LABORATORY SYSTEM FOR X-RAY NANOTOMOGRAPHY

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ABSTRACT

Using advanced X-ray technologies and X-ray scattering enhancement in signal detection, a compact laboratory X-ray scanner for 3D non-invasive imaging with 150-200 nanometers 3D detail detectability has been created. This spatial resolution in the volume terms is equal or better than can be achieved in synchrotron tomography, 5 orders better than in existing laboratory instruments and 10-12 orders better in comparison to clinical CT. The instrument is built using X-ray source with LaB₆ cathode and two electromagnetic lenses. Small-angle scattering enhances the object details up to 150-200nm. An object manipulator allows precision positioning and rotation with accuracy better than 100nm. The X-ray detector is based on an intensified CCD with single photon sensitivity. The typical acquisition cycle for 3D reconstruction of the full object volume takes from 10 to 100 minutes with collection of several hundreds angular views. Subsequent volumetric reconstruction produces results as a set of virtual slices with isotropic voxel size up to 100 x 100 x 100nm or as a 3D-model, which can be virtually manipulated and measured. The object stays in normal environmental conditions without any coating, vacuum treatment or other preparation. Unique spatial resolution in non-invasive 3D-investigation allows obtaining heretofore unachievable 3D images in the wide range of application areas, such as composite materials, carbon-based materials, fuel cells, paper and wood microstructure, biomedical applications, etc.

1. INTRODUCTION

Using modern technologies in x-ray sources, precision mechanics and detection systems, a compact laboratory x-ray scanner has been created for non-invasive imaging of internal microstructure of objects with 150-200 nanometers isotropic three-dimensional spatial resolution. The system allows the imaging of previously unattainable details of internal three-dimensional micro-architecture in a wide range of applications.

2. NANOTOMOGRAPH AS A SYSTEM

The X-Ray nano-CT instrument is based on the same general principles as clinical CT-scanners and all other micro-CT systems [1]. The object mechanically rotates inside the x-ray beam and an x-ray camera acquires the necessary number of angular projections (typically hundreds or thousands) for subsequent reconstruction of the object’s three-dimensional internal microarchitecture in the computer memory using back-projection algorithms. The differences in the new nano-CT instrument compared to previous micro-CT instruments, which allow us to reach submicron resolution, are based on (a) the use of different contrast formation physics and
(b) special selection of all the involved technical methods and devices to achieve stable and high-resolution imaging during the acquisition cycle.

A block-diagram of the nano-CT instrument is shown in Fig.1.

Fig.1. Block-diagram of the Nano-CT system.

The system contains an x-ray source with associated electronics and vacuum system, a multi-axis precision object manipulator, an intensified x-ray camera, a system controller and a computer for instrument control through the USB-port and image acquisition through an IEEE1394 (FireWire) port. All parts of the system operate under computer control from a software package, which includes four programs: 1) system control and data acquisition, 2) three-dimensional cone-beam (volumetric) reconstruction for one PC or cluster with virtually unlimited number of computers based on modified Feldkamp algorithm [2], 3) two- and three-dimensional image processing and analysis and 4) realistic three-dimensional visualization.

All parts of the system include necessary shielding against x-ray leakage and other safety devices. The scanner is of compact design with a small footprint, as shown in Fig.2. More details and specifications of the instrument can be found in [3].
3. IMAGE FORMATION FOR SUBMICRON X-RAY SOURCES

The best known phenomenon in the interaction of an x-ray beam with material is x-ray absorption, shown in the left part of Fig.3. It is used in all clinical x-ray imaging systems and CT-scanners as well as in industrial CT and micro-CT instruments. In the case of source spot-size around and under one micron another fortuitous physical phenomenon exists in contrast formation, shown on the right side of Fig.3 and called “small angle scattering” or “small angle reflection”. Any external surface or internal border between different densities inside the object creates small angle scattering (reflection) of a polychromatic x-ray beam, which (in the case of small spot-size of the source) enhances the image of the object’s external and internal edges. This image enhancement is defined by the spot size of the source and the object-detector distance. The correct choice of the object-detector distance for the high-resolution mode allows selection of the necessary mix or balance of absorption contrast and scattering enhancement. If an averaging kernel in the reconstruction program is adjusted for exact compensation of the small angle scattering in the projection image, signal-to-noise ratio will be improved without reducing spatial resolution.
4. X-RAY SOURCE, DETECTOR AND OBJECT MANIPULATOR

The x-ray source used for the nano-CT instrument is based on the “open-type” (pumped) electron-optical column identical to that used in electron microscopes [4]. It contains an electron gun with a LaB$_6$ filament, a focusing system with two electromagnetic lenses and a W or Au target to produce x-rays as a thin film on the Be-window. The accelerating voltage can be adjusted in the range 20-80kV with best focusing achievable over 20-40kV.

