High-resolution X-ray computed tomography of inhomogeneous materials

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Abstract

In this contribution, high-resolution X-ray computed tomography (XCT) results of various inhomogeneous samples including a poplar wood sample, a carbon fibre reinforced polymer sample and an AlSiCu light metal alloy are presented. These samples have been acquired with a new lab-based nano-XCT device equipped with a nano-focus X-ray tube and two different detector systems. Depending on the material system and the measurement task, the user has to choose between these two detector types that can be exchanged by a quick mounting system. Limitations on resolution and contrast-to-noise ratio (CNR) are discussed qualitatively and quantitatively for selected measurements and evaluation tasks. Structures between 500 and 700 nm could be clearly resolved. In addition the positioning accuracy of the exchangeable flat panel detector is investigated. Detector repositioning shows high reproducibility, but systematic measurement errors are in the range of half of a voxel for repeated scans of a calibrated ball bar phantom with a system voxel size of (3 µm)³.

1. Introduction

X-ray computed tomography (XCT) is a non-destructive testing method for reliable component testing and for materials characterization of inhomogeneous materials such as fibre reinforced polymers [1], light metal alloys [2] or even more complex multi-material systems and hybrid structures [3]. Even though XCT is already an established tool, high-resolution scanning within laboratory environments and reasonable scanning times, adequate sharpness, good contrast and low image noise is still a challenging task. High-resolution is required, since the microstructure (e.g. orientation, length and diameter of fibres or the interconnectivity of intermetallic phases) is important for understanding material related properties and its characterization is essential to make the right decisions in terms of new material and component design.

Figure 1. (a) Nano-XCT EasyTom 160 with mounted (b) flat panel detector, (c) CCD camera and (d) a photograph of the mounting system for a quick exchange of both detectors.
In order to meet the requirements on high-resolution and 3D characterization of various inhomogeneous material systems, a new lab-based nano-XCT system has been installed at the University of Applied Sciences Upper Austria in Wels.

2. Experimental setup

2.1 Nano-XCT device

The nano-XCT system EasyTom 160 (see Figure 1a) from the company RX Solutions is consisting of a 160 kV nano-focus X-ray tube from Hamamatsu for resolutions below 400 nm and of two different detector types: (i) a Varian flat panel detector (Figure 1b, 1920×1536 pixels, 127 µm pixel size) for high energies and short scanning times (e.g. in-situ experiments) suitable for multi-material components or high absorbing structures and (ii) a Ximea CCD camera (Figure 1c, 4032×2688 pixels, 9 µm pixel size) for low energies and low absorbing structures suitable for e.g. high-resolution applications. Figure 1d shows the mechanism to unlock the mechanical support at the rear of both imager types with a dedicated tool allowing a quick exchange of the detector systems. Plugging in the appropriate cables automatically activates the controls in the acquisition software.

2.2 Samples and XCT scan parameters

For this high-resolution study, a poplar wood sample, a carbon fibre reinforced polymer (CFRP) sample and an AlSiCu light metal alloy have been selected and characterized regarding their microstructure. Voxel sizes are ranging from (300 nm)³ to (500 nm)³. Further scan parameters are summarized in Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample size in [mm]</th>
<th>Voxel size</th>
<th>Scanning time in [min]</th>
<th>Tube voltage in [kV]</th>
<th>Detector</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poplar wood</td>
<td>diameter Ø 2 mm</td>
<td>(1.25 µm)³</td>
<td>240</td>
<td>80</td>
<td>CCD</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(500 nm)³</td>
<td>360</td>
<td></td>
<td>CCD</td>
</tr>
<tr>
<td>CFRP sample</td>
<td>cross section 5 mm x 1 mm</td>
<td>(300 nm)³</td>
<td>720</td>
<td>80</td>
<td>CCD</td>
</tr>
<tr>
<td>AlSiCu alloy</td>
<td>diameter Ø 1 mm</td>
<td>(400 nm)³</td>
<td>480</td>
<td>80</td>
<td>Flat panel</td>
</tr>
</tbody>
</table>

