



Sub-micron computed tomography (CT) for materials science



High-resolution, high-contrast X-ray microscope

What is X-ray microscopy?

Tomography is the study of the three-dimensional structure of an object by slicing it into thin sections. Microtomography implies that the slices are very thin—thin enough to be viewed by an optical microscope. Classical tomography is a tedious and time-consuming process, and can also result in significant perturbations to the sample. In X-ray tomography, the entire sample is imaged at multiple rotation angles. This multitude of images is processed by sophisticated computer algorithms to provide a three-dimensional reconstruction that can be sliced in any direction, providing new insights into the internal features of the object. X-ray microscopy provides this visualization at a resolution better than a micrometer (µm).

What is the nano3DX?

The nano3DX is a true X-ray microscope (XRM) with the ability to measure relatively large samples at high resolution. This is accomplished by using a high-power rotating anode X-ray source and a high-resolution CCD imager. A rotating anode X-ray source provides for fast data acquisition and the ability to switch anode materials easily, to optimize the data acquisition.

How does the nano3DX work?

In the nano3DX, the magnification takes place in the detector using true microscope elements. This design places the sample close to a high-resolution detector, allowing for a quasi-parallel beam experiment. This means greater instrument stability and shorter data collection times, providing the highest resolution of any X-ray microscope in its class. The nano3DX design is a vast improvement over older implementations that use a small source and a long sample-to-detector distance. Traditional geometric magnification requires a very small source and extreme stability to prevent smearing.



The graph at right illustrates the three primary anode materials available for use in the nano3DX: chromium (Cr), copper (Cu) and molybdenum (Mo), and the effects they have on the experiment. As the energy of the X-ray radiation rises, penetration increases but contrast for low atomic weight materials goes down. For aluminumdiamond metal matrix composite and silicates, Mo is preferred but for plastics and hydrocarbons, Cu or Cr is preferred. This flexibility is essential to obtaining high-quality, high-contrast images quickly.





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High 2D/3D spatial resolution

With normal methods of image projection and enlargement, it has not been possible to prevent image blur caused by movement of the X-ray focal spot during prolonged CT measurement. Rigaku Corporation uses its own unique technology to solve this problem and so achieves high spatial resolution.

- Employing a high-resolution detector
- Performing close-distance radiographic imaging using parallel beam methods
- Using an X-ray generator with highly stable output intensity and alignment
- High-precision sample stage

High contrast

The two-dimensional resolving power is shown directly in the images to the right, with a transmission image of a test pattern at 0.27 µm per pixel in which lines at 0.6 µm are resolved. A spatial resolution of 0.8 µm is achieved, even in 3D images. Carbon-fiber-reinforced polymer (CFRP) can be resolved to 0.27 µm/voxel.

The Rigaku nano3DX allows one to see into many types of samples, including

those that have low absorption contrast; for example, CFRP, or denser materials like ceramic composites. This flexibility is due to the ability to change the X-ray wavelength to enhance contrast or penetration. For example, a transmission

image collected with Cu radiation is shown at right. Rapid absorption contrast imaging of ultra-light materials is possible in minutes without the artifacts

inherent in phase contrast imaging. Ultra-light materials are typically invisible to standard CT systems or require long data acquisition times in traditional X-ray

microscopes. The difference in density between low density polyethylene (LDPE)

and polyethylene terephthalate (PET) is clearly shown in the differing gray levels.

CT reconstructed image of two types of polymers Distinguishes between LDPE (50 µm) and PET (150 µm). A density resolution of 0.13 g/cm³ is realized with high polymer materials.

Ultra wide field of view

The diagram to the left is a reconstructed image of a CFRP sample. The fibers are 7 µm in diameter. The image is 1.8 mm x 1.8 mm by 1.4 mm, with a voxel size of 0.54 µm. The volume is represented by 3300 x 3300 x 2500 voxels. This volume is more than 25X larger than the measurable volume from a single scan with other systems at this resolution in a comparable time frame. By employing a high resolution camera (3300 x 2500 pixels), with close distance quasi-parallel beam imaging, nano3DX can create 2D images larger than the field of view by using image combining technology.

Red area illustrates normal X-ray CT field of view

- 1000 × 1000 × 1000 voxels (0.54 µm/voxel)
- CT reconstructed image range: $540 \times 540 \times 540 \, \mu m$

Carbon fibers in CFRP

1.8 mm

nano3DX CT field of view

- 3300 × 3300 × 2500 voxels (0.54 μm/voxel)
- CT reconstructed image range: 1800 × 1800 × 1400 μm





 $0.27 \,\mu$ m/pixel (line spacing = $0.6 \,\mu$ m)

2D spatial resolution

CFRP 0.27 µm/voxel

3D spatial resolution



Sub-micron resolution computed tomography for m

For the elucidation and quantitative measurement of microstructures

Wide view

Capable of observation over a large area without loss of resolution.

