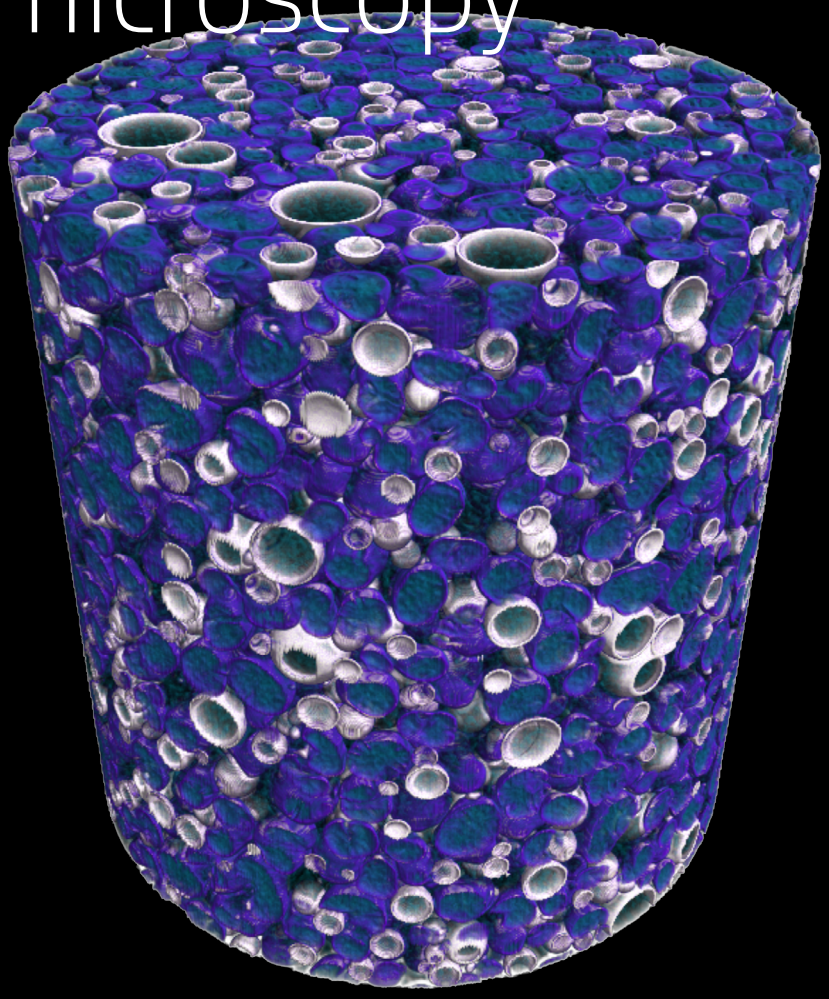
White paper

Fast, high-quality analysis of loose powders

with X-ray microscopy



Seeing **beyond**

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Date: **September 2026**

Abstract

Powder and particulate materials are common across many disciplines including biology, pharmacology, materials science, geoscience, and manufacturing. X-ray microscopy (XRM) is a powerful tool for analyzing these materials, offering non-destructive, three-dimensional insight without the need for sectioning or other destructive preparation steps. However, imaging loose powders presents a key challenge: particle motion during acquisition. In conventional step-and-shoot tomography, repeated stage acceleration and deceleration can cause particles to shift or bounce, resulting in image blur, reduced reconstruction quality, and unreliable segmentation. To mitigate this, powders are often embedded in epoxy or resin, but this introduces additional issues such as limited X-ray transmission and reduced contrast, particularly for low-Z materials or powders with similar densities to the mounting medium. The fast acquisition scanning technology (FAST) acquisition mode on the ZEISS VersaXRM 5 Insight™ addresses these limitations by enabling continuous motion scanning. By eliminating start-stop motion, FAST mode minimizes particle disturbance during imaging, allowing for sharper datasets from loose powders. With availability on both the flat panel detector and the high-resolution 4x objective, FAST mode supports improved segmentation and more reliable quantitative analysis without the need for powder embedding.

The importance of powder characterization

Powders are central to a broad range of scientific and technological applications:

In pharmaceuticals, powder structure influences tablet performance, dissolution behavior, mechanical integrity, and manufacturing consistency. Appropriate powder/particle analysis is imperative to quantify the number of different excipients, fillers, and active pharmaceutical ingredients, to check for granulation/agglomeration, and to quantify the even distribution of different particles in the dosage form.

In the geosciences, the shape, packing, and internal structure of sediments and rock particulates provide insight into natural processes and material history, as well as understanding associations and liberation potential for the mining industry.

In materials science, powders are fundamental to additive manufacturing, ceramics, catalysts, batteries, metallurgy, and composite processing.

