nanoCT®

Visualizing internal 3D structures with submicrometer resolution

Eberhard Neuser, Alexander Suppes

phoenix x-ray Systems + Services GmbH, Wunstorf, Germany
Phone: +49 5031 172 170, Fax +49 5031 172 299; eneuser@phoenix-xray.com

Abstract

High-resolution Computed Tomography widely expands the spectrum of detectable internal micro-structures. The new nanotom is the first 180 kV nanoCT system worldwide which is tailored completely to highest-resolution applications in material science, micro mechanics, electronics, geology etc. The CT results demonstrate the possibility to analyse the 3D-microstructure of material samples with minimal preparation and the exceptional resolution of less than 0.5 microns per voxel.

Keywords: X-ray, Computed Tomography (CT), reconstruction, beam hardening, nanofocus, microfocus, nanoCT, micro-CT, metrology

1. Introduction

Thanks to its 180 kV nanofocus tube the nanotom is not only suitable for low absorbing materials like organic and mineral samples and ceramics or synthetic materials, but also for nanoCT-examinations of metals or composites containing metal. It is also very useful for high resolution inspection and failure analysis of MEMS, glass fibre devices and especially of complex micro electronic components with concealed parts such as capacitors or stacked dies. The maximum object size is 120 mm in diameter and a weight up to 1 kg.

Computed Tomography with such high spatial resolution requires careful consideration of all factors which might influence the resolution, including the focal spot size of the X-ray tube, the influence of movements, vibrations and temperatures as well as the parameters of the detection system and even the reconstruction algorithm. If the CT system is coordinated very well, any internal difference in material, density or porosity within a sample can be visualized with highest resolution and data like distances can be measured. For example, it is possible to image different metal phases of alloys, to analyze the texture of fibers sized only a few microns in diameter in composite materials or to analyze the normally hidden pore network of small light metal castings or rock samples. Especially for materials which are too soft, fragile or porous for mechanical slicing, nanoCT even offers possibilities for non-destructive analysis which aren’t possible yet for microscopy.

NanoCT widely expands the spectrum of detectable micro-structures. The nanotom opens a new dimension of 3D-microanalysis and will replace more destructive methods – saving costs and time per sample inspected.
2. Motivation

The introduction of nanofocus® tubes increases the image quality of radiographic images further more. An example (shown below) with different focal spot size shows clearly the enhancement in spatial resolution. By using these high resolution tubes in Computed Tomography a further increase of quality regarding spatial resolution should be possible.

Figure 1: resolution increase due to nanofocus®-tube technology
In computerized tomography resolution and object size are generally coupled to each other. The better the resolution the smaller the sample size will be. A rough schematic showing the size ratio is shown below in figure 2.

In microCT resolutions in the µm-scale are state of the art. Resolutions in the sub micrometer scale have been dedicated to synchrotron technique. The introduction of high resolution nanofocus®-tube technology allows focal spot sizes below one micrometer. Therefore the resolution of CT-systems equipped with such high resolution nanofocus®-tubes could be further increased. So called nanoCT®-Systems are pushing forward into application regions which have been exclusive to expensive synchrotron technique so far.

Figure 2: resolution and typical object sizes

3. nanoCT-System – nanotom®

3.1 nanotom®

The nanotom (shown in figure 3)- the first high end nanoCT-Sytem word wide – is especially tailored for highest CT resolution. The whole system design was made for CT measurements in the sub micrometer scale. Therefore a highly stable granite based manipulation system and also a special nanofocus X-ray tube stabilized for CT applications have been chosen. Also the other components including the control software datos|x (which handles all relevant steps like acquisition, filtering, reconstruction, visualization, etc.) are optimized for this purpose.
**Specifications**

- Designed for resolution in the nanometer scale (voxel sizes up to 500nm)
- 180kV nanofocus<sup>®</sup> tube - CT Edition stability optimized
- granite based manipulation including high precision rotation stage with air bearing
- 5 MegaPixel flat panel detector
- Sample diameter from 1-120mm for a broad range of applications
- datos|x software covering all CT-tasks like: taking projection data, reconstruction, filtering, visualization, …
- applications in material sciences, electronics, metrology, …
- compact cabinet design 160 x 142 x 74 cm<sup>3</sup>
3.2 nanotom® system resolution – microCT versus nanoCT®

To show the improved resolution in CT a comparison of microCT and a nanoCT with an identical sample and parameters was performed. Only the focal spot size of the tube is different. The sample was a dried fern with a diameter of 0.8mm. The voxel size in both CTs was 500nm. The reconstructed slices show a clearly visible improve of spatial resolution due to sub micrometer focal spot.

