White Paper

True 0.3µm Nanoscale Resolution with the

EclipseXRM-900™

3D X-ray Microscope

EclipseXRM™ introduces a patent-pending architecture to achieve its breakthrough resolution capabilities, achieving both 0.3µm (300nm) resolution and the highest resolution for large working distances. In this technical white paper, we review the enabling components and additional advantages of EclipseXRM's innovative design.

This white paper will review EclipseXRM's advanced x-ray tomography capabilities.

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SIGRAY

True Nanoscale Resolution with the **EclipseXRM-900™ 3D X-ray Microscope**

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System Overview: Sigray EclipseXRM is a next-generation 3D x-ray microscope (XRM) that offers the highest resolution performance capabilities of flexible x-ray microscopes on the market. The system provides 0.3µm true spatial resolution (Fig 1), far surpassing the previous leading systems at ~0.5 µm resolution. Submicron resolution is preserved even at long working distances, enabling imaging samples in situ and zooming in at progressively higher resolutions on a large sample without needing to cut it down. Applications of this versatile tool span from semiconductor failure analysis and 3D printing to geology and biology.

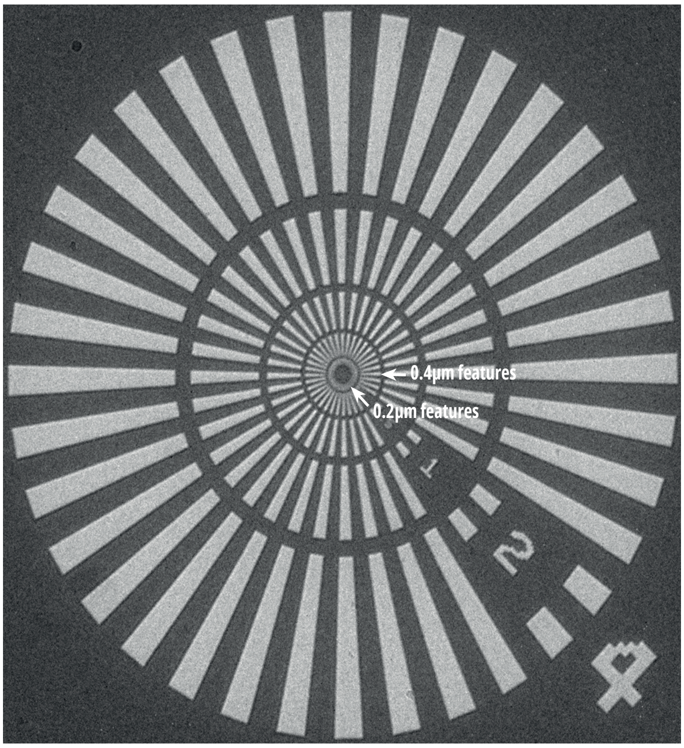

Figure 1: *Siemens star resolution standard, clearly showing 0.3µm spatial resolution capabilities. The outer rim of the innermost circle starts at 0.2µm and the outer rim of the second innermost circle starts at 0.4µm linepairs.*

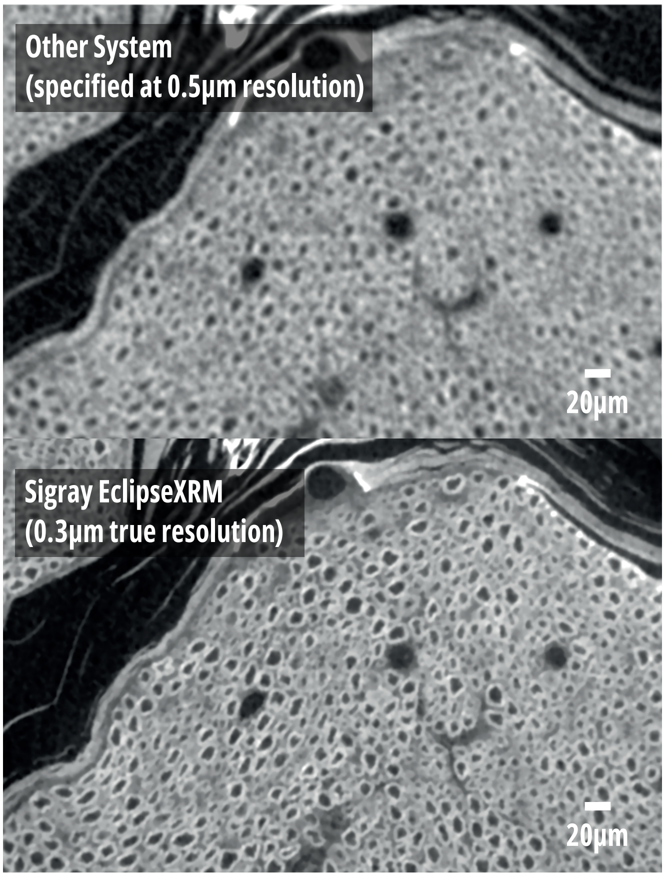

Figure 2: Increased resolution on mouse sciatic nerve samples, clearly showing axons and myelin sheaths with EclipseXRM at 0.3µm.

