A comparative study of high resolution cone beam X-ray tomography and synchrotron tomography applied to Fe- and Al-alloys

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\textbf{Abstract}

X-ray computed tomography (XCT) has become a very important method for non-destructive 3D-characterization and evaluation of materials. Due to measurement speed and quality, XCT systems with cone beam geometry and matrix detectors have gained general acceptance. Continuous improvements in the quality and performance of X-ray tubes and XCT devices have led to cone beam CT systems that can now achieve spatial resolutions down to 1 \( \mu \text{m} \) and even below. However, the polychromatic nature of the source, limited photon flux and cone beam artefacts mean that there are limits to the quality of the CT-data achievable; these limits are particularly pronounced with materials of higher density like metals. Synchrotron radiation offers significant advantages by its monochromatic and parallel beam of high brilliance. These advantages usually cause fewer artefacts, improved contrast and resolution.

Tomography data of a steel sample and of two multi-phase Al-samples (AlSi12Ni1, AlMg5Si7) are recorded by advanced cone beam XCT-systems with a \( \mu \)-focus (\( \mu \text{XCT} \)) and a sub-\( \mu \)-focus (sub-\( \mu \text{XCT} \)) X-ray source with voxel dimensions between 0.4 and 3.5 \( \mu \text{m} \) and are compared with synchrotron computed tomography (sxCT) with 0.3 \( \mu \text{m/voxel} \). CT data features like beam hardening and ring artefacts, detection of details, sharpness, contrast, signal-to-noise ratio and the grey value histogram are systematically compared. In all cases \( \mu \text{XCT} \) displayed the lowest performance. Sub-\( \mu \text{XCT} \) gives excellent results in the detection of details, spatial and contrast resolution, which are comparable to synchrotron-XCT recordings. The signal-to-noise ratio is usually significantly lower for sub-\( \mu \text{XCT} \) compared with the other methods. With regard to measurement costs “for industrial users”, scanning volume, accessibility and user-friendliness sub-\( \mu \text{XCT} \) has significant advantages in comparison to synchrotron-XCT.

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1. Introduction

High resolution X-ray computed tomography (XCT) has gained considerably in importance and acceptance for the examination and 3D-characterization of materials and industrial specimens in recent years [1–10]. XCT systems with cone beam geometry with a \( \mu \)-focus or a nano-focus X-ray tube in combination with a high speed and high resolution flat panel matrix detector are widely used in materials science [1–5]. Using cone beam XCT, a specimen is placed on a rotary stage 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 projections to a volume dataset using, for example, the filtered back projection algorithm. At each position of the resulting dataset a grey-value is calculated in the scanned sample volume, which corresponds to the effective X-ray attenuation coefficient \( \mu(x,y,z) \). The major problems of cone beam XCT with a polychromatic source are the limited transparency of the sample, the restricted geometrical accuracy and resolution and measurement artefacts. Artefacts are structures resulting from the reconstruction algorithm of the dataset, which do not correspond to a real sample feature and which lead to problems with contrast, noise as well as measurement interpretation and dimensional accuracy. These problems are more pronounced for polychromatic cone beam XCT as compared with XCT with a monochromatic source. Continuous improvements in X-ray tubes and XCT devices have led to cone beam CT systems which can now achieve resolutions down to 1 \( \mu \text{m} \) (\( \mu \text{XCT} \)) and even below (sub-\( \mu \text{XCT} \)) [2,4,5,11].

Synchrotron radiation offers significant advantages by its adjustability, partial coherence and nearly parallel beam of high brilliance [6]. These advantages cause fewer artefacts, improved contrast and resolution, as well as faster recording for synchrotron tomography (sxCT). SXCT has been known since mid-1980 s and resolutions available now are below 1 \( \mu \text{m} \), e.g. at ESRF/ID19.
allowing in-situ experiments due to the short measuring period [3,6]. Spatial resolution of about 180 nm has recently been achieved for relatively large metallic samples on ESRF-ID22 by using magnifying KB-mirrors [10]. In addition, sXCT produces also phase contrast due to the high lateral coherence of the beam, so that even interfaces between phases with very low absorption contrast can be displayed [2,3]. Phase contrast for cone beam sub-μXCT was also reported but to a much lower extent [11]. The specifications of typical XCT systems (X-ray source and voxel sizes) are compared in Table 1.