Figure 4: microCT of a dried fern
Figure 5: nanoCT® of a dried fern

nanoCT®:
voxel size: 500 nm
focus spot size: 800 nm

Figure 6: A comparison - nanoCT® versus microCT
3. Metrology

Modern CT-Systems allow to perform metrology tasks on the reconstructed volumes and surfaces. The process chain for metrology could be described as follows (see figure 7).

![Figure 7: Process chain for metrology](image)

After taking a complete set of 2D-Projections the volume reconstruction is performed. The volume is modelled as a three-dimensional matrix of voxels, where each voxel value represents the corresponding local attenuation coefficient of the scanned object. To get high quality results, certain correction techniques should be applied onto the projections in order to reduce the artefacts that are caused by scattering of radiation and beam hardening.

After the volume is reconstructed, the next critical step is the extraction of the surface model from the volume model. The surface model is described by a triangulated mesh or point cloud and is a standard interface for many inspection tasks like metrology or for reverse engineering. After the surface is extracted, measurement tasks like variance analysis or geometrical measurements including fitting of geometrical primitives can be performed on the surface data.

3.1 Advanced surface extraction

The commonly used technique for surface extraction is the ISO-Surface method. With this method, a threshold greyscale defining the margin between two materials (usually between the air and the object) is set by the user. Voxels with the greyscale equal to the chosen threshold value form the ISO-Surface, whereby subvoxel accuracy can be achieved. From the detected surface points (called vertices) a triangulated surface mesh can be calculated.

The described method is fast but has also a very strong drawback. Namely the reconstructed volume can be affected by some artefacts due to beam hardening or scattered radiation. In this case, the margin between air and material does not have the same greyscale for every volume region (Figure 8).

![Figure 8: Surface disturbance caused by artefacts (Al-sample with screws)](image)

On the left, the volume is shown which is affected with artefacts caused by steel screw in aluminium. In the middle, a volume slice at the bottom of aluminium part is shown. Voxels that belong to the extracted ISO-surface are displayed with yellow colour. As can be clearly seen from the figure, the surface is not at correct position: it is shifted...
locally into the object or in the air depending on the position in the volume. The explanation for this behaviour can be given with the profile along dotted line (shown on the left in the figure). The profile part that belongs to the aluminium should have constant grey values. But a significant deviation can be observed.

To avoid this drawback a new advanced surface extraction method was developed by phoenix|x-ray. This method utilizes a global greyscale threshold value as well as information about greyscale distribution in a local neighbourhood of every voxel. The figure 9 shows the comparison of the classic ISO-surface method and the new advanced method.

The figure 9, left shows a reconstructed volume of aluminium part with steel screws in it. It is clearly visible, that the bottom plane is affected by artefacts. From this volume the surface extraction was performed with two methods: with classical ISO-surface method and with the new advanced method. On both surfaces, a plane was fitted as shown in figure 9, in the middle. After that, the deviation of each surface point to the fitted plane was calculated and colour coded. The deviation distribution clearly shows that the surface which is extracted using the new advanced method much less affected by artefacts. This is illustrated in the figure 10 by using surface profiles.
If a volume contains different materials only one margin between two materials can be extracted using ISO extraction method. In contrast to this, the new advanced method can also be used for extraction of surfaces between all materials simultaneously (figure 11).

![Figure 11: Simultaneous extraction of surfaces between different materials](image)

This figure shows an example of a sample consisting of two materials: titanium, coloured grey in the volume, and Trovidur-plastics, coloured red. The new advanced method is able to extract all three margins at the same time.