Background

For years, spatial resolution of 0.5µm was well known to be the spatial resolution limit of even the highest end x-ray microscopes (XRMs). The only way to surpass this was to use x-ray optics such as zoneplates, but such systems operate at such low x-ray energies (5-8 keV) that samples often need to be cut down to tens of micrometers in diameter, usually with expensive, dedicated sample preparation tools. For flexible XRMs that can accommodate a wide variety of sample types and shapes, 0.5µm was seen as the limit.

At 0.3µm spatial resolution, EclipseXRM shatters the previous resolution limitation of flexible XRMs. To understand how it achieves its breakthrough performance, it is important to first understand how resolution has previously been approached.

How Other Systems Achieve Resolution

The resolution of a flexible (non-zoneplate) x-ray microscope is determined by:

$$\delta_r = \frac{1}{M}\sqrt{(M-1)^2S^2 + D^2}$$

Where M is the geometric magnification, S is the source spot size, and D is the effective detector pixel size. Quite simply, resolution is the weighted convolution of the source spot size and the detector pixel size.

There have been two dominant approaches to using this equation to achieve the highest resolution:

1. Geometric magnification

Most microCT/XRM architectures use a small spot x-ray source (small S) and a flat panel detector (large D). To minimize resolution (δr), large geometric magnification M is used, therefore weighing the resolution equation primarily to the small spot size S.

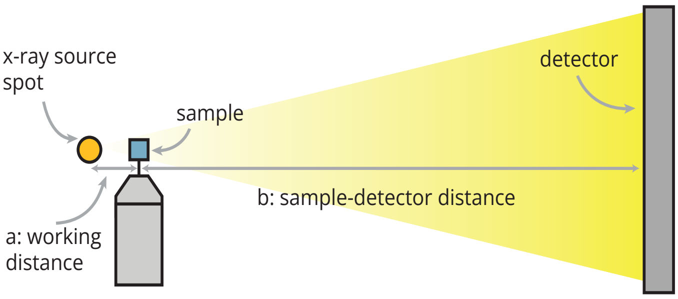

Figure 3: *Geometric magnification architecture*

Challenges with this geometric magnification-based approach include:

- **Poor resolution at long source-sample distances (working distance):** Larger samples (or samples in-situ) require the sample to be placed further from the source, limiting geometric magnification M. Without a large M, the pixel size of the detector becomes a limiting factor for the resolution.
- **Short source filament lifetime:** Small spot sizes often are achieved using open (unsealed) x-ray tubes, which have poor cathode lifetimes of only a few hundred hours. Exchanging the filaments can be time-consuming and costly.

2. Two-stage magnification

An alternative architecture, often marketed as "two-stage magnification", uses a sealed x-ray source (small to medium S) and an objective magnification detector, in which a scintillator is used with an objective lens from an optical microscope (e.g., 10X, 20X, 40X) and coupled to a CCD (or CMOS) detector sensor. Such a "two-stage" architecture does not change the fundamental XRM resolution equation, but simply results in a small effective detector pixel (small D).

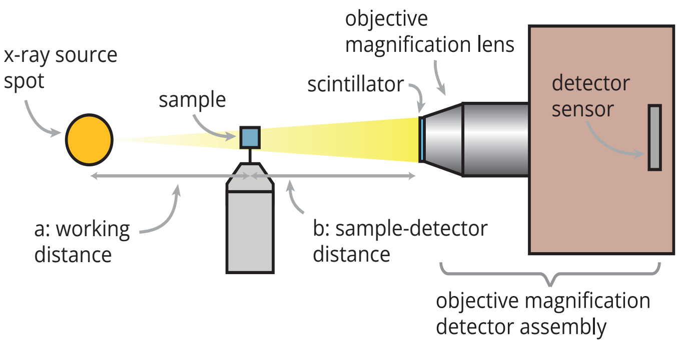

Figure 4: *Two-stage magnification architecture*

Challenges with this two-stage magnification approach include:

- **Low detector efficiency:** Higher magnification objectives (such as 20X or 40X) must be coupled to very thin scintillators. These scintillators detect very few x-rays, limiting their practical value. Most often, these systems are operated using lower magnification (e.g., 4X) lenses and thus operate at far lower spatial resolution than advertised (0.5μm spatial is only achievable using a 40X).
- **Costly source replacements:** Sealed x-ray tubes do not require filament changes, but instead require the entire x-ray source to be replaced every few thousand (e.g. 5000) operating hours.