There have been several investigations of cone beam XCT and sXCT applied to various materials [7,11–13]. μXCT and sXCT for the 3D-characterization of inhomogeneities in steel are presented in [7], but no quantitative comparison was carried out. The possible applications of sub-μXCT are described in [11], but no comparison with sXCT is presented. A quantitative comparison was carried out in [12], but the resolutions (voxel sizes) used are in the range of several micron (2–10 μm) and thus not in the sub-μm-XCT region. In [13] cone beam μXCT and sXCT are compared quantitatively but no sub-μXCT cone beam XCT measurements were performed. In summary, there are no systematic investigations on metallic samples with up-to-date spatial resolutions down to 1 μm (voxel sizes below 1 μm) published.

This paper deals with the application of high resolution cone beam XCT in comparison with synchrotron XCT applied to Fe- and Al-based samples containing several phases. The voxel sizes for the investigations presented here are in the sub-μm region. The XCT-data are analysed with respect to measurement artefacts, detection of details, sharpness, contrast and signal-to-noise ratio. The advantages and restrictions of the various methods are presented and discussed.

2. Experimental

2.1. Samples

One steel and two Al alloys were investigated; all the samples included various inhomogeneities such as inclusions, pores or metallic phases with a higher or lower density than the matrix resulting in distinguishable features in the corresponding CT-data-sets. A summary of these samples together with the inhomogeneities present can be found in Table 2.

Regions of the samples were investigated by metallographic analysis and scanning electron microscopy to identify the different inhomogeneities and phases investigated. The elemental analysis was carried out by energy dispersive X-ray analysis (EDX).

2.2. Computed tomography

2.2.1. CT-data recording

The μ-focus XCT-measurements were performed with two μ-focus cone beam XCT-devices, namely a v-tome-X s constructed by GE Sensing & Inspection Technologies phoenix/ X-ray equipped with a 240 kV-μ-focus tube and a 1024 × 1024 pixel a-Si flat panel detector by Perkin Elmer and a Rayscan 250E-XCT-device constructed by Rayscan Inc. The Rayscan 250E system is equipped with a 225 keV-μ-focus tube by Viscom and a 1024 × 1024 a-Si flat panel matrix-detector by Perkin Elmer. The target for both devices is made of tungsten and the minimum size of the X-ray focal spot for both sources is about 5 μm. The minimum voxel sizes for both systems are around 3 μm, the actual value used was 3.5 μm for all measurements. The tube energies used for scanning were between 90 and 160 kV, the current between 25 and 70 μA and the number of projections 1000–1400. This resulted in a measurement time between 60 and 180 min.

The sub-μm-CT X-ray tomograms were scanned using a nanotom 180NF CT device developed and manufactured by GE Sensing & Inspection Technologies phoenix/ X-ray with a 180 keV high power nanofocus tube and a 2300 × 2300 pixel Hamamatsu detector. Further details can be found in [11]. Targets made of tungsten and molybdenum was used at a voltage of 50–60 kV and a current between 325 and 400 μA. The voxel size used was between 0.4 and 0.6 μm, and the number of projections 1500–1700. These parameters required measurement periods between 120 and 240 min. The X-ray tube of the nanotom is equipped with an external liquid cooling system to ensure stable measurement conditions and to minimize the thermal influences during the long-time scans.

The ID19 beamline of ESRF—European Synchrotron Radiation Facility in Grenoble [6] provided monochromatic X-rays with 29 kV in parallel beam geometry. The sXCT projections were recorded by a 2D-CCD camera with 2048 × 2048 pixels and an effective pixel size of 0.3 μm. For reconstruction 1500 projections were acquired. The samples were measured in phase contrast mode using a distance sample-to-detector of 39 mm. The recordings took about 15 min. Further details can be found in [7,8,15].

