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Non Destructive 3-D Investigation of Nanocomposite Materials

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***Abstract*-**The paper presents a non-destructive method that aims to reconstruct an image through a nanocomposite material based on the attenuation of X-ray. For analyse we used a commercially available Scyscan 1174 desktop X-ray microtomography unit, that allows us to visualize and measure complete three-dimensional object structures without sample preparation or chemical fixation.

I. Introduction

An acquaintance of the microscopic internal structure of a sample plays an important role in improving to understand properties or function of samples. Usual methods such as electron microscopy or optical microscopy can provide this kind of information in samples which can be sectioned and stained. However, many samples cannot be investigated in this way as sectioning may be impractical or destroy the structures the investigator wishes to image. In such cases, some form of non-invasive imaging is required [1 - 3].

The X-ray computed tomography (CT) represents a non-destructive method that aims to reconstruct an image through an object based on the attenuation of X-ray. The method was developed in the 1970's and was initially intended for medical purposes. This method of imaging avoids several important limitations of conventional X-ray radiology. CT avoids the superimposition inherent to radiographic imaging of producing slices in the third dimension in a non-destructive way with contrast discrimination up to 1000 times better than a conventional radiograph [4 - 6].

In Figure 1 is presented the evolution for X-ray microtomography, which combines the advantages offered by the X-ray tomography, non-invasive method and three-dimensional analyse, and microscopy, which permits a high resolution (μm, nm) of results.

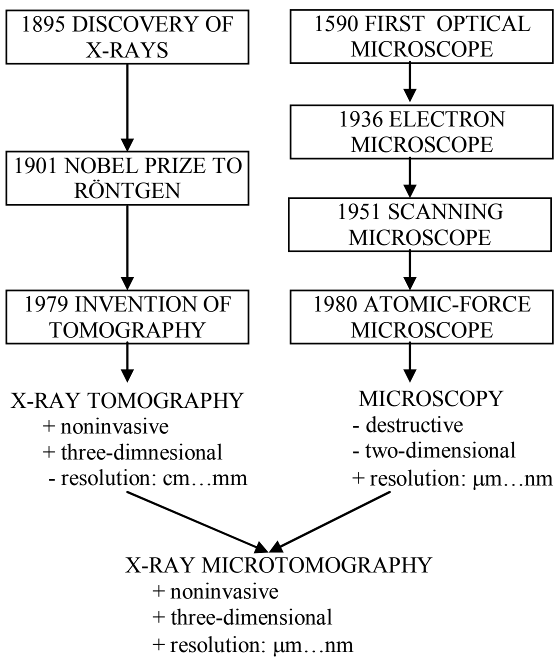


Figure 1. Evolution of X-ray microtomography.

II. Principles of X-ray microtomography

The technique of X-ray microtomography is based on the interaction of X-rays with matter. When X-rays pass through an object they will be attenuated in a way depending on the density and atomic number of the object under investigation and of the used X-ray energies. By using projection images (cf. roentgen photos) obtained from different angles a reconstruction can be made of a virtual slice through the object. When different consecutive slices are reconstructed a 3D visualisation can be obtained.
An X-ray shadow images represent a two-dimensional projection from a three-dimensional object. In the simplest case, we can describe the X-ray illumination as parallel. In this approximation, each point on the shadow image contains the integration of absorption information inside the three-dimensional object along the path of the corresponding partial X-ray beam (Figure 2).

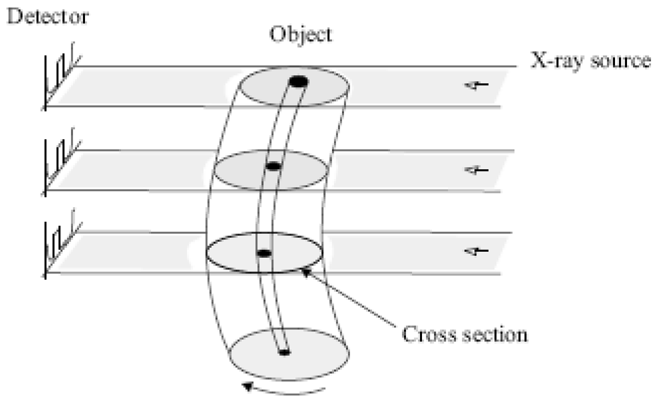


Figure 2. The principle of X-ray microtomographic measurements.