Patent-Pending Architecture

EclipseXRM's 0.3μm capabilities eclipse the previous resolution limitation of flexible XRMs. This achievement is enabled by a combination of breakthrough technology in both x-ray source and x-ray detectors to minimize both S and D in the resolution equation.

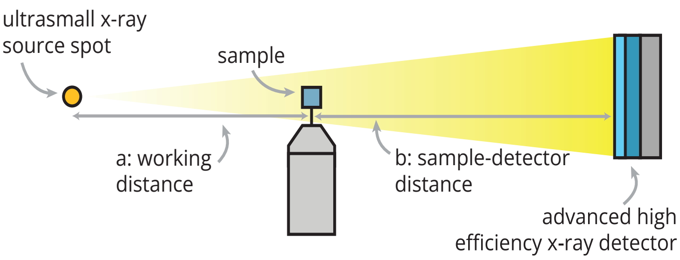

***Figure 5:** Sigray EclipseXRM architecture, featuring breakthroughs in x-ray source (to achieve a small spot size) and in the x-ray detectors*

Breakthrough #1: X-ray Source

Sigray's EclipseXRM features a novel open x-ray tube that uses state-of-the-art electron optics to focus electrons onto a structured x-ray target to produce a sub-0.3μm x-ray source. Advanced focusing controls ensure the same high performance throughout the entire operating parameter space.

Unlike other open tubes, the innovative electron optics design enables long cathode lifetimes of over several thousand hours of operation, compared with the few hundred hours of most open tubes. The much longer cathode lifetime overcomes the traditional lower maintenance advantage of using a sealed tube instead of an open tube. In fact, the cathode lifetime is comparable to that of the **entire** sealed x-ray source. Thus the cost of ownership is reduced compared to competing systems using sealed x-ray tubes because only the EclipseXRM cathode filament needs to be exchanged rather than the complete x-ray source assembly.

Breakthrough #2: X-ray Detector

EclipseXRM comes standard with both a LFOV (large pixel count) flat panel detector and a high resolution detector with a small pixel CMOS sensor. This detection system enables the fastest scan times for high resolution imaging on the market, providing a 10X gain (Fig. 6) in efficiency over detectors such as those based on high magnification objective lenses.

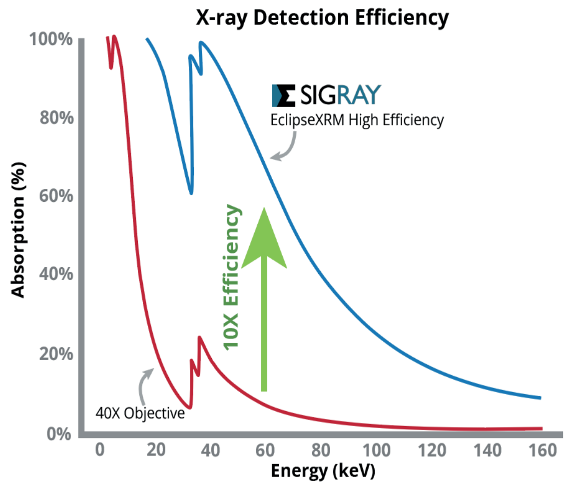

***Figure 6:** EclipseXRM uses thick, high efficiency scintillators for high resolution imaging, providing a 10X gain in efficiency.*

One major advantage is that the EclipseXRM uses higher efficiency and thicker scintillators for high resolution imaging than systems that rely on high magnification objectives (e.g. 20X and 40X).

A challenge with high magnification objectives is their shallow depth-of-focus (DoF), as shown in Fig. 7. For example, a standard 40X lens has a DoF of only 1.0μm [1].

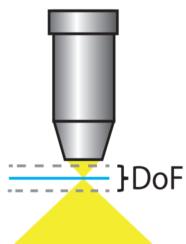

***Figure 7:** Depth of Focus (DoF) of a high magnification objective lens is typically extremely shallow, leading to inefficient detection.*

The limited DoF of high magnification objectives necessitate ultrathin scintillators that fit within the DoF to prevent blurring. However, most x-rays pass through these ultrathin scintillators without being absorbed or used, leading to poor detection efficiency. This inefficiency is why 40X objective lenses are rarely used on two-stage magnification systems for day-to-day imaging. In fact, the practical spatial resolution of even the highest resolution XRMs on the market **are typically ~0.7-1.0μm rather than the 0.5μm advertised** because 40X objectives are almost never used.