Table 3 gives an overview of the most important measurement parameters for μXCT, sub-μXCT and sXCT measurements. Two μXCT and sub-μXCT measurements were performed with the steel sample (scan A and scan B).

2.2.2. CT-data evaluation

The cone beam XCT-data were reconstructed by means of a filtered back projection Feldkamp-algorithm [2,7]. For some of the cone beam XCT-data a beam hardening correction was performed [16]. The sXCT data were reconstructed by using a filtered back projection Feldkamp algorithm.
projection algorithm for parallel beam geometry [6,8]. The reconstructed XCT-data were visualised and processed with the software VGStudio MAX 2.0 by Volume Graphics GmbH. Various parameters were evaluated for interpreting and classifying the XCT-data-sets quantitatively: sharpness, contrast, signal-to-noise ratio and a feature of the grey-value histogram (the width of the material peak).

In order to evaluate the sharpness of the CT-data, the grey-values for air and material were transformed to values between 0 and 65,535 in all CT-measurements. Line profiles were deviated and the mean value of the rising and falling exterior edges of the metallic samples was considered as a parameter for the sharpness. The $\mu$-XCT, sub-$\mu$XCT and sXCT-data were evaluated in the same way and the values determined were compared relatively to each other.

Line profiles were taken to quantify the contrast of the different measurements in the first step, and contrast-values were determined at certain inhomogeneities based on the following formula:

$$\text{contrast} = \frac{\text{grey-value}_{\text{Material}} - \text{grey-value}_{\text{inhomogeneity}}}{\text{grey-value}_{\text{Material}}}$$

Table 3
Overview of the most important CT-measurement parameters for the various samples and for the various XCT-measurement systems.

<table>
<thead>
<tr>
<th>Sample/CT-parameter</th>
<th>$\mu$XCT</th>
<th>Sub-$\mu$XCT</th>
<th>sXCT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Steel</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Voltage (kV)/beam energy</td>
<td>Scan A</td>
<td>Scan B</td>
<td>91</td>
</tr>
<tr>
<td>Voxel size ($\mu$m)</td>
<td>3.5</td>
<td>3.5</td>
<td>0.5</td>
</tr>
<tr>
<td>Beam current ($\mu$A)</td>
<td>67</td>
<td>55</td>
<td>325</td>
</tr>
<tr>
<td>Target</td>
<td>W</td>
<td>W</td>
<td>W</td>
</tr>
<tr>
<td><strong>AlSi12N1</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Voltage (kV)/beam energy</td>
<td>160</td>
<td>50</td>
<td>29</td>
</tr>
<tr>
<td>Voxel size ($\mu$m)</td>
<td>3.5</td>
<td>0.4</td>
<td>0.3</td>
</tr>
<tr>
<td>Beam current ($\mu$A)</td>
<td>25</td>
<td>400</td>
<td>–</td>
</tr>
<tr>
<td>Target</td>
<td>W</td>
<td>Mo</td>
<td>ID19 beamline</td>
</tr>
<tr>
<td><strong>AlMg5Si7</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Voltage (kV)/beam energy</td>
<td>160</td>
<td>50</td>
<td>29</td>
</tr>
<tr>
<td>Voxel size ($\mu$m)</td>
<td>3.5</td>
<td>0.4</td>
<td>0.3</td>
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<td>Beam current ($\mu$A)</td>
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</tr>
<tr>
<td>Target</td>
<td>W</td>
<td>Mo</td>
<td>ID19 beamline</td>
</tr>
</tbody>
</table>

By a polynomial interpolation of parts of the line profiles it was possible to determine grey-values of the material ($=\text{grey-value}_{\text{Material}}$) and grey values of the inhomogeneities ($=\text{grey-value}_{\text{inhomogeneity}}$). The same inhomogeneities were evaluated for all three kinds of XCT-measurements.