Another drawback of the ISO-surface method is that the threshold value is user-dependent, with the consequence that the repeatability of measurements can suffer. Another advantage of the new advanced method is the settings for the surface extraction are estimated automatically without any user interaction. This is especially important for the reproducibility of the results of CT-scans, e.g. for GR&R-tests.

### 3.2 Image correction techniques

As already mentioned in the previous section, volume artefacts caused e.g. by beam hardening and radiation scattering can significantly disturb the surface extraction. These physical effects result in the fact that the air as well as the scanned object has inhomogeneous greyscales. In order to reduce this problem, correction techniques should be applied either on the volume or on the projections.

The next figure (figure 12) shows a calibrated sphere plate with dimensions of 20×20 mm and with 4×4 sphere calottes. This object was used in experiments to demonstrate the influence of the beam hardening correction technique on the surface extraction. The advantage of this object is that the plate and the spheres are very exact manufactured regarding the position and deviation from the ideal sphere form. The sphere plate is designed and calibrated by the PTB – German national metrology institution and is used to evaluate the performance of CT-based metrology. The calibration uncertainty for the position of the central points of the spheres is less than 1.5µm and the form uncertainty is less than 2µm.
The middle part of the figure 13 shows three planes fitted on the surface that was extracted from the volume where no correction techniques were applied. The colour coding is used to indicate the deviations of the surface points from the fitted planes. Strong deviations are visible, they indicate position-dependent convexities and concavities on the bigger plane that should be planar. Small planes at the side of the plate show strong bends of the surface as well.

To analyse the effect of correction techniques, the same raw projection data were used and image correction techniques were applied to reconstruct a “better” volume. From the volume the surface was extracted and three planes were fitted as described before. The right image in the figure 13 shows colour coded deviation between fitted planes and the surface whereby the same colour range is used. As can be seen, the deviations are much weaker and the surface that corresponds to the planes is not as bended as before.

The figure 14 illustrates again the advantage of applying image correction techniques. The spheres were fitted in the extracted surface and the deviation between spheres and surface points is computed. On the left no correction techniques were applied and strong position-dependent deviations are visible. In contrast to this, on the right side appropriate results after using of correction techniques are shown. One can see that artefacts are significantly reduced.
3.3 Characteristic values for Metrology

According to ISO 10360 [1] following important characteristic values are used for metrology (e.g. with optical systems): probing error – form (PF), probing error – size (PS) and Error for indication of size measurement E. It is worth to mention, that there exist no international standards for measurements with CT and for determining of characteristic values describing the performance of a CT based metrology instrument. Nevertheless it is possible to perform metrology tasks with CT, as it will be shown in this section. As test artefacts, spheres and sphere plates are usually used for determination of characteristic values. For this reason the sphere calotte plate described above seems to suit well for measurement of characteristic values for CT.

3.3.1 Probing error form

To evaluate optical metrology instruments, test spheres made of ceramics or steel are used to determine the probing error. Other materials are permitted as well. According to ISO 10360, gaussian regression sphere is computed from measurement points i.e. surface points that are probed optically. The regression sphere is determined according to the least-squares method. The characteristic probing error PF is the range of radial deviations between the measurement points and the calculated regression sphere:

\[ PF = R_{\text{max}} - R_{\text{min}}, \]

where \( R_{\text{max}} \) and \( R_{\text{min}} \) is the maximum resp. the minimum distance of a point from the sphere centre. At least 25 points should be taken for determination of PF.

Instead of a sphere, a single calotte of the calotte plate described above is taken for measuring of \( PF \). For a reconstructed volume with a voxelsize of 30µm a value for \( PF \) of 40µm was measured. For the fit of the regression sphere ca. 35,000 points were taken as well as for the measuring of radial deviation to compute the \( PF \) value. Since the surface datasets extracted from CT come up with a very high number of points, taking much more than 25 points seems to be reasonably.

3.3.2 Probing error size

As an additional characteristic of the probing error, the error in the diameter, PS, is determined. This quantity is obtained from the difference between the measured and the calibrated diameters:

\[ PS = D_{\text{meas}} - D_{\text{cal}}, \]

with \( D_{\text{meas}} \): measured diameter of the sphere and \( D_{\text{cal}} \): the calibrated diameter.