In Situ & Resolution at Large Working Distances

In addition to its leading performance in absolute resolution for ideal samples, Sigray EclipseXRM's major x-ray source and detector innovations result in the highest resolution **at large working distances** on the market.

This is important for imaging samples *in-situ* (under environmental conditions such as mechanical strain, temperature, etc.) and for samples that cannot be cut down to under a few mm in diameter (larger samples). High quality imaging of samples *in-situ* and larger samples require the ability to achieve high resolution at large working distances (source-sample distance).

It is well known that resolution for geometric magnification based XRMs degrade rapidly as a function of working distance. Even two-stage magnification based XRMs degrade slightly, with best achievable resolution a little under 1µm for a 50mm working distance. EclipseXRM's patent-pending architecture enables maintaining superior resolutions <0.5µm spatial resolution, even for a 50mm working distance, as shown in Fig. 8.

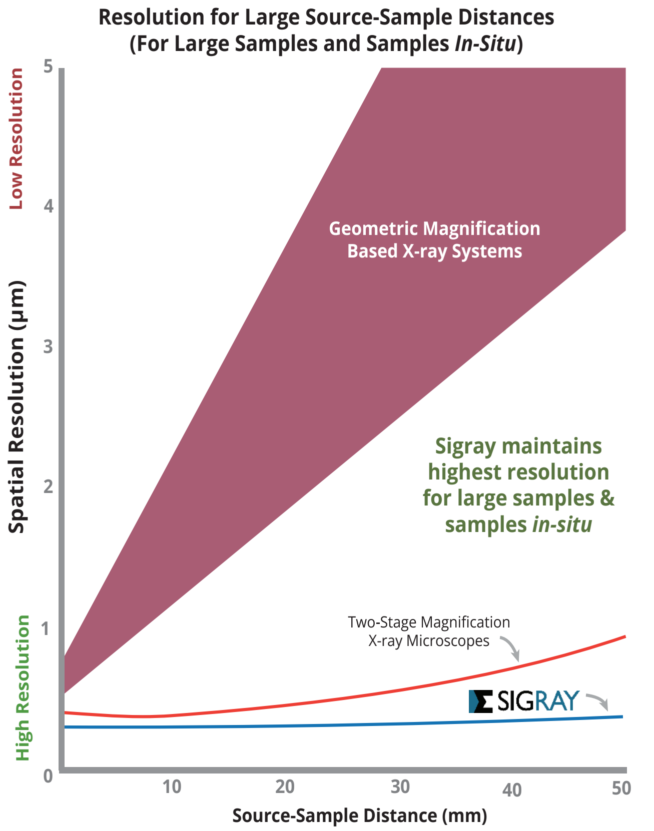

Figure 8: *EclipseXRM maintains the highest resolution for large working (source-sample) distances.*

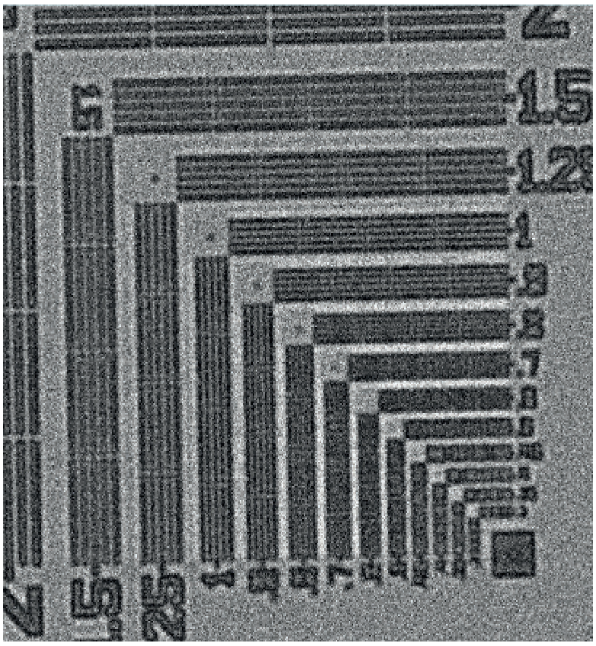

Figure 9: *Resolution target imaged at 50mm source-sample distance, showing submicron spatial resolution.*

Enhanced X-ray Microscope Capabilities

EclipseXRM offers a set of machine learning based software and automation features to maximize the usability and flexibility of the system. As an R&D-focused company, Sigray is constantly expanding the system's feature list and willing to work with customers to develop new features.

A subset of EclipseXRM's additional tomography features are described in the following sections.