The standard deviation ($\sigma$) for the material-grey-values was set as the denominator for the signal-to-noise ratio (SNR). SNR was calculated by the following formula:

$$\text{SNR} = \frac{\text{grey-value}_{\text{Material}} - \text{grey-value}_{\text{inhomogeneity}}}{\sigma}$$

The data were evaluated in the following way to compare the grey-value histograms of the $\mu$XCT, sub-$\mu$XCT and the sXCT-measurements. The FWHM-values (full widths at half maximum) were calculated for the material-peak in relation to the grey-value difference of the material- and the air-peak. These evaluations could only be done for the XCT-results of the steel sample, where the absorption contrast dominates the measurements. For the Al-samples this comparison was not possible, since phase contrast dominates the sXCT-data.

3. Results

3.1. Computed tomography results of steel samples

In the following the CT-results, CT cross-sectional images of slices of the steel sample measured by $\mu$XCT, sub-$\mu$XCT and sXCT are presented in Fig. 1: 3 inhomogeneities are observed in Fig. 1(a) and 2 inhomogeneities in Fig. 1(b). Target metallography and EDX revealed non-metallic inclusions in the CT-images shown in Fig. 1(a) and pores for the CT-images shown in Fig. 1(b).

The CT-data for $\mu$XCT are rather diffuse due to the much larger X-ray emission spot and voxel size compared with the much sharper images obtained by sub-$\mu$XCT and sXCT. Therefore, the sub-$\mu$XCT and sXCT-data reveal many more details. Beam hardening artefacts are visible for $\mu$XCT and sub-$\mu$XCT, whereas the sXCT-data show no beam hardening manifested with more or less homogeneous grey values for steel. Noise is mostly present in the sub-$\mu$XCT data-set. Ring artefacts are most pronounced for the sXCT-data, whereas the cone beam XCT results show almost no ring artefacts. Ring artefacts are well known and caused by faults of the detector [1,2].
Fig. 2 shows CT-cross-sectional images of a different region of the steel sample (scan B) measured with a different \( \mu \)-XCT-device as compared with scan A (see Table 3). In this case a beam hardening correction of the CT-data was performed so that the grey values within the material are constant similar to the sXCT-data. In Fig. 2 the difference between \( \mu \)XCT and sub-\( \mu \)XCT becomes evident. \( \mu \)XCT gives a rather diffuse picture with a much lower resolution of details, but a rather low noise, whereas sub-\( \mu \)XCT gives much sharper data with more details, but the dataset has a higher noise level.

In Fig. 3 enlarged sXCT-slices of different types of inhomogeneities are presented. The spatial and contrast resolutions are so good, that four different grey value contrasts appear. These contrasts can be classified as pores, low-density inclusion, steel and high-density inclusion [7,8]. In both cone beam XCT-methods investigated it is not possible to distinguish between pores and inclusions. These results show the much better contrast resolution of sXCT as compared with cone beam XCT.

3.2. Computed tomography results of aluminium samples

In Fig. 4 XCT-results for an AlSi12Ni1 sample are shown, where different metallic phases are visible. Ni- and Fe-aluminides appear as high-absorbing inhomogeneities within the Al matrix due to the high density and atomic number of Ni [14]. In the \( \mu \)XCT-dataset areas with aluminides can be identified but an accurate segmentation and quantification is not possible as a result of blurred edges. The sub-\( \mu \)XCT and the synchrotron-XCT measurements show the high-absorbing aluminides in much more detail. The XCT-data for all three measurement methods show no beam-hardening and no ring artefacts. The data for sXCT show not only an absorption contrast but also a phase contrast. This results in a much better contrast for interfaces. Therefore, the eutectic Si also becomes visible in the sXCT-dataset.

