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Laboratory X-ray tomography for non-destructive testing of specimens and materials at the nanoscale

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Abstract

Advances in miniaturization from micro to nano-scale have had dramatic impacts on our lives. Especially consumer electronics, material sciences, chemical engineering, or biology are strongly profiting from nanotechnology. However, the tremendous achievements in all of these areas would not have been possible without corresponding material analytics techniques.

The NanoXCT project funded by the European Union’s FP7 programme develops a compact X-ray computed tomography system for laboratory use, which allows for a non-destructive and fully three-dimensional characterization of specimens and materials from micro to nanoscale. The targeted specifications yield a wide field-of-view of up to 175 µm at a voxel size of 50 nm and 1 mm at 285 nm voxel size. NanoXCT facilitate these targets by a custom developed NanoXCT X-ray source in combination with a high precision focusing and emission system. Furthermore, a highly sensitive, photon counting, wide field-of-view, small pitch X-ray detector concept is included in the system. The concept is completed by a precision manipulation system, which allows for alternative scanning geometries. Furthermore a software environment was developed for large data processing and analysis of the generated scan data.

In this work we introduce the NanoXCT system on component level and present the achieved results on various applications. The NanoXCT software tools, which are purpose developed for this application will be induced and applied on the data generated.

Keywords: Industrial 3D X-ray computed tomography, Nano X-ray computed tomography, NanoXCT, Non-destructive testing

1. Introduction and Motivation

Conventional material analytics for nano-scale characterization covers a wide spectrum of different techniques ranging from destructive methods (e.g., focused ion beam FIB), surface inspection methods (e.g. scanning electron microscope SEM, Atomic force microscopy AFM), to 2D methods (e.g. X-ray Diffraction Analysis XRDA). All these techniques share the fact, that they focus on individual aspects of materials characterization and consequently they are not intended to provide a comprehensive representation of a specimen including internal and external 3D-structure analysis as well as a chemical analysis without destroying the sample. The NanoXCT project [1] targeted to overcome this limitation.

In this work we present the results achieved during the lifetime of the NanoXCT project, which was initiated in 2011 and funded by a grant of the European Union’s FP7 programme between May 2012 and April 2015. NanoXCT put its main focus on implementing a novel technique to facilitate fully three dimensional and nondestructive structural and chemical characterizations of internal and external features at the nano-scale by applying a combination of two techniques: X-ray computed tomography for structural characterization and integrated chemical characterization through Multi Energy XCT and K-Edge absorptiometry. The main objectives of the project are found in the design, the development and finally the implementation of a compact X-ray computed tomography system for nondestructive chemical and structural characterization of nano-materials and components. A core requirement for the design of the NanoXCT system since the formation of the project idea was consequently avoiding expensive X-ray optical elements and not relying on a synchrotron source, which would both extend the costs and constrain the application areas of the targeted NanoXCT device. During the project lifetime the following goals were realized:

- Development of a compact, laboratory scale demonstration system. The targeted specifications of this demonstration system, which may be individually reached (in accordance to the application area), are outlined in Table 1.

Targeted NanoXCT demonstration system specifications	
Scanning time:	~ 10 hours
Field of view:	1 mm
Specimen size:	<= 1 mm^3
Voxel size:	50 nm
Analysis modes:	3D structural and chemical analysis

Table 1: NanoXCT demonstration system specifications

- Development of a novel nano focus X-ray source specifically designed for nano-scale characterization.
- Development of a detection system which employs a novel wide field of view small pitch detector concept with photon counting and spectral information capabilities.
- Facilitating nano-scale chemical characterizations by a combination of multi energy XCT and/or K-Edge absorptiometry.
- Design and implementation of NanoXCT reconstruction algorithms
- Design and implementation of specific algorithms to address qualitative and quantitative evaluation of nano scale structural and chemical characterization.

In the following sections the individual components of the NanoXCT device demonstrator are explained. Furthermore we show results of scans from samples of various application areas and conclude with remaining challenges.

2. NanoXCT source

The X-ray source developed by Excillum and the Royal Institute of Technology, shown in Figure 1, is a transmission type nano-focus X-ray tube designed to reach a minimum electron beam spot-size of 100 nm, which would result in a theoretical line-spacing resolution of 50 nm if diffraction effects are disregarded.