Because powder behavior is often governed by three-dimensional structure, X-ray microscopy (XRM) has become an important tool for non-destructive characterization. XRM allows researchers to inspect powders, particles, and granular assemblies without physically cutting, polishing, embedding, or otherwise altering the sample. This capability is especially valuable when the sample must be analyzed in its final form, such as a pharmaceutical tablet, packaged product, or naturally occurring particulates.

Despite the advantages of X-ray microscopy, loose powders remain difficult to image well. Traditional tomography is commonly performed using the sequential tomographic exposure process (STEP) approach, also called step-and-shoot. In this acquisition mode, the sample stage rotates to a specified angular position, stops, image is acquired, and the stage then moves to the next angular position. This process is repeated hundreds or thousands of times during a scan.

For rigid, monolithic samples, this approach works well. For loose, unpacked powders, however, the repeated acceleration, deceleration, and stopping of the rotation stage can disturb the sample: individual particles may shift, bounce, settle, or rearrange during the scan.

Figure 1 demonstrates the impact of start-and-stop motion on loose particulates. This can lead to issues during reconstruction of the final volume; the subsequent blurring reduces image sharpness, obscures particle boundaries, and can compromise downstream analysis.

The problem becomes especially important when the goal is to segment individual particles from reconstructed volumes. Segmentation requires clear, stable boundaries between particles and surrounding void space or neighboring phases. If particles move during acquisition, the resulting reconstruction may contain smeared features, indistinct edges, or artifacts that make automated segmentation difficult.

Conventional workaround – embedding powders in a matrix

To reduce motion during tomography, many researchers embed powders in a stabilizing medium such as epoxy or resin. This approach immobilizes the particles and can improve mechanical stability during scanning. However, embedding introduces several important limitations:

- The embedding medium can absorb X-rays and reduce the transmitted intensity available from the powder itself. This can degrade signal quality, especially when imaging small particles or weakly absorbing materials.
- If the powder is composed of low-Z elements or has density similar to the mounting matrix, contrast between the powder and the matrix may be severely reduced. In some cases, the particles of interest may become difficult or impossible to distinguish from the surrounding medium.

Embedding also changes the sample environment. For applications where the goal is to image the powder in its natural, processed, packaged, or final state, embedding can defeat the purpose of non-destructive analysis. In pharmaceutical, geological, and materials science workflows, preserving the sample “as-is” can be essential.

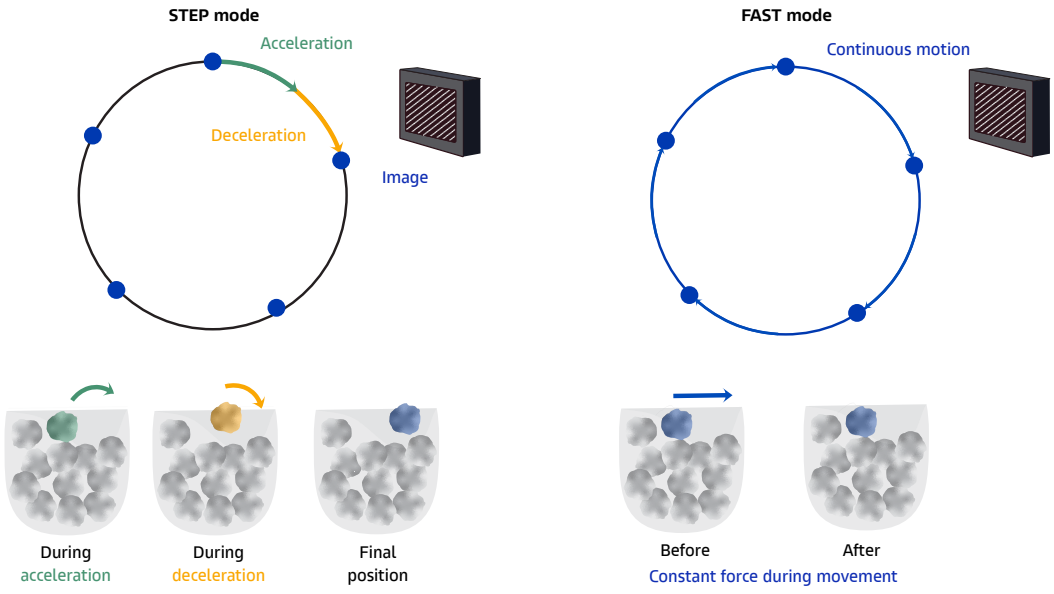


Figure 1: STEP mode versus FAST mode stage movement imaging modalities.