In Fig. 5 shows XCT results for an AlMg5Si7 sample. Pores and Fe-aluminides are detectable by all three methods. Mg2Si-phases are also recognizable within the sub-\( \mu \)XCT and sXCT datasets [15]. This is depicted in Fig. 6 for the sub-\( \mu \)XCT-data set, where the detectable features, pores, Fe-aluminides and Mg2Si, are shown with a higher magnification. The results for the AlSi12Ni1 sample show once again, that \( \mu \)XCT gives rather diffuse pictures with a much lower resolution of detail but a rather low noise, whereas sub-\( \mu \)XCT and sXCT give much sharper data with more details and a better contrast. Also sXCT shows a phase contrast for the AlSi12Ni1-dataset which results in a more pronounced representation of the interfaces and edges. In addition, the sXCT-data present less noise compared with those of sub-\( \mu \)XCT.

Fig. 2. Comparison of cross section of the steel sample (scan B) between \( \mu \)XCT with (3.5 \( \mu \)m\(^3\))/voxel (left) and sub-\( \mu \)XCT with (0.5 \( \mu \)m\(^3\))/voxel (right). The CT-data are beam-hardening corrected.

Fig. 3. Different contrast levels detected in the sXCT-scan: (a) pore, (b) low-density inclusion enlarged from Fig. 1(a) and (c) pore and low-density inclusion, (d) pore surrounded by a low density and a high-density inclusion.

Fig. 4. Comparison between \( \mu \)XCT, sub-\( \mu \)XCT and sXCT of an AlSi12Ni1 sample; cross-sectional CT-images are shown. The voxel sizes are 3.5, 0.5 and 0.3 \( \mu \)m, respectively.

4. Discussion

Different features of the CT-data were analysed qualitatively and quantitatively in order to compare the cone beam XCT and synchrotron XCT.

Measurement artefacts and detection of details are now discussed from the point of view of quality. Beam-hardening artefacts cannot appear in the sXCT-data due to the monochromatic nature of the source used. To a certain extent, beam-hardening artefacts are present in the cone beam XCT-data of
steel, but these artefacts can be corrected so that the grey-values for steel are as constant as in the synchrontron XCT-data. Both cone beam XCT and synchrotron XCT-data show ring artefacts, but they do not appear very prominently. Only in the sXCT-data of the steel sample the ring artefacts are distinctive. Ring artefacts are well known and caused by faults of the detector pixels [1,2].

In the detection of details μ-XCT shows worst performance. μ-XCT can distinguish between air and steel as well as between steel and inclusions but it is not possible to differentiate between pores and inclusions. For the Al-sample μ-XCT can tell apart between air, Al-matrix and the higher absorbing Ni- and Fe-aluminides. These restrictions are caused by the limited spatial and contrast resolution of μXCT. Sub-μXCT and sXCT display the best performance in detection of details. It is possible to identify four different phases using sXCT (air pores, low density inclusion, steel matrix and high density inclusion) in the steel sample. For the AlMg5Si7 sample, sub-μXCT and synchrotron-CT datasets reveal four different phases: air pores, Mg-Si, AlMg-matrix and Fe-phases. The spatial resolution of sub-μXCT and sXCT seems to be very similar, but the rather high noise which is always present in the sub-μXCT data and the better contrast in the s-XCT-data achieved by phase contrast leads to higher quality images for s-XCT.

The following properties were evaluated quantitatively: sharpness, contrast, signal-to-noise ratio and full width at half maximum of the material peak in the grey value histogram. The results are shown in the next figures for the three investigated materials using the various XCT-methods.

Fig. 5. Comparison between (a) μXCT, (b) sub-μXCT and (c) sXCT of an AlMg5Si7 sample. The voxel sizes were 3.5, 0.5 and 0.3 μm, respectively.

Fig. 6. Detailed CT-cross-sectional picture showing three different phases in the sub-μXCT measurement of the AlMg5Si7 sample. The voxel size was 0.5 μm.

Fig. 7. Comparison of the sharpness-values at the exterior edges of steel, AlSi12Ni1 and AlMg5Si7 investigated by μXCT, sub-μXCT and synchrotron-XCT. The sharpness is presented in relative numbers. Value 1 equals a sharpness of 25,700 grey-values per μm. The strong phase contrast contribution has to be taken into account for the sXCT-data of the Al-samples.