For the calibrated sphere calotte plate, for every calotte, a sphere was fitted and the \( PS \) value was calculated. Figure 15 shows the results for surfaces extracted with standard ISO method and with advanced surface extraction method described above.
In the diagram, the calculated deviation is shown for every calotte. It is clearly visible that with the advanced surface extraction method, a significant reduction of the probing error $PS$ can be achieved. With this data, $PS$ can be specified as $50 \, \mu \text{m}$ if standard ISO method is used, and $PS = 10 \, \mu \text{m}$ in the case of advanced method. Another effect can be seen from last diagram: In the case of ISO method, the deviations show some kind of periodical behaviour depending on the calotte place. This can be explained with the help of local distribution of deviations between the fitted sphere and surface points, compare with figure 14, left part. This deviation distribution shows a strong dependence on the calotte position in the plate.

### 3.3.3 Accuracy of size measurement

To determine the accuracy of size measurement, artefacts like ball bars with two or more balls or ball spheres can be used. So the sphere calotte plate is well suitable to examine the accuracy.

To do this, all 16 spheres are fitted and the measured distance between the centres of each sphere is compared with the calibrated distance. All together, 120 distances are evaluated. Figure 16 shows the deviation between measured and calibrated distance vs. the distance value itself.
From these diagrams, the accuracy of size measurement using ISO surface can be specified as ±6 µm and using the advanced surface extraction method ±3 µm.

4. Application examples

4.1 Material science

A typical task in material sciences is the investigation of drilling cores. In order to avoid time consuming and destroying procedures like polished specimen techniques the use of non destructive CT is an alternative. Below a reconstructed slice of an oolitic carbonate is shown. In this case information regarding the pore network and the inner structure of the carbonate was examined.

Figure 16: Oolitic carbonate

With courtesy of IFP (Paris, France)
4.2 Electronics – Light Emitting Diode (LED)

In electronics the miniaturization process needs high resolution test equipment in the µm scale. Especially in packaging development detailed microscopic 3D imaging techniques are needed. The following example shows a visualization result of a CT scanned Light Emitting Diode. Within this high resolution 3D data a material distinction with Cu, Au and Si is possible.

![Figure 17: visualization of reconstructed LED](image-url)
4.3 Electronics – SMD inductor

Another electronics example is the examination of a defective SMD inductor. The part has a size of 2mm x 1.2mm (0805 package size). The CT reveals the complex inner layer structure of the inductor. An investigation with 2D X-ray imaging did not show the broken layer. The CT investigation with the nanotom® clearly resolves the broken layer.

Figure 18: SMD inductor – (package size 0805; 2mm x 1.2mm)

Figure 19: layer structure of the inductor

Figure 20: defect layer
4.4 Connection technique - laser welding

Modern connection techniques like laser welding are in an interesting application for CT. With non destructive CT it is possible to study the quality of the weld and the pore distribution. An example is shown in the images below.

Figure 21: Laser-welding
4.5 Automotive – λ-sensor

In automotive industries more and more complex parts are used. As an example a λ-sensor has been scanned with the nanotom\textsuperscript{®}. In this case the complex inner geometry of the sensor could be inspected without destroying the sensor. Other topics are the condition of the oxygen sensor itself and the electrical connection of the sensor.

Figure 22: λ-sensor
5. Conclusions

The first industrial nanoCT – System nanotom® enlarges the resolution of industrial cone beam CT to the sub micrometer scale by using state of the art nanofocus™ tube technology.

With correction methods like beam hardening correction and advanced surface extraction the quality of the nanoCT scans could be further improved. This is especially needed in new CT application areas like metrology. As an example a sphere calotte plate from the National German Metrology institute (PTB) was scanned with the nanotom®. The metrology results for the accuracy of the size measurements are in the range of 3µm which is 1/10 of the voxel size.

For a wide range of applications the benefit of nanoCT® could be shown.

References

1. ISO 10360-2, Geometrical Product Specifications (GPS) Acceptance and reverification tests for coordinate measurement machines (CMM), Part 2: CMM used for measuring linear dimensions.