The x-ray detector contains an intensified 1280x1024 pixels / 12-bit digital CCD camera with direct connection to PC (IEEE-1394). The intensified CCD with Be-window includes 18 mm format MCP-based image intensifiers, fiber-optically coupled to a Sony ICX285 CCD-sensor: the gain is 5000 cd/sq.m/lx, 62 lp/mm resolution. The camera is mounted on the translation stage with 25mm travel / 1µm repeatability for alignment.

The object manipulator can move the specimen stage between the camera and the detector to change image magnification. The object position closest to the detector corresponds to the minimum magnification with a field of view (maximum object diameter) near 10mm. An object position close to the source corresponds to the maximum magnification with the voxel size of the image (at the object location) close to 100nm.

One of the most important parts of the nano-CT system is the object X-Y-Z positioning and rotation system. The necessity to acquire several hundred angular views during 10-60 minutes from a well-defined object location requires very high accuracy and stability. Therefore for object rotation in the nano-CT instrument we use an air-bearing with integrated stepping motor and optical encoder and with the following specifications: flatness <100nm, eccentricity <100nm, wobble <2.5microrad, repeatability 0.001deg, speed up to 360 deg/sec, load up to 5 kg. The rotatable part of this stage floats in an airflow layer without physical contact to the static part. The top of the rotation stage holds an X-Y-Z manipulator for high-precision specimen positioning: Z axis -10mm travel with 400nm resolution, XY axis – piezo drives with 0.5mm travel with 400nm steps. All electronics for X-Y-Z drives in the top of the air-bearing rotation.
stage are powered through a non-contact slotted transformer and controlled by wireless infrared datalink. A special microprocessor controller in the top of the rotation stage controls X-Y-Z drives and continuously measures local temperature in the top of the stage. Temperature measurement allows compensation for thermal extension of the metal parts in the object support with overall accuracy around 150nm.

5. APPLICATION EXAMPLES

To confirm the possibility of three-dimensional reconstruction at the submicron level of spatial resolution, we performed a scan of a platinum (Pt) wire with 600nm nominal diameter as a test object. This wire was supplied as Ag-coated “Wollaston wire” with an overall outside diameter of approx. 0.1mm. The silver outer sheath may be dissolved by Nitric Acid.

To verify the diameter of the Pt wire we used SEM imaging. Fig.4a shows the Pt wire with partially dissolved silver coating on top of a mesh with 5x5µm openings. Measurements show that the Pt core has a diameter of 600-800nm.

Fig.4. SEM-image of the Pt-wire (a), a nano-CT x-ray shadow image (b) and a reconstructed nano-CT cross section with 170nm voxel size (c).

Fig.4b shows two-dimensional X-ray shadow image of the same Pt wire object in the Nano-CT system. Subsequent acquisition and reconstruction were done for a smaller part of the volume, containing only Pt-wire and keeping the thick Ag-part outside the field of view. One of the reconstructed cross sections is shown in Fig.6c. The cross section through the thin Pt wire can be seen as an ellipse since the reconstruction plane crosses the wire at a significant angle. The voxel size in the reconstructed cross sections is 170nm, the scanning time is 30min (one scan contains information for reconstruction of several hundreds cross sections).

Carbon fiber materials are widely used as a composites and porous filters in the fuel cells. Fig.5 shows the scanning results for one of such samples – filter material named “carbon paper” - in our Nano-CT instrument. A shadow image (Fig.5a) does not display any recognizable internal structure, but reconstructed cross-section (Fig.5b) and 3D-rendering (Fig.5c) clearly shows the fibers and connecting structures inside the sample.
Fig.5. A shadow x-ray image (a) a reconstructed cross section (b) and a 3D rendering (c) of the internal microstructure of a “carbon paper” sample scanned with 400nm isotropic voxel size.

CONCLUSIONS

The compact Nano-CT scanner described here allows non-invasive investigation of the three-dimensional internal microstructure of a wide range of objects and materials, with isotropic spatial resolution in the range of hundreds nanometers.

REFERENCES