Fig. 8. Comparison of the contrast-values of steel, AlSi12Ni1 and AlMg5Si7 investigated by μXCT, sub-μXCT and synchrotron-XCT.
values between cone beam XCT and sXCT can be compared qualitatively but not quantitatively. It is clear that the phase contrast gives steeper edges and therefore high sharpness values. The sharpness-data for steel-Fe and for the cone beam XCT results can be compared also quantitatively. As expected from the results shown in Figs. 1–5, the sharpness increases from $\mu$XCT to sub-$\mu$XCT and to sXCT for all samples. This behaviour is more pronounced for the results of the Al-based samples due to the strong phase contrast within the sXCT-data.

An evaluation of the contrast is shown in Fig. 8, where the relative contrast for the different XCT-datasets is depicted. For steel and the AlSi12Ni11, the best contrast is given by sXCT, whereas for the AlMg5Si7 samples the best contrast is obtained by sub-$\mu$XCT. This is caused by the amount of phase contrast in the sXCT-data for the AlMg5Si7-sample.

The signal-to-noise ratio (SNR) for the different measurements is shown in Fig. 9. Usual measurement-times for the various different methods were used. For $\mu$-XCT the measurement time was around 120 min, for sub-$\mu$XCT around 180 min and for sXCT 15 min. The highest SNR-values are for the $\mu$-XCT and for the sXCT measurement, depending on the material and on the measurement parameters. A comparison of the SNR for a definite measurement time reveals the best results for sXCT. In all three cases, the SNR is lowest for sub-$\mu$XCT since in this case the intensity of the X-ray source is lowest.

A further common method for the evaluation of CT-data is an analysis of the grey-value histogram: for this evaluation the features of the air and material peaks are analysed. Only an evaluation of the histogram of steel was meaningful due to the strong phase contrast obtained for the Al-alloys. The results are shown in Table 4. Here, where the full widths at half maximum of the material peaks relative to the distance between the air peak and the material peak for the dataset of Fig. 1 are listed. It can be seen, that $\mu$XCT and sXCT have the lowest values. This demonstrates the lower noise and fewer artefacts for these two data-sets. The material peak for the sub-$\mu$XCT-data set is broadened due to higher noise and due to beam hardening effects.

The investigations presented here show, that cone beam XCT at high resolution can give comparable results to synchrotron-XCT. Sharpness and contrast resolution for cone beam XCT can be almost as good as for sXCT-systems. The biggest problem with sub-$\mu$XCT devices is the rather low intensity of the nano-focus tubes which results in high noise and low SNR-values. However, sub-$\mu$XCT offers many other advantages, especially reduced measurement costs, a usually higher scanning volume, better accessibility and user-friendliness. The results and conclusions of the investigations presented here are summarised in Table 5.

5. Conclusions

Three X-ray computed tomography methods, namely cone beam XCT-systems with a $\mu$-focus and a sub-$\mu$m (nano-focus) X-ray source as well as synchrotron computed tomography, were
A steel sample and two Al-samples (AlSi12Ni1, AlMg5Si7) containing different inhomogeneities and material phases were measured. CT-measurement with voxel sizes in the range 0.3–3.5 μm were performed. CT data features like beam-hardening, detection of details, sharpness, contrast, signal-to-noise ratio and the grey value histogram were evaluated and compared. The results are:

- Beam-hardening artefacts were present in cone beam XCT-data of the steel sample, but can be corrected fairly easily.
- sXCT and sub-μXCT give data with the highest detection of details, sharpness and have the best spatial and contrast resolution. In several cases sXCT and sub-μXCT gave very similar results.
- μXCT is always inferior with regard to detection of details, sharpness, spatial and contrast resolution.
- sub-μXCT suffers from a rather bad signal-to-noise ratio due the limited power of X-ray tubes with focal spots below 1 μm.

All in all, sXCT gives the best results, but the quality of sub-μXCT-data comes quite close in certain cases. This means that high resolution cone beam XCT is a very useful and attractive method for materials science and industrial research, since the costs are much lower and both accessibility and user-friendliness are much better than for sXCT.

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