To achieve this, it is based on similar electron optics as used in the MetalJet-D2 from Excillum, but with a transmission anode, instead of a liquid-metal-jet-anode. The transmission target consists of a thin tungsten layer on a diamond substrate, where the diamond is used for heat conduction and thermal stability while the tungsten is used for its X-ray emission properties. Thin layers are used to minimize electron diffusion in the tungsten, which would result in a larger spot-size than the initial electron beam spot size.

One of the major mechanical features with the nano-focus X-ray tube is a wedge-shaped front to maximize geometrical magnification with a cone-shaped sample holder (used for mechanical stability, see Figure 4) by allowing a minimal source-object-distance. Another feature is the extensive integrated water cooling, to minimize thermal fluctuations and drift.

Figure 1: The nano-focus X-ray tube, with the wedge-shaped front, which enables a cone-shaped sample holder for increased sample stability. Also visible is the extensive integrated water-cooling.

As illustrated in Figure 2, the nano-focus X-ray tube is able to resolve 150 nm JIMA lines and spaces, where the resolution limit is believed to be driven by diffraction effects.

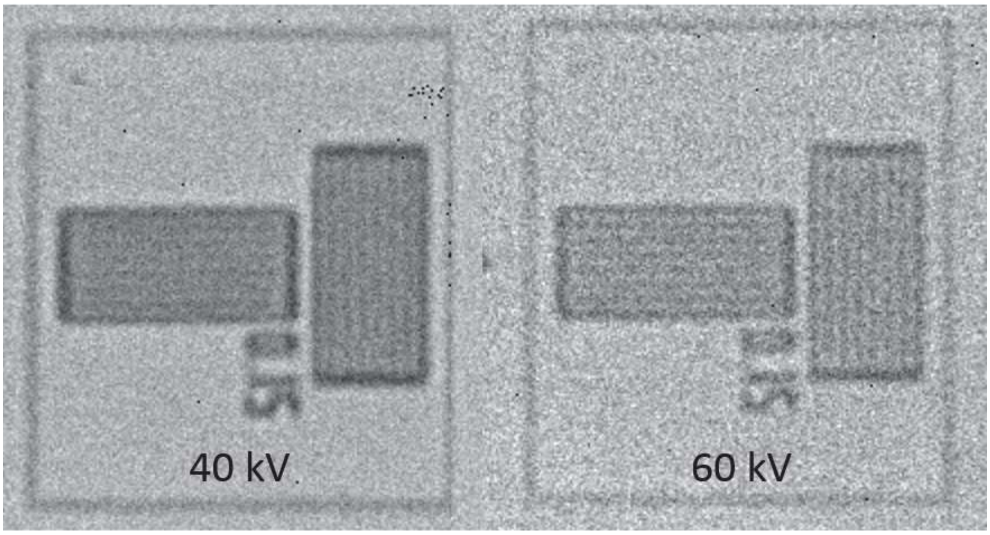

Figure 2: Together with the NanoXCT detector, the nano-focus X-ray source is able to resolve 150 nm JIMA lines and spaces.

3. NanoXCT detector

3.1. Detector requirements

The second key component in the NanoXCT system is the detector subsystem. To achieve the goals of the project, there are several requirements on the detector:

- Low noise especially at low photon flux that is typical for nano focus spot size
- Small pixel size for short focus object distance at given magnification
- Good spatial resolution at given pixel size
- Possibility to obtain spectral information from detector

- At least 3000 pixels wide; multiple modules if necessary

To meet these requirements, a detector working in photon counting mode is suitable, as it can provide low noise even at very low flux, a direct converting sensor layer for good spatial resolution and an energy threshold for obtaining spectral information. Different concepts and base tiles of photon counting detector types were evaluated.

3.2. Detector hardware and software implementation

The detector is based on four Timepix hexa detector modules (see Figure 3). The hexa modules consist of 3×2 Timepix base tiles and have a total of 768×512 pixels with a pixel size of 55 µm. The resulting total detector size is 3072×512 pixels. It provides an adjustable energy threshold for spectral imaging capabilities. An adapter board connecting the Timepix hexa modules with the readout system was designed allowing the readout of all four hexa modules using one Fitpix readout.

As photon counting detectors provide different parameters to be controlled and set compared to standard digital detectors working in integrating mode, a new detector software interface was developed and implemented. To simplify the handling of the detector in routine applications, this interface will not allow access to all parameters to the user but instead restrict these settings to low-level maintenance, automatic calibration and equalization routines.