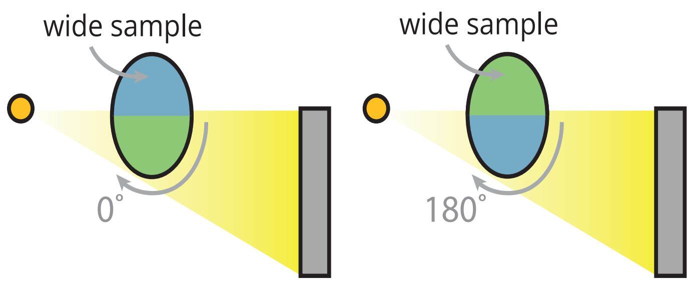

Figure 10: *Overhead view of* ***offset tomography****, which provides up to ~2X the FOV for wide samples. This approach is preferred to horizontal (wide field) stitching because it is more efficient and does not suffer artifacts.*

Advanced Scan Modes: Offset and Helical Scanning

Samples generally differ in height and width. Offset tomography and helical tomography are advanced routines designed for wide and tall samples (such as rock cores for O&G and mining studies) respectively and come standard on EclipseXRM.

It is important to note that offset and helical tomography provide far superior results compared to horizontal (also called wide field) and vertical stitching, which are also possible using the EclipseXRM but are slower and can suffer from stitching artifacts (or must have significant overlap between tomographies to minimize such artifacts, thereby reducing scanning efficiency).

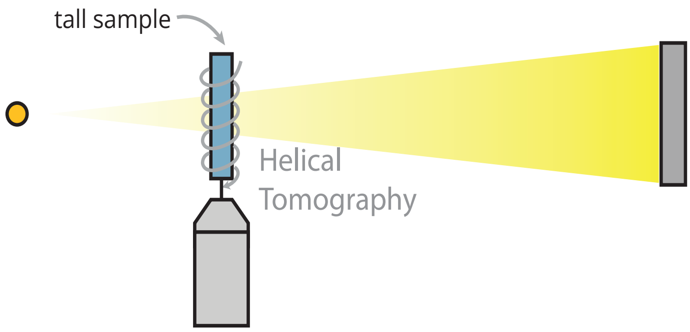

Figure 11: *Side view of* ***helical tomography*** *for tall samples, such as rock cores for oil & gas and mining. This approach is preferred to vertical stitching because it is more efficient and does not suffer artifacts.*

