23<sup>rd</sup> International Conference on Composite Materials (ICCM23) Belfast 30<sup>th</sup>/07 – 4<sup>th</sup>/08 2023

### EFFICIENT USE OF MICRO-TOMOGRAPHY FOR IN-DEPTH CHARACTERIZATION OF COMPOSITES

F. Forsberg, P. Fernberg, Z. Al-Maqdasi, V. Petkov, H. Lycksam and <u>R. Joffe</u>



Luleå University of Technology, Luleå, SWEDEN



### **Motivation**

The X-ray Microtomography (XMT) has been increasingly used in studies on polymer composites. However, often it is used solely for the sake of having images that catch attention. While properly processed these 3D-scans can give unique information about the micro-structure, defects, damage and other features of composites.

However, to gain such information one requires advanced knowledge of tomography as well as image processing.

### **Objectives**

The aim of the current paper is to demonstrate versatility and usefulness of the XMT for characterization of the micro-structures of different types of polymer composites based on the studies carried out at LTU.



### XMT



### **Equipment and Methodology**

The XMT-studies were carried out using a Zeiss Xradia 510 Versa. The tube voltage and output effect for the XMT-scanning was set to obtain desired field-of-view (FOV), resolution and contrast.

Typically FOV is within 2 x 2 mm  $\dots$  12 x 12 mm, with 2-12  $\mu$ m voxel size, 50-60 kV / 4-5 W, exposure time 2...20s.

The 3D image analysis was carried out using the software Dragonfly Pro (ORS) but in some cases in-house codes were employed.



### **Equipment and Methodology**





### **Use of XMT (Polymer Composites)**

Typical micro-structural parameters:

- Shape/geometry of reinforcement
- Fiber volume fraction
- Void content
- Fiber dimensions and orientation
- Bundle/layer dimensions

Not analyzed
Quantified
Estimated
Difficult to do
Not possible

#### Characterization of damage:

- Type of damage (e.g. cracks, delaminations, fiber breaks)
- Amount of damage
- Location of damage

LULEÅ UNIVERSITY OF TECHNOLOGY

2

3

4

5

6

### **Results: natural fiber composites**





#### Flax fiber orientation depending on type of reinforcement

Miettinen A., Joffe R., Pupure L. & Madsen B, "Identification of true microstructure of composites based on various flax fibre assemblies by means of three-dimensional tomography", ICCM20



### **Results: natural fiber composites**





The porosity measurements for composite with Tencel fibers (Wf = 40%).

> 2D: 32.5% 3D: ~30%

Compares fairly well with porosity calculated from theoretical and measured densities of composite.

Joffe R., Pupure L., Berthold F. & Varna J., "Micro-structure and Mechanical Properties in PLA Reinforced with Cellulosic Fiber Sheets Made by Wet Forming Method", CompTest2017



### **Results: natural fiber composites**







#### Copper-coated regenerated cellulose fiber

Al. Maqdasi Z., Emami N., Joffe R., Chouhan S.S., Ouarga A., Hajlane A., "Functional cellulose fibers for smart composites", FiBreMoD Conference 2019.



### **Results: void content**









#### Determining void content, size, distribution

Joffe R., Forsberg F., Lycksam H., Sjögren A. ICTMS2017



### **Results: damage**





Miettinen A., Joffe R., Pupure L. & Madsen B, "Identification of true microstructure of composites based on various flax fibre assemblies by means of three-dimensional tomography", ICCM20



### **Results: thin-ply composite**





Thin-ply carbon fiber composite (Oxeon, layer thickness 25 μm)





Thin-ply carbon fiber composite, (Oxeon, layer thickness 25  $\mu$ m). Details/defects on fiber/matrix level are visible.

**Results: thin-ply composite** 



3

5 6

8

### **Results: damage**







XMT allows much more comprehensive visualization and quantification of microdamage than OM

Non-crimp carbon fiber composite with damage (micro-cracks)



### **Results: damage (ageing)**



3

8

6

### **Results: damage (ageing)**





## (A) Baseline non-aged(B) Baseline aged

Network of micro-cracks that cannot be detected by use of OM



**(B)** 

### **Results: damage (ageing)**





# (C) Thin-ply non-aged(D) Thin-ply aged

XMT allows to detect micro-cracks in thin-ply composite laminates



### Conclusions

With the use of right software (tailored for composites) the 3D scans from XMT allow to access information about composite micro-structure, defects, damage and other features that are not available by use of any other technique.

For instance, the OM or SEM does not allow detection of network of micro-cracks or important characteristics of defects (e.g. shape of voids and micro-cracks and their path/directionality).

Specimen preparation (e.g. polishing) for other imaging techniques may alter characteristics of micro-structure and damage.

## Thank you attention!







#### 3D XMT at LTU

#### Polymer Composites at LTU

