distribution of cellulose fibre reinforced polymer

OBERÖSTERREICH
University of Applied Sciences



3D images after fibre extraction showing the medial axes of each fibre:

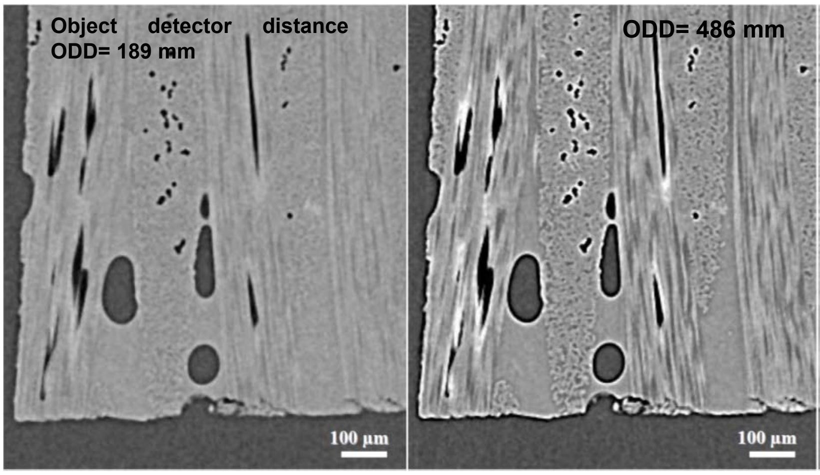
a. fibres longer than 200 µm
b. fibres between 100 and 200 µm
c. fibres between 50 and 100 µm
d. Visualization of all fibres

	Average length (start-end)	Average length (traced)	a11	a22	a33	No of fibres
Whole volume	109,6	116,2	0,24	0,04	0,73	8936
Class 1 (200 - 794 µm)	221,1	251,1	0,15	0,02	0,83	541
Class 2 (100 – 200 µm)	123,2	139,6	0,21	0,03	0,76	4349
Class 3 (50 – 100 µm)	69,8	72,5	0,27	0,05	0,68	4046

Results 2

- Propagation based phase contrast CT of CFRP

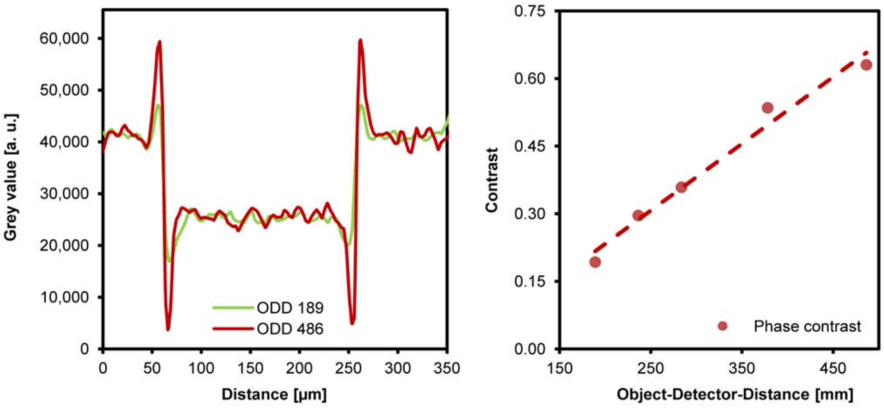
Propagation based phase contrast CT of CFRP



$(2.75\ \mu m)^3$

- Phase contrast increases with increasing ODD
- Detectability of details is improved by the phase contrast

Phase contrast CT of CFRP



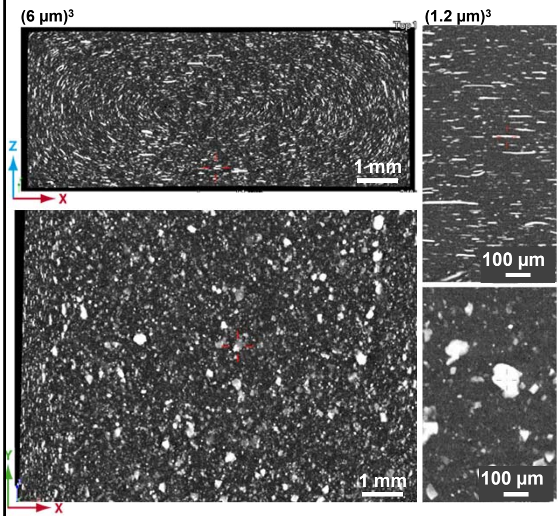
J. Kastner et al, Materials characterization 64, 79-87 (2012)