Figure 3: Left: Open detector housing with the four Timepix hexa modules and auxiliary electronics. Right: Closed detector with entrance window.

3.3. Detector calibration

Aside from a bad pixel selection and masking, routines for threshold equalization and energy calibration were developed and implemented.

The Timepix detector offers 4 bits for threshold equalisation to compensate pixel-to-pixel variations of the actual energy threshold of each pixel due to manufacturing variations. For the generation of the threshold equalization mask a flat-field threshold equalization was implemented. It requires homogeneous illumination of the detector, optimally by the X-ray spectrum which the detector will later be used with. An advantage of the flat-field threshold equalization is the relatively short acquisition time. Also, the equalization can be performed at any energy level above the noise edge and suited to the desired energy threshold.

Furthermore, an energy calibration tool was implemented allowing both chip-wise and pixel-wise energy calibration using $K\alpha$-Lines of elements as e.g. copper, zinc, zirconium, molybdenum, silver and tin. The peaks of these emission lines are detected automatically from a threshold scan and linked to the respective threshold DAC register value. In reversion, this allows to set the threshold DAC value to a calibrated energy value.

4. NanoXCT demonstration system

The NanoXCT demonstration system is a prototype device and experimental platform for reaching the top-level specification of this project. The main objectives for the system integration were:

- Create mechanically and thermal stable design of the complete instrument
- Design an object manipulator with necessary accuracy and stability
- Design a sample holder as well as a positioning device
- Provide a proper integration of X-ray source and detector with associated electronics
- Develop shielding enclosure corresponding to laboratory safety standards
- Integration of Hard- and Software components

The final NanoXCT demonstration system is based on a flexible standard optical table (1800 mm x 900 mm) combined with a shielding cabinet made of a steel-lead sandwich compound. The front door can be opened vertically and allows complete access to the components inside. Two labyrinths on the back side are giving plenty of space for cables, sensors and other equipment. All peripherals like the electrical installation, controllers for the X-ray source and the manipulation system as well as a water cooling system are installed inside a 19” control rack.

The object manipulation system (see Figure 4) is based on nano positioners combined to a stack of stages giving 4 degrees of freedom. There is a magnification axis with a range of 50 mm and two other linear stages with 5 mm range each. The rotational axis is 360° endless. Beside the object manipulation system the device has also a detector manipulation system. Here, also a set of axis was stacked in order to allow a flexible position of the detector such that demands concerning flux optimization and field of view can be met.

The design of the sample holder was mainly influenced by the need for high mechanical strength, suitable X-ray characteristics regarding scatter and attenuation, low cost, easiness to machine as well as a possibility of detaching the sample holder from the system for sample preparation. The final sample holder consists of two elements: One holder base made of brass and one sample holder made of aluminum. While the brass part is mounted to the positioners the aluminum body can be detached and removed from the system for the purpose of sample preparation.

Figure 4: Specimen holder and object manipulation stage of the NanoXCT demonstrator

Controls for all components (X-ray Source, X-ray detector and all manipulators) are integrated into a NanoXCT Software. The user front end was implemented as a graphical user interface (GUI) which allows controlling all components separately in one user interface as well as preparing and starting measurements. This also includes preoperational steps like calibrations of the detector and the positioners but also a first impression of the most recent projection image taken by the detector. Although the system is calibrated mechanically with according equipment to a maximum extent, there are still some remaining static and also non-static misalignments, which become relevant at very high magnifications. Therefore additional software-based correction methods have been developed. In order to compensate for movements of device components during the actual XCT scan several preprocessing steps are performed with hardware and software involved. Such movements are mainly caused by thermal drifts and effects within the X-ray tube, both with long time constants. Besides that, a special phantom with high density structures giving high contrast has been developed that allows the determination of static and reproducible angle-dependent rotary stage errors as well as detector misalignments. This is achieved by comparing actual and nominal particle tracks during an XCT scan of this phantom. The determined misalignments are corrected before the CT reconstruction is started.