The scanning time has been limited to a maximum of about 12 hours for a reasonable scanning effort. A maximum tube voltage of only 100 kV can be used in the case of the fine-focus LaB₆ filament and the CCD camera. Higher kV settings are not advisable, since they will most probably damage the CCD camera. Even though, lower kV settings generally lead to higher contrast due to higher X-ray attenuation differences for the investigated material systems, the image blur caused by the X-ray tube’s focal spot increases slightly with lower kV settings. This image blur can lead to a contrast drop and thus lowering the resolution. Therefore 80 kV have been preferably used for the study of all materials.

In general, the detector integration time and averaging have to be carefully set to limit free beam grey values of CCD and flat panel projection data and to avoid saturation.
effects, especially for the CCD camera, since it is more sensitive to these issues. Further detector specific properties have been considered for acquisition parameter selection: (i) the maximum integration time in the case of the flat panel should be less than 2000 ms in order to minimize possible image lag and (ii) the image averaging of the CCD camera has to be set at least to a value of three to eliminate bright and noisy pixels in CCD projection images. Previous simulation-based studies have shown that the CCD camera is not applicable for high-resolution scanning of particular light metal alloys, since the CCD camera leads to higher image noise and thus to lower CNR values as compared to the flat panel detector [4].

2.3 Test phantoms and ball bar

The 2D imaging capabilities of the nano-XCT have been tested with a JIMA mask ‘RT RC-04’ made of line-pairs down to 100 nm in line and space size. A calibrated ball bar with two ruby spheres and the QRM ‘MicroCT Bar Pattern NANO Phantom’ have been used to perform systematic tests on the 3D tomographic acquisition capabilities. The corresponding scan parameters can be found in Table 2.

<table>
<thead>
<tr>
<th>Phantom</th>
<th>Voxel size</th>
<th>Scanning time in [min]</th>
<th>Tube voltage in [kV]</th>
<th>Detector</th>
</tr>
</thead>
<tbody>
<tr>
<td>QRM ‘MicroCT Bar Pattern NANO Phantom’</td>
<td>(0.75 µm)³</td>
<td>300</td>
<td>80</td>
<td>Flat panel</td>
</tr>
<tr>
<td>Ball bar phantom (scan series)</td>
<td>(3 µm)³</td>
<td>25</td>
<td>90</td>
<td>Flat panel</td>
</tr>
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2.4 Analysis of exchangeable detectors and positioning precision and accuracy

In order to study the positioning precision and accuracy of the exchangeable detectors a measurement series has been carried out over two consecutive days. Figure 2 shows the measurement sequence in more detail. Before starting the measurement series a procedure for calibrating the system geometry has been performed in order to reduce geometrical inaccuracies. This includes homing of all axes and acquiring projection images of a dedicated phantom with a steel ball for different measurement positions. The system calibration has only been performed once for the whole series. At the beginning of each measurement day, a beam alignment procedure as well as a gain offset correction has been carried out.

![Figure 2. Measurement strategy for studying the positioning accuracy of the exchangeable detectors (flat panel detector only)](image_url)

The scan series was aiming to analyse the influence of the detector mounting precision on the magnification by repeated ball bar measurements. Therefore, the detector has been repeatedly removed and remounted from its support in between the individual
scans. The actual measurement position of the ball bar has been approached only once after system calibration and it has not been replaced during scanning. The variance in magnification is determined for a series of ball bar measurements with a calibrated distance of 3.9796 mm at a constant magnification of 42.2 and a voxel size of (3 µm)³. This investigation has been done so far only for the flat panel detector, since it is more versatile, when it comes to dimensional measurements of larger parts or materials characterization tasks due to a larger field of view and a better spectral acceptance for larger and higher dense objects. Previous studies showed that the flat panel detector is more applicable for higher dense objects [4] than the CCD camera, which is also the case for the ball bar and the QRM test phantom. Suitable test phantoms will be depicted in the future to repeat the investigations also with the CCD camera.