- · Detector with a large number of pixels
- 3D display of large amounts of data
- Close-distance radiographic imaging using quasi-parallel beam methods
- Capable of imaging samples over an area larger than the field of view by combining images

Tablet consisting of a two-layer structure Field of view: 14.2 \times 10.1 mm, 4.32 $\mu m/voxel$





Combined microscope image (patent pend

High contrast

High density resolution allows for imaging of light element materials in composites and detection of air spaces and micro-cracks in drug preparations.

- Uses a unique high-brightness X-ray generator and optical system
- Selectable X-ray energy according to the sample and test objectives



High resolution

Capable of 2D/3D observation of the interior of specimens, such as composite materials and drug preparations, at the sub-micron level with high resolution.

- Equipped with a newly developed high-resolution X-ray detector that delivers a maximum resolution of 0.27 µm/pixel
- Optical system is stabilized to prevent blurring over a prolonged period of data collection



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Rigaku



ling) of CFRP that is larger than the field of view

Rigaku Corporation has developed the nano3DX as a high-resolution 3D X-ray microscope that combines Rigaku's unique high-brightness rotating anode X-ray generator with a proprietary high-resolution CCD X-ray camera, to provide high-contrast computed tomography at the sub-micron level.

The nano3DX is able to observe a wide field-of-view while retaining high 2D/3D spatial resolution. It provides improved density resolution compared to conventional X-ray microscopes. The Rigaku nano3DX assists in the elucidation and quantitative measurement of microstructures, including biomaterials, high-polymer materials, composite materials and light metals.

The Rigaku nano3DX is equipped with a fully featured graphical user interface to facilitate activities ranging from research and development to quality control.

Easy to use

The nano3DX is designed for easy mounting of samples. The CCD camera moves back from the sample to allow access to the sample stage. Once the sample is mounted, alignment is performed using the automated 4-axis (XYZ and rotation) stage and on-axis imaging system. Data collection starts with a single 2D transmission image to check the experimental setup, and then a series of images is collected and processed to generate a three dimensional computed tomogram that can be imported into a number of viewing and analysis packages.



nano3DX application examples

Polymer composites

Carbon fibers, epoxy resin, and voids of CFRP materials, that once were considered to be difficult to analyze using X-ray imaging, can now be distinguished clearly. The structure of carbon fibers, cracks, and voids are observed (right) in three dimensions, and the number, volume, and direction of voids are measured.

Pharmaceuticals

Cracks, density, thickness of the coating layer, and foreign particles admixed in during tablet preparation, can be observed with high contrast and high spatial resolution. The dimensions and volume distribution of particles can be measured at the micron level.



4.32 µm/voxel

High polymer blends

The phase-separated structure of polymer blend and the three-dimensional structure of polymer foam were difficult to observe previously. Polyvinylidene fluoride (PVDF) in polypropylene (PP) can be observed in high contrast.



0.54 µm/voxel



Light metals

Distribution of the orientation of carbon nanotube (CNT) aggregates, which affects thermal conductivity, can be observed. Below is a reconstruction of a high thermal conductivity AI composite material containing single wall CNT.





Plant seed

Seed of the *Nandina domestica* plant. Rigaku nano3DX allows the observation of the internal structure without cutting or staining.



4.32 µm/voxel

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Specifications

X-ray source	High-brightness rotating anode
Voltage / current	20 – 50 kV, ≤30 mA
Anode targets	Cr, Cu, Mo
Detector type	X-ray CCD camera
Area	3300 x 2500 pixels
Resolution	0.27 to 4.32 µm/pixel
Field-of-view	0.9 x 0.7 to 14 x 10 mm
Dynamic range	16 bit
Sample stage	5-axis automatic
Computer	
CPU	2 Intel Xeon processors
HDD	2 TB
Memory	128 GB
Monitor	24 inch
Operating system	Windows® 7 64 bit
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Dimensions	1300 (W) x 60 (D) x 1640 (H) mm
Weight	600 kg
Cooling system	External water chiller (standard)
External radiation leakage	≤1 µSv/h

Installation conditions

Water supply*	6 L/min at 25℃ or 13 L/min at 32℃	
Power	Main unit	200 VAC, 3-phase, 15 A
	Computer	100 – 240 VAC, 15 A
	Water chiller	100 – 240 VAC, 3-phase, 20 A
Environment	Requires a low humidity air conditioning facility	

*An air-cooled type water supply device may be used instead of the standard water-cooled type water chiller.

Software

Data collection: nano3DX	X-ray transmission microscopy and CT reconstruction.
Graphics and measurement software: nano3DCalc	Labeling, volume maps, surface area, center of gravity, orientation including median trimming, 2D/3D graphics and other image processing.

nano3DCalc

Proprietary software provides 3D computed tomography images. Example shows capsule drug particles: 3D image (left) and volume map of voids (right).



Layout





Backed by Rigaku

Since its inception in 1951, Rigaku has been at the forefront of analytical and industrial instrumentation technology. Today, with hundreds of major innovations to our credit, the Rigaku Group of companies are world leaders in the field of analytical X-ray instrumentation. Rigaku employs over 1,100 people worldwide in operations based in Japan, the U.S., Europe, South America and China.





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