5. NanoXCT analysis software

A software environment was implemented to address NanoXCT data analysis requirements:

- Specialized data reconstruction, including reconstruction of compositional information from Multi Energy XCT and K-Edge absorptiometry;
- Large high-resolution data handling
- Characterization of complex nanomaterials
- Visual and quantitative integration of structural and chemical information

The integration platform for NanoXCT data visualization and analysis is the software Avizo [3], dedicated to materials science, digital rock analysis and industrial inspection. A set of extended tools and workflows address image filtering, noise reduction, segmentation, features extraction and measurements. In particular, a new workroom integrates the InSpectr module for spectral and composition data. InSpectr [1] offers a number of techniques for visualization and data analysis of the generated XCT data in combination with spectral data as well as element maps of the same specimen. The implemented techniques within NanoXCT may be subdivided with regard to the following three analysis tasks: Global material composition analysis, local material composition analysis, and analysis of unknown and foreign materials.

Addressing the task of global material composition analysis, the InSpectr module provides information on the elements which are contained in the specimen as well as in which quantity those elements are contained. For this purpose various techniques have been implemented such as calculating and visualizing aggregated spectra as well as histograms of spectral data, overlaying the characteristic energy lines for elements of interest and finally spectral functional boxplots, which adopt the visual metaphor of boxplots to visualize the five important statistical characteristics of numerical datasets: median, first and third quartile, and minimum and maximum values.

Regarding the local material decomposition analysis magic lenses have been implemented, which allow to peek into spectral data or elemental maps given the context information of XCT data in the slice views. Together with the spectral color image as well as the element maps both slicer and the 3D view allow for a fast and easy localization of elements of interest. In addition, the InSpectr module allows for adjusting the transparency of the spectral data or elemental map overlays. Brushing in the spectrum view highlights the regions with spectra leading through the selected region in the XCT slice. Spectrum and concentration probing links information on the local spectrum and composition to the XCT slice view.

Regarding the task of analysing unknown and foreign materials, the InSpectr module allows to display reference spectra in the spectrum view. When hovering with the mouse over the periodic table the corresponding reference spectra as well as the characteristic energy lines an element of interest is shown.

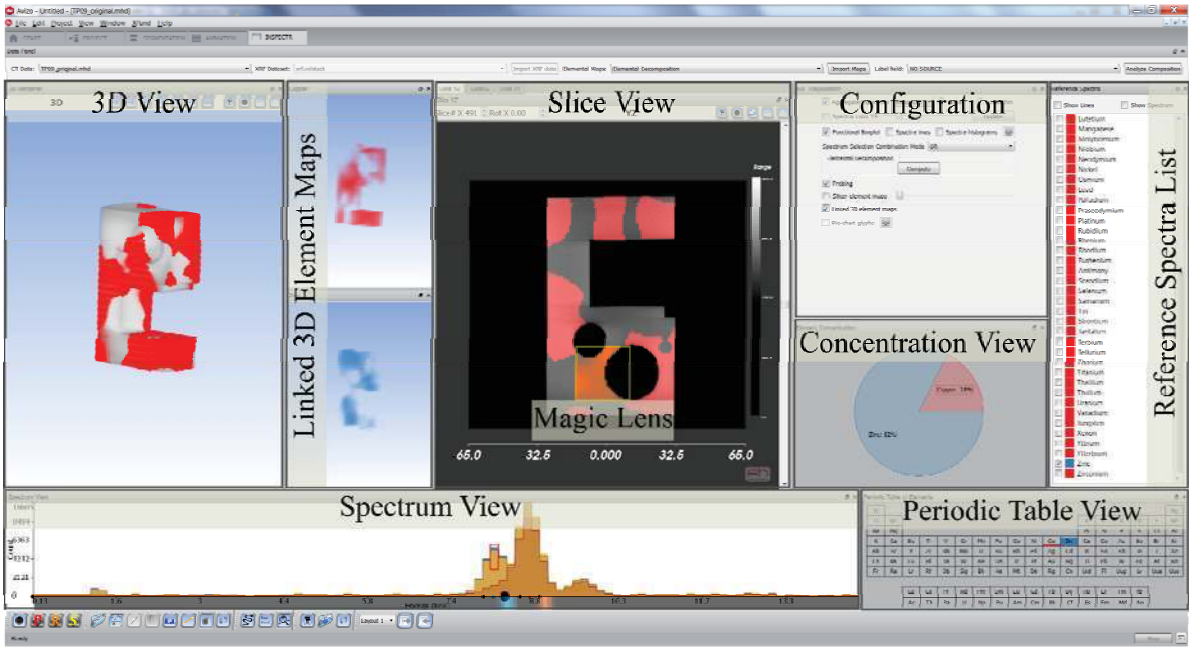

Figure 5: The InSpectr interface contains an XCT slice view, a pie chart and periodic table view showing the element concentration at the current mouse position in the slice view, a reference spectra list and the spectrum view.