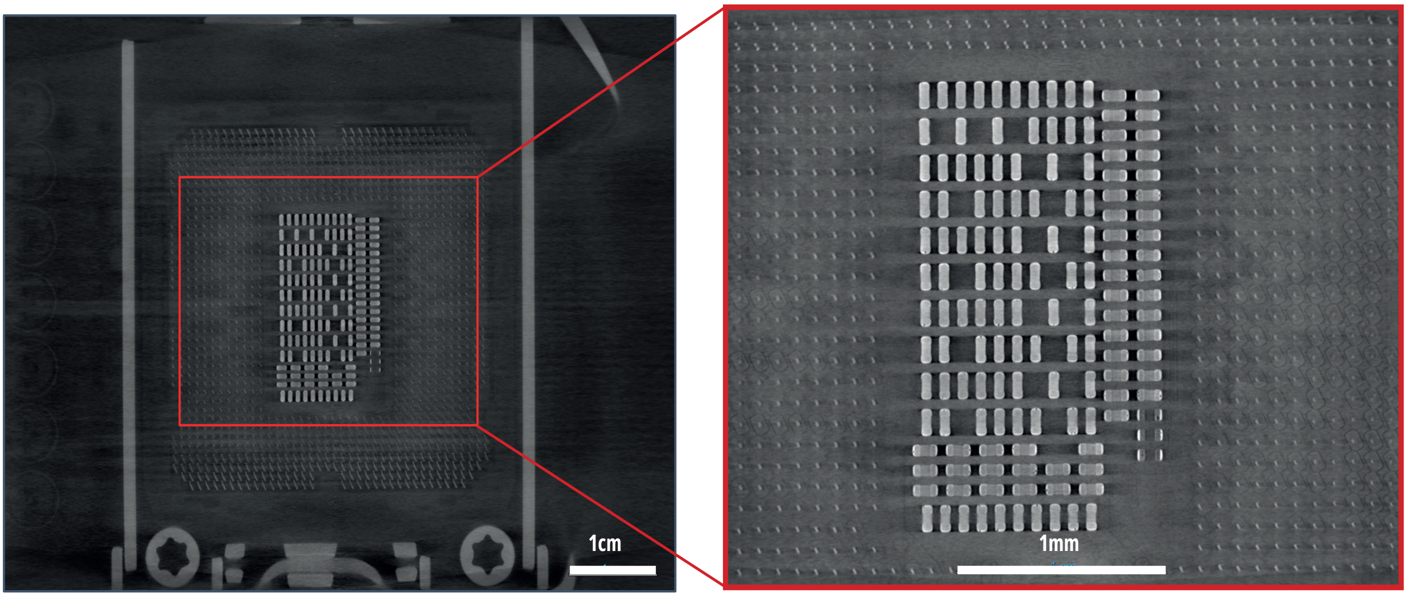

Figure 12: *Offset tomography enables imaging of large samples, such as a 76mm wide and 60mm tall motherboard. Left image is the entire FOV imaged at 26µm voxel and right is a zoomed in tomography taken at 11µm voxel.*

Enhanced X-ray Microscope Capabilities

Superior Phase Contrast Performance

Almost all x-ray imaging, from medical and dental x-rays to x-ray microscopy, relies on the absorption of x-rays. However, low atomic number (low Z) samples such as biological or polymeric samples do not substantially absorb x-rays, making them difficult to image. Even samples of higher atomic numbers but of similar x-ray absorption, such as minerals and elements in rocks and ceramics, prove to be challenging.

Phase contrast imaging measures the refraction of x-rays rather than the absorption. It can provide up to 1000X higher contrast than x-ray absorption. The challenge is that it requires a coherent approach to imaging. With most systems on the market, this means the use of gratings and/or moving the sample unnecessarily far from the x-ray source (propagation based phase contrast), which severely limits throughput.

EclipseXRM provides superior phase contrast performance by using one of the most coherent x-ray sources on the market. Due to the source's coherence, samples do not need to be retracted far from the x-ray source. Furthermore, the system uniquely enables phase contrast imaging even at higher x-ray energies, enabling phase contrast for mid Z samples. The images can be further analyzed using Sigray's phase retrieval algorithms (see Fig. 13).