Results 3

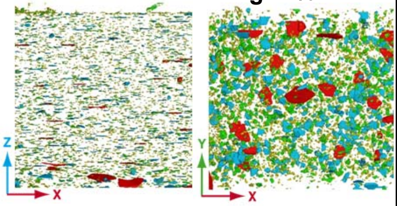
- **CT of particle filled polymers**

CT of PP with Mica (injection moulded)

FH OBERÖSTERREICH
University of Applied Sciences



Mica-content 1 Weight-%



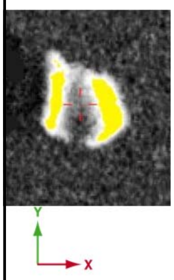
Properties:

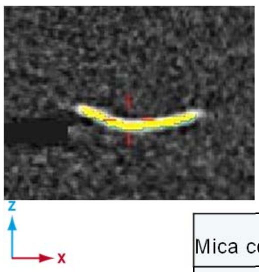
- Density: PP matrix: ~0.9 g/cm³
  Mica: ~2.8 g/cm³

D. Salaberger, et al.; Comparison of particle size distributions derived from 2D and 3D analysis for particle filled polymers; *Polychar 2012.*

CT of PP with Mica (injection moulded)

FH OBERÖSTERREICH
University of Applied Sciences





Mica content wt.		10 %	1 %
Region	[µm³]	966 x 938 x 814	951 x 928 x 811
Particle count		37407	5646
Weight fraction from volume*	[%]	12,1	1,3
Size X*	[µm]	16,9	13,2
Size Y*	[µm]	6,17	5,26
Size Z*	[µm]	16	13,55

* Mean values

EU- FP7 Nano-XCT

- EU-FP7 Nano-XCT (2012-2015, 4,5 Mio €) www.nanoxct.eu - Compact XCT system for non-destructive characterization of nano materials: First prototype at the end of 2014!

Targeted NanoXCT demonstration system specifications:	
Scanning time:	~ 10 hours
Field of view:	1 mm
Probe size:	<= 1 mm³
Voxel size:	50 nm
Targeted price of volume product:	750 k€
Analysis modes:	3D structural and chemical analysis

EU-FP7 QUICOM

University of Applied Sciences

- EU-FP7 QUICOM (2012 – 2015, 5.1 Mio €) www.quicom-project.eu, Quantitative inspection of complex composite aeronautic parts using advanced X-ray techniques

Targeted QUICOM technology platform	
Robot based XCT	For large and complex aeronautic components, reaching a reconstructed volume of 5000 mm³ at voxel sizes of 35 µm; up to 40 cm cumulative material thickness; 2 m * 2 m @FHG labs; up to 2 m * 40 m or beyond @production site
Fast process integrated XCT	X-ray radioscopy, tomosynthesis and CT process for fast and reliable inspection of small high volume parts
Software demonstrator	Large data analysis and visualization, hardware accelerated methods and techniques
CFRP modeling and simulation	For CFRPs based on XCT data including flaws to characterize precise mechanical properties as well as the development of a progressive damage model of CFRP and a neural network trained using this model
Advanced XCT techniques	particularly multispectral CT, using recently emerged semiconductor detectors, and phase contrast technique
Multi energy XCT	Optimization for scanning and evaluation of composite metallic parts with less artefacts and robust results
High resolution XCT	Optimized spatial and contrast resolution, Evaluation of carbon fiber distribution and orientation
X-ray simulation	Dedicated simulations for each of proposed part type for optimal XCT scans

Conclusion

- Cone beam CT is a powerful tool for characterisation of fibre- and particle filled polymers. One can get qualitative and quantitative 3D information about:
  - Fibre orientation, fibre length and diameter distribution
  - Geometrical properties of particles
  - Pores and other inhomogeneities
- Even for a polychromatic X-ray cone beam system phase contrast effects can occur.

CT Team of University of Applied Sciences Upper Austria

Prof.(FH) Priv.-Doz. DI Dr J. Kastner
DI D. Salaberger
DI(FH) Dr. C. Heinzl
DI(FH) M. Reiter
DI(FH) B. Plank, MSc
A. Reh, MSc
DI Ch. Gusenbauer
Mag. Elena Sell
Eva Kremshuber, BA
A. Mustafa, BSc
J. Weissenböck, MSc
Dr. A. Amhirkanov, MSc
Mag. Dr. S. Senck
Ch. Hannesschläger, MSc
B. Fröhler, MSc
St. Zaunschirm, BSc
H. Zauner, BSc
Ch. Affenzeller, BSc

Welcome to Conference on Industrial Computed Tomography (ICT):

Find all relevant information on www.3dct.at/ict2014 !