When hovering over the slice view, the corresponding spectrum is displayed and the local material decomposition is shown in the centration view. Also the periodic table view allows to locally identify materials. Using these functionalities an easy identification of an element of interest is facilitated. An overview of the InSpectr interface fully integrated in Avizo is shown in Figure 5.

In order to fulfill the goals regarding the chemical analysis features, different approaches were studied, namely fluorescence pin-hole imaging, confocal fluorescence imaging and K-edge imaging. The method of K-edge imaging was chosen because it provides a number of advantages, especially that no additional hardware is required. Our solution combines different aspects of the above mentioned approaches and goes beyond the current state of the art, by acquiring spectroscopic data with a 2D imaging detector, that is then analyzed for K-edges (position in the spectrum, amplitude) to identify and quantify the material composition of the penetrated object, all in the context of micro-scale imaging. The images obtained from the K-edge algorithm represent the areal density of the respective element. The areal density of the element with the K-edge under investigation a_K is the projection of the partial density of the respective element in the object volume. The 2D image data resulting from this step represent the areal density of the K-edge material in one image and the areal density of the residual material in a second image. From the areal density images the 3D tomographic images can be calculated using a CT reconstruction algorithm. As an example silver (Ag) and molybdenum (Mo) wire with a diameter of 50 µm were imaged. In the resulting K-edge images both materials are clearly separated (Figure 6).

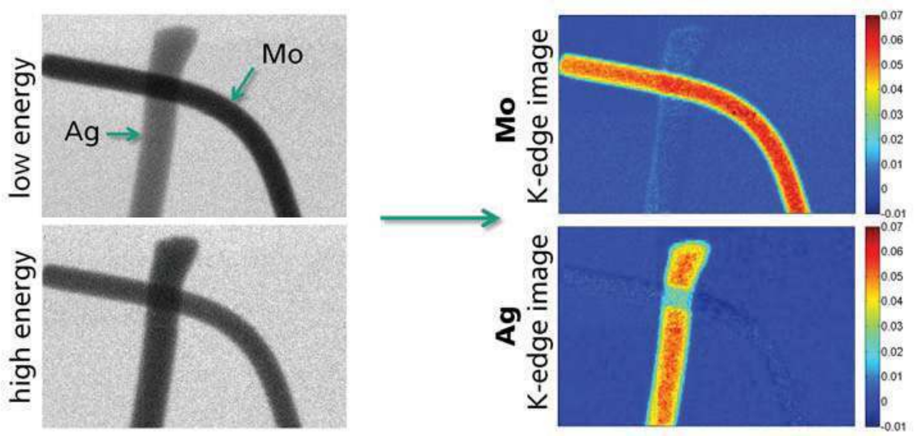

Figure 6: Basic transmission images and resulting K-edge images of Ag and Mo wires with 700 nm pixel size

6. Results

Regarding results we present some of the scans, which were done with the NanoXCT device demonstrator. Figure 7 shows the head of a mosquito. The compound eyes of the insect are clearly visible as well as the parts of the sucker and the leg and even some hair structures.
The second example shows one of the very first scans done with a preliminary setup of all NanoXCT components (see Figure 8). This sample contains polymeric material with nanofillers. The rendering shows mainly shows the nanoscaled filler materials while the matrix is transparent. In the center of the image the top of the sample holder is visible.