During data acquisition the microtomography scanner collects 2-dimensional X-ray images through the object from different views, typically ± several hundreds images through 180-degree object rotation. Corresponding lines from all views are used afterwards in the reconstruction procedure to create a cross section image by a tomographical back projection algorithm with convolution and correction for fan beam [1, 2]. The set of reconstructed cross sections from all layers contains full information about the object's internal architecture obtained non-destructively. On the base of this data the internal microstructure can be measured for numerical comparison of different objects or for correlation to physical properties. During scanning in micro-CT instruments the object is positioned in normal environmental condition without any preparation.

A tomography scanner records the shadows from many different angles, which are then reconstructed as cross-sectional images through the object. Reconstruction of objects can be illustrated, in simplest case, by an object with only one point having significant absorption. Along the ray path, there will be a decreasing intensity of the shadow of absorption in the object area. The shadow line is recorded in the computer memory as an array of pixels (picture elements), suggesting the area of the possible position of the object. When the object is rotated, another shadow line will add another array of pixels. After several rotations, the position of the absorption point becomes more and more defined and can be localized by superimposing all the shadow lines through a "back-projection" process (Figure 3). This is followed by a process of convolution to eliminate blurred areas around the point, resulting from the superposition of lines in different orientation. In the same way cross-sectional images can be obtained from an infinite number of points and from sequential cross-sectional slices, allowing a three-dimensional image of the object to be reconstructed. In the case of reconstruction from an infinite number of projections one can get an image with a good definition of the absorption area position inside the initial object.

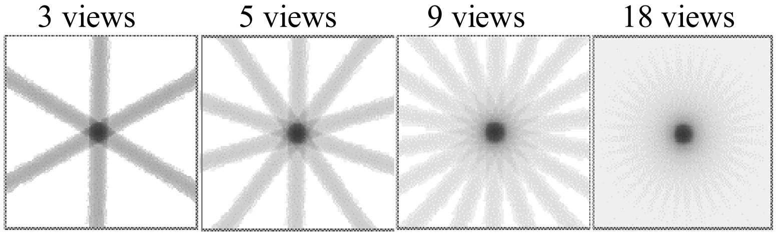


Figure 3. An illustration of back projection process in microtomography for a single point object (Skyscan 1174 Instruction Manual).

III. Results and discussions

The practical analyse was performed with Scyscan 1174 desktop X-ray microtomography for a nanocomposite magnetite sample (polysulfone with magnetite scale of 10nm), which can be used for electromagnetic shields. The sample was imaged at Source Voltage of 50 kV and Source Current of 800 uA, with a 1280×1024 camera resolution, and the scanning resolution set to 10 microns per pixel. Each scan was performed through 180 degrees, with 0.7 degrees per rotation, a frame averaging of 2, and random camera movement set to 10. The X-ray source and the detector remained in a fixed position. The reconstruction of these images into bmp files was performed by NRecon version 1.5.1.1 (also made by Scyscan). All analysis was done with CTAnalyser software version 1.8.1.5 that is an application for deriving quantitative parameters and constructing visual models from scanned datasets obtained with SkyScan micro-CT instruments.

The structure of the experimental system [7] used is that presented in Figure 4.

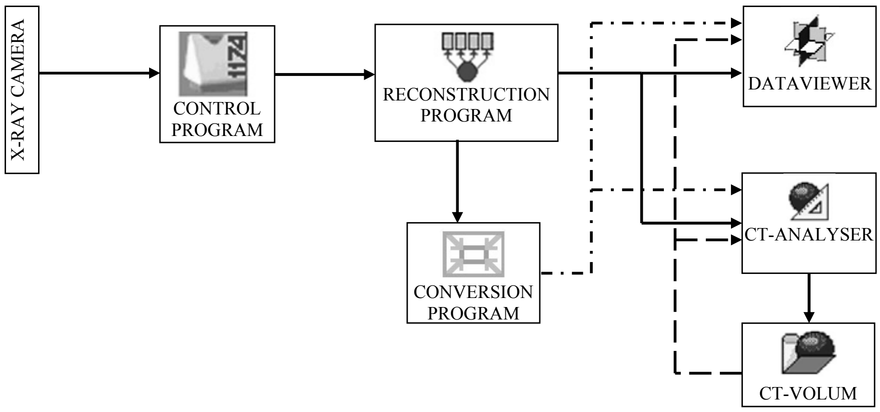


Figure 4. The experimental system.