3. Experimental results

3.1 Test phantoms

The 2D projection image of the JIMA mask in Figure 3a clearly indicates the capability of the nano-focus X-ray source to resolve line pairs with 400 nm. Figure 3b shows an XCT slice image of a central part of the QRM ‘MicroCT Bar Pattern NANO Phantom’ acquired with the flat panel and a voxel size of (750 nm)³. Data has been reconstructed with half of the native voxel size. All of the smallest features that are either realised as vertical (Figure 3, #1) or horizontal bar pattern (#2) with 1 µm in line and space width as well as holes with a diameter of 1 µm (#3) could be resolved. Since there are no suitable test phantoms in 3D with features sizes in the nanometre regime commercially available for further resolution testing, we have chosen real samples for identifying the physical resolution limits of the nano-XCT for tomographic operation. Figure 3c shows a 3D rendering of the ball bar that has been evaluated for determining position accuracy.

![Figure 3. (a) 400 nm line pairs of the JIMA mask captured by a 2D projection image, (b) XCT slice image in a central region of the QRM test phantom with zoomed images of vertical bars with 1 µm in line and space width (#1), horizontal bars (#2) and 1 µm holes (#3) and (c) a 3D rendering of the ball bar](image-url)
3.2 Exchangeable detectors and positioning accuracy

Table 3 shows the measurement results of scan series regarding all relevant geometrical values such as centre-of-rotation (COR) values determined per scan, uncorrected and corrected system voxel sizes, scaling error, evaluated centre-to-centre ball bar distances (VGStudio Max 3.0 and ‘advanced surface determination’) and errors to measurements performed by a calibrated coordinate measurement machine (CMM) given in mm and voxel. Furthermore, the table shows the magnification errors interpreted as source-to-detector (SDD) errors. The evaluated scan series of repeatedly remounting the detector shows a high reproducibility below 1 µm for the measured length. The resulting measurement errors show a bias in the range of half a voxel to the tactile reference measurement. Repeated experiments depicted that this bias might not be related to the detector replacement. Therefore, the influence of the system calibration procedure on the observed systematic offset has to be investigated in more detail in future studies.

Table 3. Centre of rotation (COR) misalignments, uncorrected and corrected system voxel sizes, scaling error, centre-to-centre ball bar distance, deviations to CMM values given in mm and units of voxel size and the magnification error interpreted as source-to-detector (SDD) error, magnification = 42.2, pixel size = 127 µm

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<td>-0.205</td>
</tr>
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</table>

3.3 High-resolution scans of inhomogeneous materials

Figure 4 shows the results of high-resolution scans of a poplar wood, CFRP and AlSiCu alloy sample. All high-resolution scans below (1 µm)³ in voxel size have been acquired as region-of-interest (ROI) scans of the central sample region with continuous rotation mode in order to blur the contribution of the material that is outside of the field of view.

Fast-growing trees such as poplar wood are being seriously considered for future supply needs. Some of the properties of poplar make it appealing for certain applications and some of them not [5]. Therefore it is crucial to improve material knowledge of its cell structure by performing e.g. high-resolution studies of both uncompressed and densified wood [6]. The measurement task for this study is to obtain the microstructure (vessels, cell walls, stomata, etc.) of a selected uncompressed poplar wood sample. Figure 4a shows a 3D rendering of the poplar wood sample acquired with an overview scan and a voxel size of (1.25 µm)³. The rendering is showing a transition between latewood and earlywood. The inner white circle indicates the scan volume of the ROI scan acquired with a voxel size of (500 nm)³. Figure 4b shows an axial slice image of this ROI scan.
with two marked regions (white squares) that are displayed with a higher zoom on the right. These regions show besides small pores within the cellular structure also gap-like structures of the poplar wood microstructure that are between 700 and 900 nm in size.