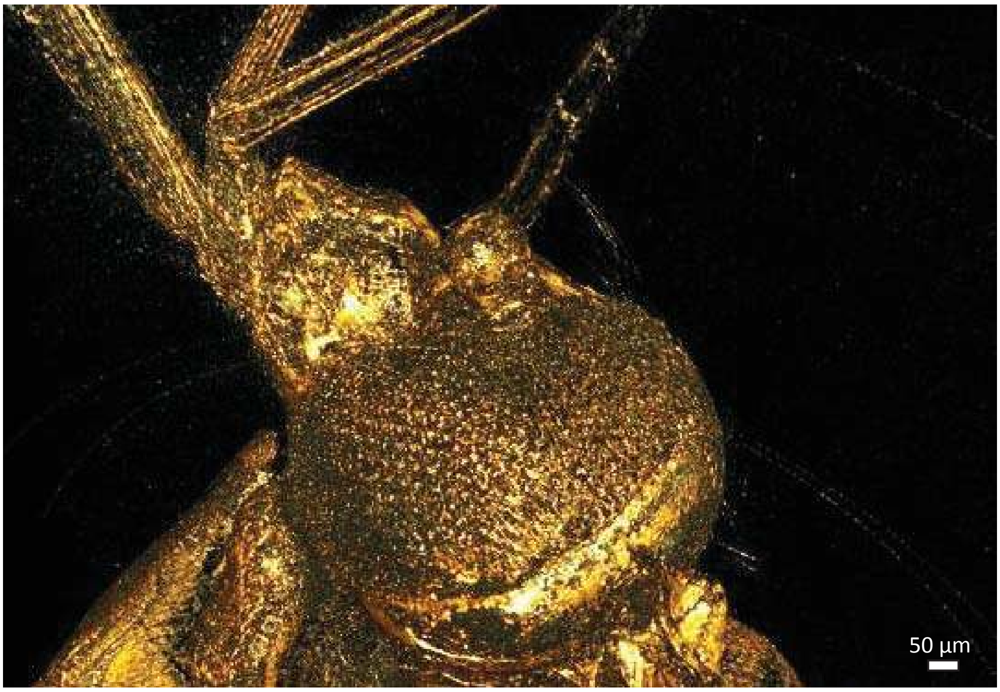

Figure 7: Head of a Mosquito. The compound eyes of the insect can be seen as well as parts of the sucker and a leg

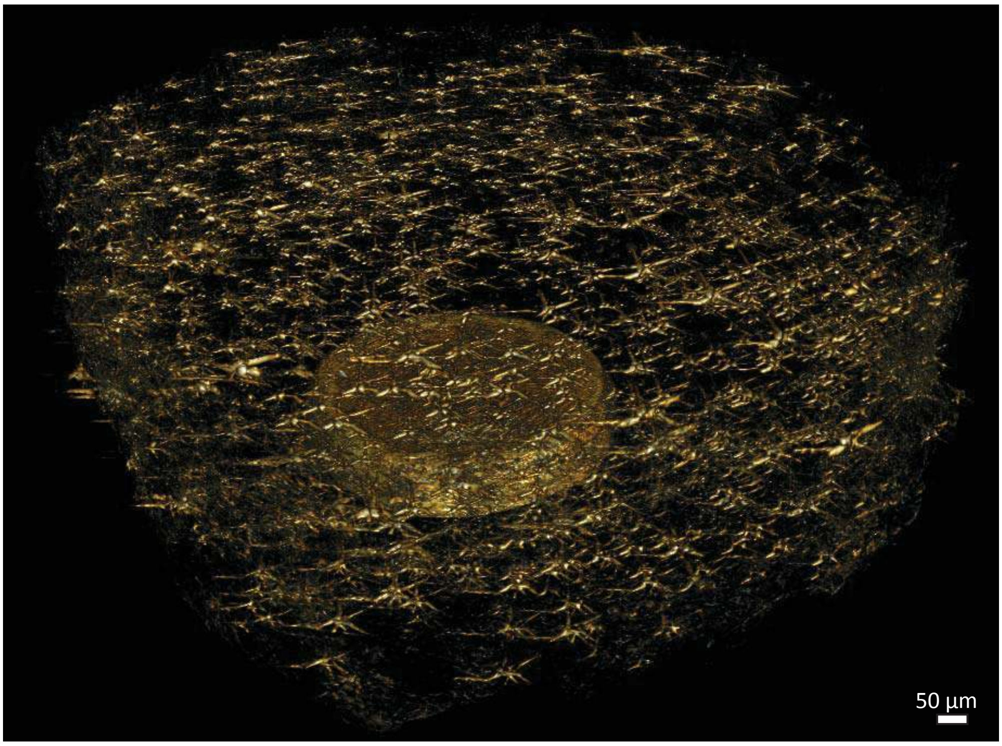

Figure 8: Polymeric material with nanofillers

7. Summary and Conclusions

To summarize, the NanoXCT consortium successfully implemented a compact X-ray computed tomography system for non-destructive chemical and structural characterization of nano-materials and components, which was demonstrated to be fully functional at the end of the NanoXCT project. Now the system undergoes a detailed testing phase to evaluate and determine the full potential of the system. In addition, all components are continuously refined in order to transfer the generated results into a series of NanoXCT products in near future.

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