The control program acquires images from the X-ray camera. During acquisition the control program saves all angular projections which can be opened and shown as a movie by several supplied programs, such as DataViewer and CT-Analyser. Reconstruction is performing by reconstruction program, which uses either a single computer or a computer cluster with several identical or different PCs connected by networks. The reconstruction program works using float-point data values for internal calculations during reconstruction and afterwards permits the operator to define the density window as a range of the reconstructed values. The conversion program allows the size of all reconstructed cross sections to be reduced to a selected fraction of the original size. The CT-Analyser program for 2D/3D processing and analysis can work directly with reconstructed datasets. During rendering the CT-Analyser program can save 3D-models of reconstructed objects for sending them to the CT-Volum program for realistic visualization [7].

After the end of acquisition for all angular projections, we started the reconstruction of virtual cross section slices by clicking the “Start Reconstruction” button in the toolbar. To show reconstruction results we begin the DataViewer program, which displays the reconstructed cross sections as a slice-by-slice animation. We load the full reconstructed 3D space into DataViewer and visualize three orthogonal slices crossing at any selected point inside the object (Figure 5).

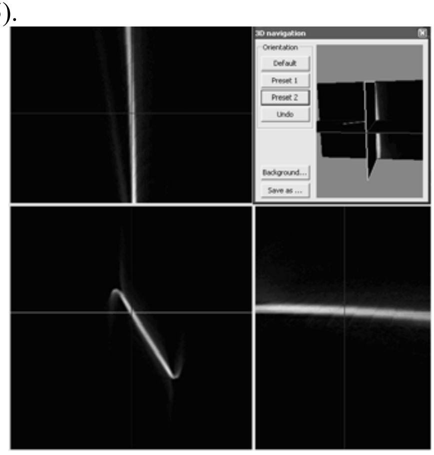
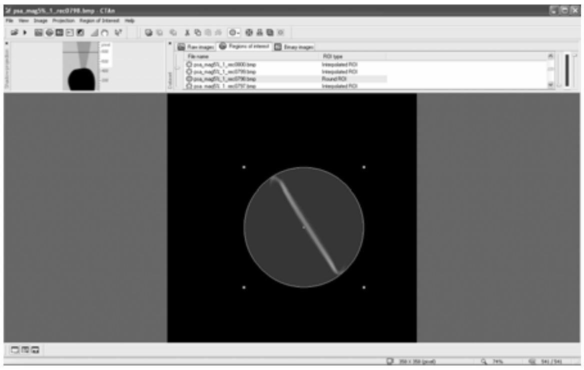
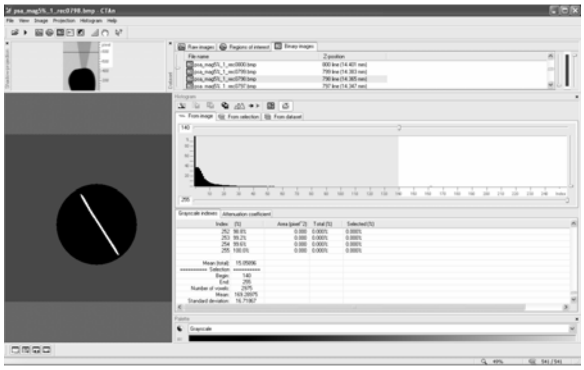


Figure 5. View mode of sample with three intersecting orthogonal sections.