Figure 4. High-resolution XCT results of a (a-b) poplar wood sample acquired with $(500 \text{ nm})^3$ voxel size, (c-d) a CFRP sample with $(300 \text{ nm})^3$ voxel size and (e-f) an AlSiCu alloy with $(400 \text{ nm})^3$ voxel size acquired with the nano-XCT device.

CFRP is widely used for lightweight applications in the automotive and aeronautic field. Figure 4c shows a 3D rendering of the ROI scan volume of the CFRP sample with a voxel size of $(300 \text{ nm})^3$ and four different orientated carbon fibre bundles embedded within a large resin-rich area. The diameter of the individual carbon fibres is in the
range of 7-8 µm. The measurement task is to achieve best possible image quality in terms of high resolution, high contrast and low noise in order to segment the individual carbon fibres and to detect potential failures (such as pores, etc.) within the epoxy resin. One of the marked positions in Figure 4d reveals one small pore with a diameter of about 2.7 µm whereas the other marked position is used to determine the gap between two individual carbon fibres that is in the range of about 500 nm. Edge enhancements due to a high spatial resolution and refraction effects facilitate the identification of individual fibres and different material phases with similar attenuation coefficients as it is the case for carbon fibres and epoxy resin.

Figure 6e shows a 3D rendering of an AlSiCu sample with a cubic cut-out at the top corner that has been acquired with the flat panel detector and a voxel size of (400 nm)³. Lightweight alloys based on aluminium have become of great importance e.g. in the field of engineering for manufacturing parts that are used for transportation (e.g. Al cylinder heads). The scanned AlSiCu material system consists of an Al-matrix, in which Si-particles are embedded, as well as various, higher-dense and complex intermetallic aluminium and copper phases with very fine structures. The measurement task is to segment each intermetallic material phase and quantify its microstructure. Figure 6f shows the smallest measureable details within the fishbone-like structures of the intermetallic phases that are within the range of 700 nm. The image quality of the flat panel detector shows high contrast and low image noise being suitable for segmenting and quantifying higher-dense material phases. In addition, larger Si particles in the µm range can be identified within the Al-matrix that is usually quite though to resolve due to similar attenuation coefficients of Al and Si.

4. Conclusions

A physical resolution between 500 and 700 nm (approximately two times of the used voxel size) could be achieved by first high-resolution nano-XCT scans of selected material systems such as poplar wood, CFRP and AlSiCu light metal alloy with reasonable measurement times below 12 hours and acceptable signal-to-noise ratio. The nano-XCT system is suitable for the high-resolution characterization of biologic and polymeric samples as well as light metal alloys for revealing their microstructure. In the case of the poplar wood sample the cell walls with internal features such as pores and gap-like structures in the range of 700 and 900 nm could be identified. For the CFRP sample refraction effects lead to edge enhancements that could be used for further segmenting of individual carbon fibres for retrieving characteristics such as fibre orientation, length and diameter. The high-resolution scan of the AlSiCu sample revealed two complex intermetallic phases with fine fishbone-like structures in the range of 700 nm that could be segmented and visualized in 3D. In addition, larger Si particles within the Al-matrix could be resolved, but at the moment, the data quality is not sufficiently to segment these Si-particles in an automated way.

Even though the system precision regarding repositioning of the flat panel detector shows high reproducibility of less than 1 µm, replacing the detector leads also to systematic measurement errors in the range of half of a voxel that has been tested for a repeatedly scanned ball bar phantom with a voxel size of (3 µm)³. The precision seems sufficient for materials characterization and dimensional measurement. For dimensional
measurements or for extracting geometrical features such as fibre length or diameter it is advisable to perform a scale correction by measuring a calibrated ball bar phantom in addition to improve the accuracy. The observed bias that decreases the accuracy might be caused by the system calibration procedure. At the moment, statements regarding precision and accuracy for voxel sizes below (1 µm)^3 are not possible, since suitable calibrated reference standards are not available. Further high-resolution applications and test phantoms will be targeted in future studies to explore the full potential of the nano-XCT device regarding resolution limits and overall system accuracy.

Acknowledgements

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