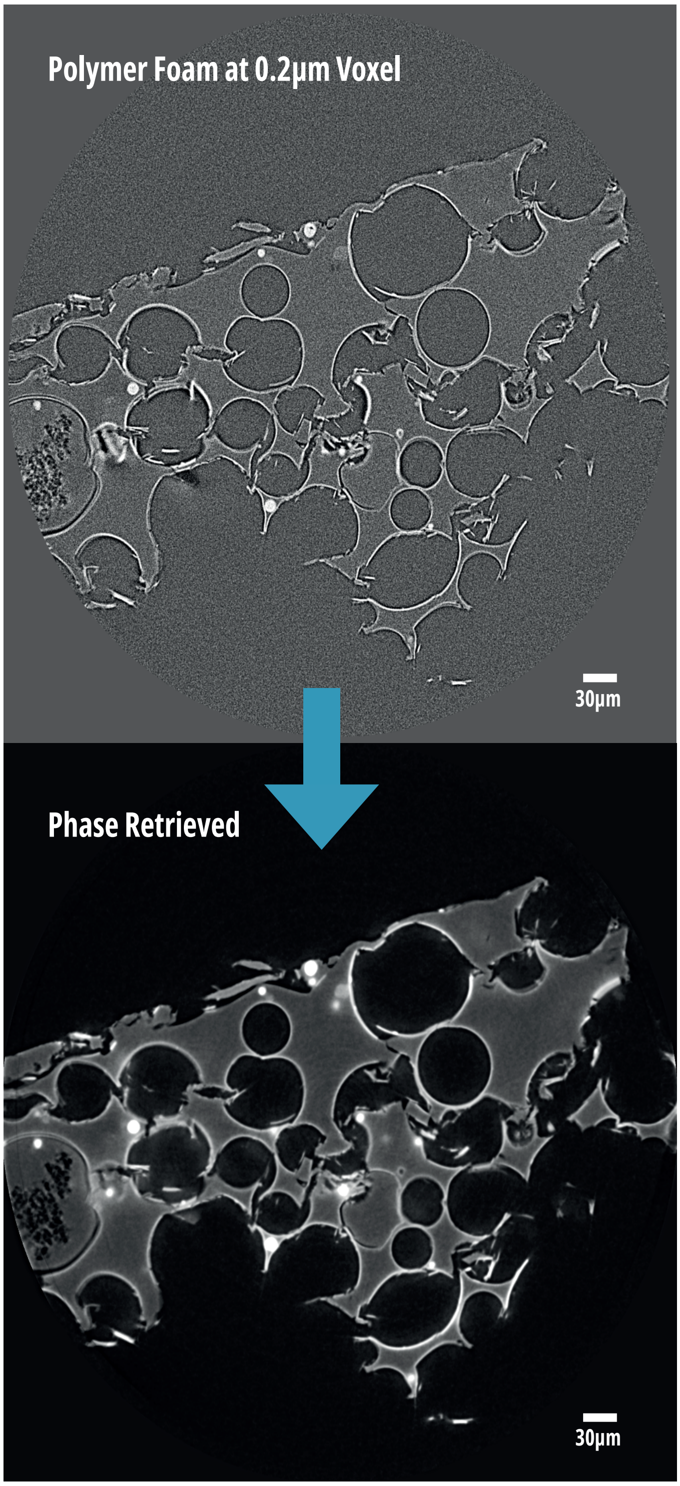

***Figure 13:** Polymer foam imaged using propagation phase contrast (top) and at a voxel size of 0.2µm on EclipseXRM. Below shows the tomography after applying Sigray's phase retrieval algorithm for segmentable, quantifiable data.*

Enhanced X-ray Microscope Capabilities

Multi-Spectral Source (MSS)

EclipseXRM offers the possibility of near-monochromatic (single energy) imaging with a second x-ray source. This Multi-Spectral Source (MSS) incorporates three to five x-ray target materials, each producing a different x-ray energy (Fig. 14). For example, a Cr target produces an 80% monochromatic beam of 5.4 keV energy. Switching between targets is motorized and achieved with just a push of a button in Sigray's acquisition software. In comparison, conventional x-ray imaging uses a single tungsten (W) target that is primarily polychromatic (a broad band of x-ray energies).

The MSS provides exceptional performance for soft and low density materials, such as insects, fixed tissue, carbon fibers, and li regions of battery experiments. As shown in Fig. 15, using an optimized target of the MSS provides a substantial increase in visibility when compared to a conventional W target.

In addition, the target presents opportunities for quantitative elemental studies using dual energy (for such applications as mineralogy) or for optimizing a target to minimize absorption by a dominant element.

X-ray Source Target Material	Characteristic X-ray Energy
Ti	4.5 keV
Cr	5.4 keV
Cu	8.04 keV
Au	11 keV
Mo	17 keV
W	Primarily broadband
Others upon request	

Figure 14: *Multi-Spectral Source (MSS) features up to 5 different, customizable x-ray target materials in the same source body. The types of x-ray target materials offered produce near-monochromatic illumination, offering advantages such as increased contrast and enabling quantitative imaging.*

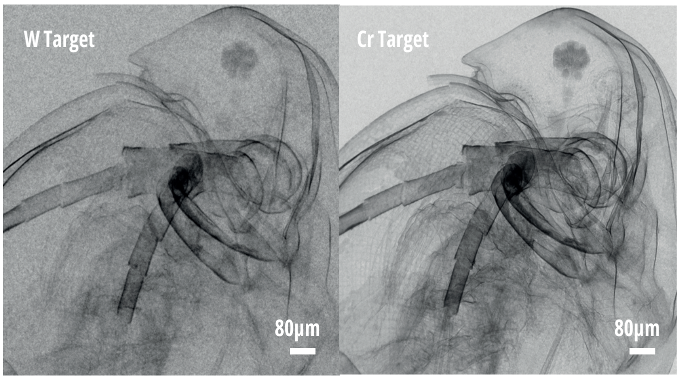

Figure 15: *Daphnia (water flea) projection using a conventional tungsten x-ray target (left) and Cr target on MSS (right). Orders of magnitude higher contrast using MSS is possible due to its ability to provide monochromatic beams of low x-ray energy for optimized transmission and contrast.*

Enhanced X-ray Microscope Capabilities

Sample Handling Robot

For busy central laboratories, Sigray has developed a robotic sample handler that queues up samples without needing manual assistance. The Sample Handling Robot (SHR) system features 14 sample stations for 24/7 operation over multiple days (such as long weekends). The sample stations can be further augmented by using sample holders that hold multiple small samples (e.g., 5 smaller samples per station for a total of 70 samples).