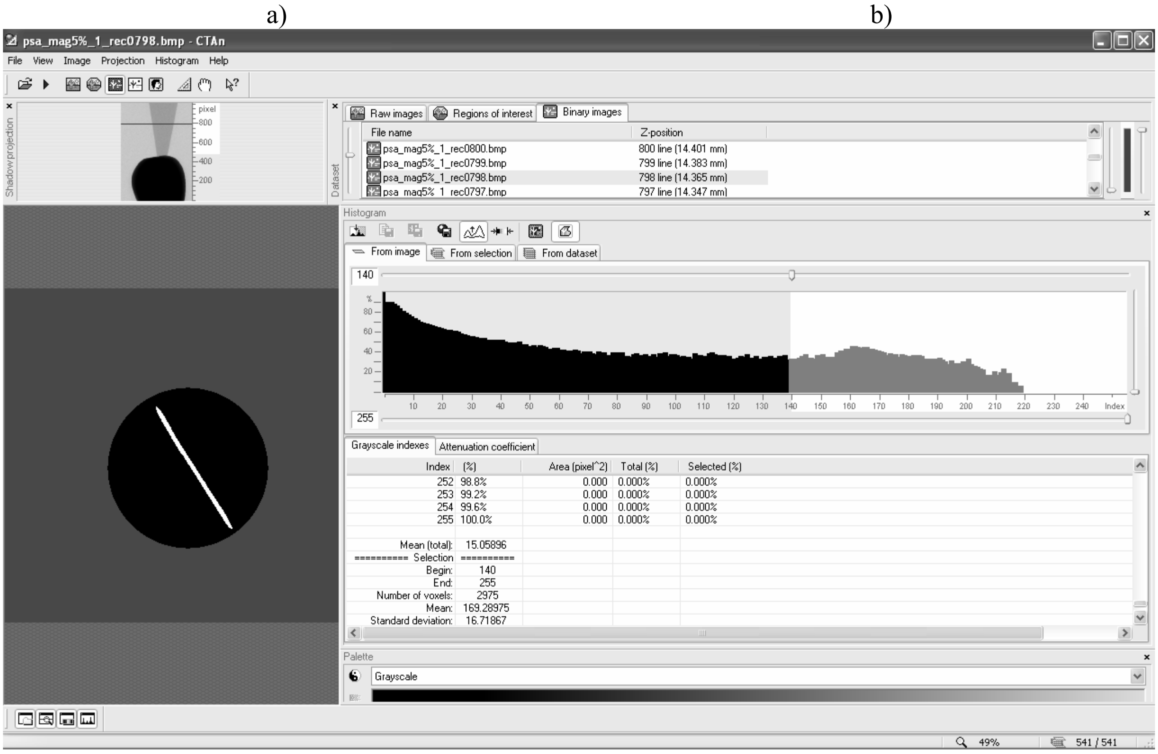
For image analysis and visualization of reconstructed scan datasets we used CT-Analyser program (CTAn) for 2D/3D analysis. In Figure 6 we presented the region of interest and the binary images viewing mode.



a)



b)



c)

Figure 6. a) Region of interest; b) and c) The binary images viewing mode.

We chose a circular region of interest presented in Figure 6a, which allows us to delineate regions of interest on dataset images.

In Figure 6b images white colour represents areas with brightness within the range of the binary threshold selection (“solid”), and the areas outside this selection are black (“space”).

In Figure 6c the Histogram bar is divided in two parts. At the top is a window with the histogram of brightness distribution displayed. Below this is a text table of this distribution.

The histogram table has five columns, which display (1) the absolute values of image brightness, (2) the relative brightness as a %, (3) the absolute value of the image area with this brightness, shown in current units of measurement, (4) the area with a specific brightness as a % of the total area with all brightness values, and (5) the area with a specific brightness as a % of the total area with brightness within the selected range.

At the bottom of the table the mean brightness value from the whole histogram is shown – “mean (total)”. Further data from the brightness histogram are given: the boundaries of the binary selection, the total number of dataset voxels within the binary selection, the mean brightness within the binary threshold selection, and the standard deviation, standard error of mean and the 95% confidence limits relating to the brightness mean value within the threshold limits.

With CTAn program we exported a 3D-model of the object into the CTvol program for visualization (Figure 7). The 3D model created in CTAn we saved in a .p3g file type which has a spatial unit of voxels, and saves a given model with a much smaller file size and opens quicker in CTVol.

When selecting the pore network for the binary images we made possible to create a 3D model by volume rendering (Figure 7). This model can be used for visualisation, rotation, translation, flying-through the object, making avi-files.

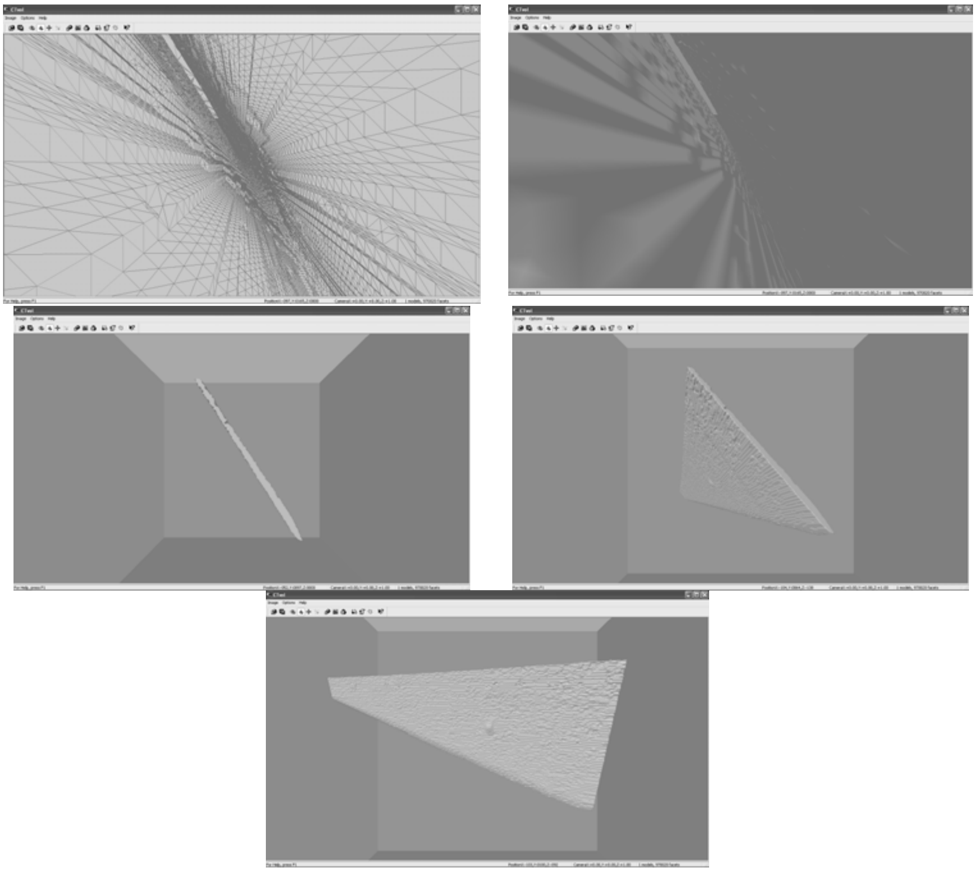
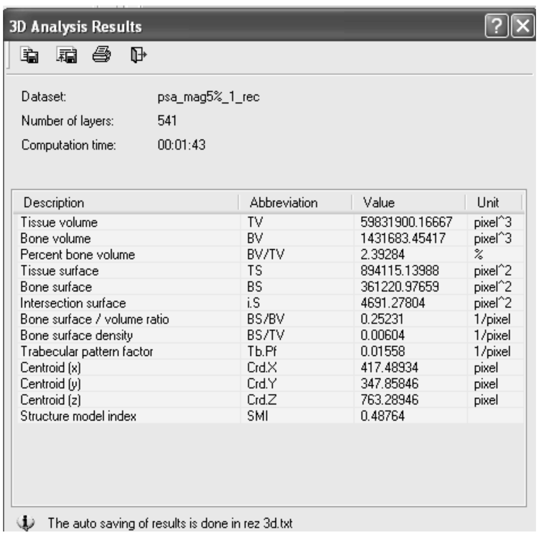


Figure 7. 3D volume rendering of the reconstructed sample.

Using 3D analysis we can obtained for our sample the calculated parameters such as volume, surface density, centroid and others, which are displayed in Figure 8.



3D Analysis Results

Dataset: psa_mag5%_1_rec
Number of layers: 541
Computation time: 00:01:43

Description	Abbreviation	Value	Unit
Tissue volume	TV	59831900.16667	pixel^3
Bone volume	BV	1431683.45417	pixel^3
Percent bone volume	BV/TV	2.39284	%
Tissue surface	TS	894115.13988	pixel^2
Bone surface	BS	361220.97659	pixel^2
Intersection surface	i.S	4691.27804	pixel^2
Bone surface / volume ratio	BS/BV	0.25231	1/pixel
Bone surface density	BS/TV	0.00604	1/pixel
Trabecular pattern factor	Tb.Pf	0.01558	1/pixel
Centroid (x)	Crd.X	417.48934	pixel
Centroid (y)	Crd.Y	347.85846	pixel
Centroid (z)	Crd.Z	763.28946	pixel
Structure model index	SMI	0.48764	

The auto saving of results is done in rez 3d.txt

Figure 8. 3D analysis results.

IV. Conclusions

Applications using X-ray microtomography offer major advantages include five areas: new product development, process control, noninvasive metrology, materials performance prediction, and failure analysis. Microtomography allows relatively inexpensive inspection; with accurate three-dimensional measurements, materials with critical flaws can be eliminated before subsequent costly manufacturing steps. Integrating CT data of as-manufactured components into structural analysis programs seems very promising, particularly for anisotropic materials such as nanocomposite materials.

Acknowledgment

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