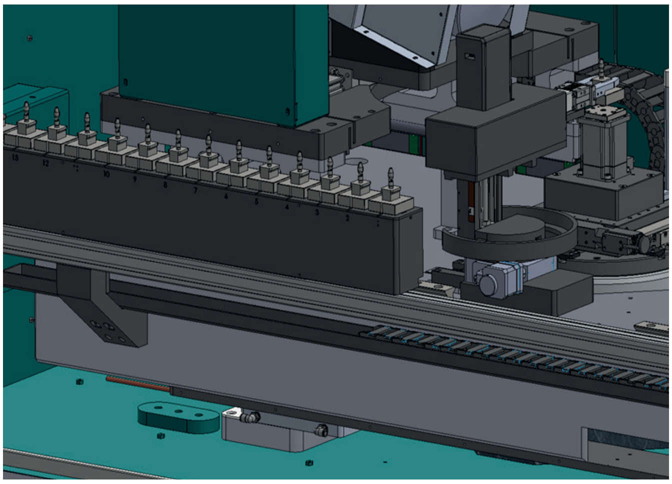

Figure 16: *Robotic sample handling robot designed for EclipseXRM to allow operatorless exchange of samples for overnight or weekend scans.*

Intuitive Software

Even beginners that do not understand the physics behind computed tomography can walk up to the EclipseXRM and use the system. Sigray's BestTomo™ interface in its XRM Companion acquisition software quickly identifies the best scan parameters for the optimum image quality scan for any sample with the push of a button, selecting the accelerating voltage (kV), power, filter, and exposure time per projection.

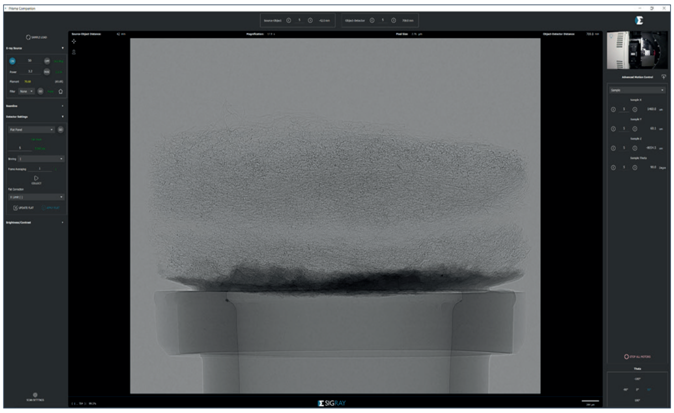

Figure 17: *Sigray's acquisition software minimizes the number of clicks required for acquiring the highest quality tomographies.*

GigaRecon

Sigray's GigaRecon reconstruction package is the fastest on the market, providing efficient multi-GPU reconstruction through standard and iterative reconstruction approaches and multiple artifact suppression methods. Additional information can be found in Sigray's white paper on Advanced X-ray Software [2].

Open Source Controls

For advanced users, Sigray provides python scripting for the reconstruction module and uses an open source controls software (PyEpics) for enabling additional functionality and/or adding new hardware. Additional information can be found in Sigray's white paper on Advanced X-ray Software [2].

EclipseXRM-900™ Specifications

Parameter	Specification	
Spatial Resolution[a]	300nm spatial resolution Voxels down to sub-10nm	
Resolution at 50mm Working Distance	<500nm spatial resolution	
Source(s)	Primary: Nanofocus Source	Optional Secondary: Multi-Spectral Source (for Biological Samples & Polymers)
Target	Tungsten on diamond	Multiple target materials (up to 5) w/ diamond Examples: Ti, Cr, Fe, Cu, Rh, W, Mo, Au
Power & Max Voltage	16W, 160 kV	100W, 50 kV
X-ray Detectors		
High Resolution detector	High efficiency detector with <5μm pixels	
Large FOV detector	Standard Flat Panel detector: 6.7MP with 50 μm pixels Larger formats (13MP or 27MP) available upon request.	
Rotary Stage	Standard Air Bearing: 100 x 40 x 100 mm XYZ, 10 kg load (recommended) *Optional Alternate Mechanical: 50 x 100 x 50 mm XYZ, 25 kg load	
Additional Features	Phase contrast with phase retrieval Offset tomography Helical scan	
Workstations	Linux-based Control Workstation Windows-based Analysis Workstation (recommended specifications found at ref [3])	
Software	Acquisition: Sigray3D Intuitive Software with Machine Learning Reconstruction: Sigray GiganRecon - Fasteset reconstruction algorithm on the market Advanced Analysis: Optional ORS Dragonfly and/or Avizo Data Analysis	

[a] Spatial resolution for absorption mode measured with 2D resolution standard. Spatial resolution is a stricter standard than other "resolution" figures of merit such as pixel, voxel, and minimum detectable feature.

References

1. Nikon Microscopy U: https://www.microscopyu.com/microscopy-basics/depth-of-field-and-depth-of-focus
2. Sigray White Paper*: D Vine et al. Advanced 3D X-ray Software: GigaRecon.
3. Sigray White Paper*: J Gelb. Offline PC Selection for XRM Data Analysis.

*White papers available upon request with your sales representative or by emailing sales@sigray.com.

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