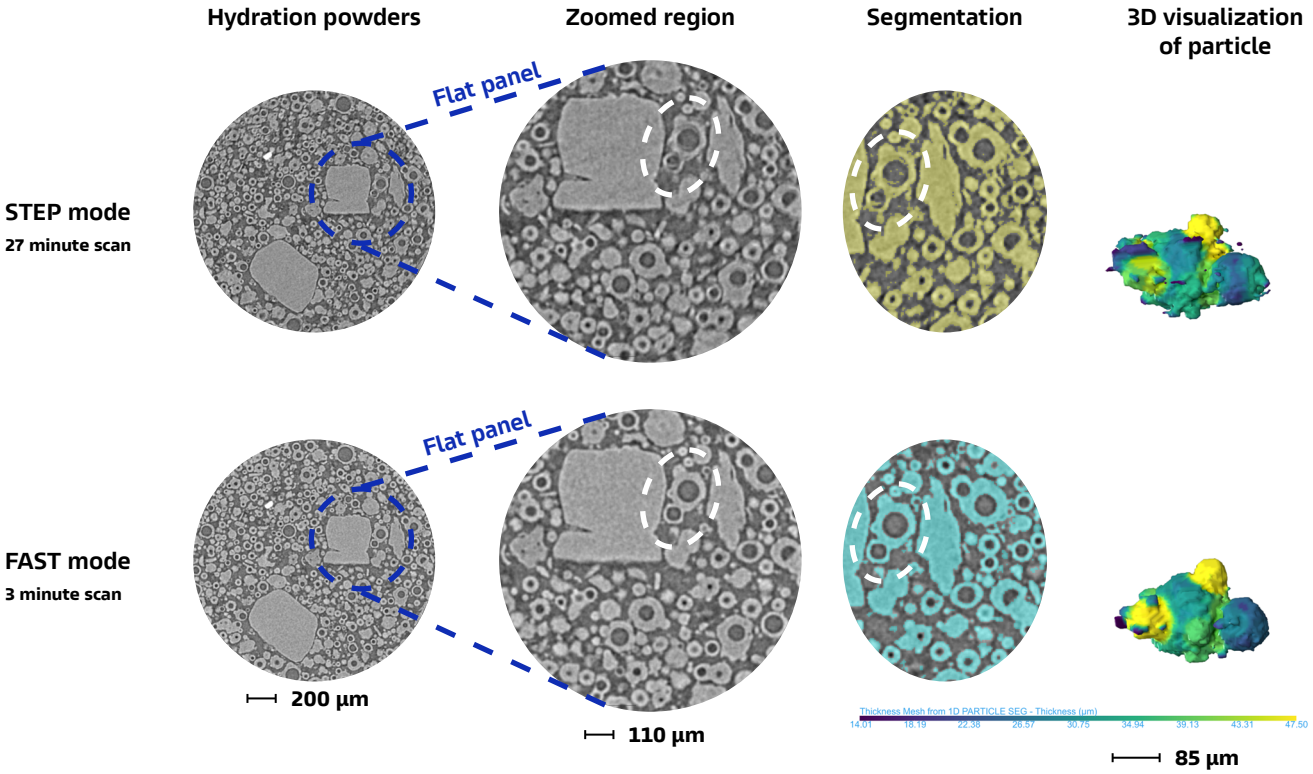


Figure 2: The advantages of FAST mode for unembedded powders compared to traditional step-and-shoot imaging. Datasets take a fraction of the time to collect with the added benefit of sharper images.

FAST continuous motion scanning

ZEISS VersaXRM 5 Insight includes the Fast Acquisition Scanning Technology (FAST) mode, which enables continuous motion scanning during X-ray microscopy acquisition. Traditional X-ray CT and X-ray microscopy rely on the Sequential Tomographic Exposure Process (STEP) imaging mode, also called step-and-shoot. While continuous-motion acquisition was previously available on other Versa systems for flat-panel imaging, the addition of the CMOS camera now enables this capability on the higher resolution 4x objective as well.

The key benefit of FAST mode for powder imaging is not simply faster acquisition: the most important advantage is mechanical stability during the scan. FAST mode changes the acquisition dynamics by keeping the sample in steady continuous rotation. Once the scan is underway, the powder is not subjected to the same repeated stop-start events that occur during conventional acquisition. As a result, the particles remain more stable throughout the scan, and the final reconstructed volume exhibits sharper image quality. Figure 2 clearly demonstrates the FAST mode advantage. The sharp boundaries in FAST mode scans enable clearer visualization of sample defects and only require a fraction of the acquisition time.

Enabling high-resolution FAST acquisition and segmentation of powders

The availability of FAST mode on the 4x objective is especially important for segmentation workflows at higher resolution. Many samples contain a range of particle shapes and sizes, ranging from nanometers (nm) to centimeters (cm). Collecting

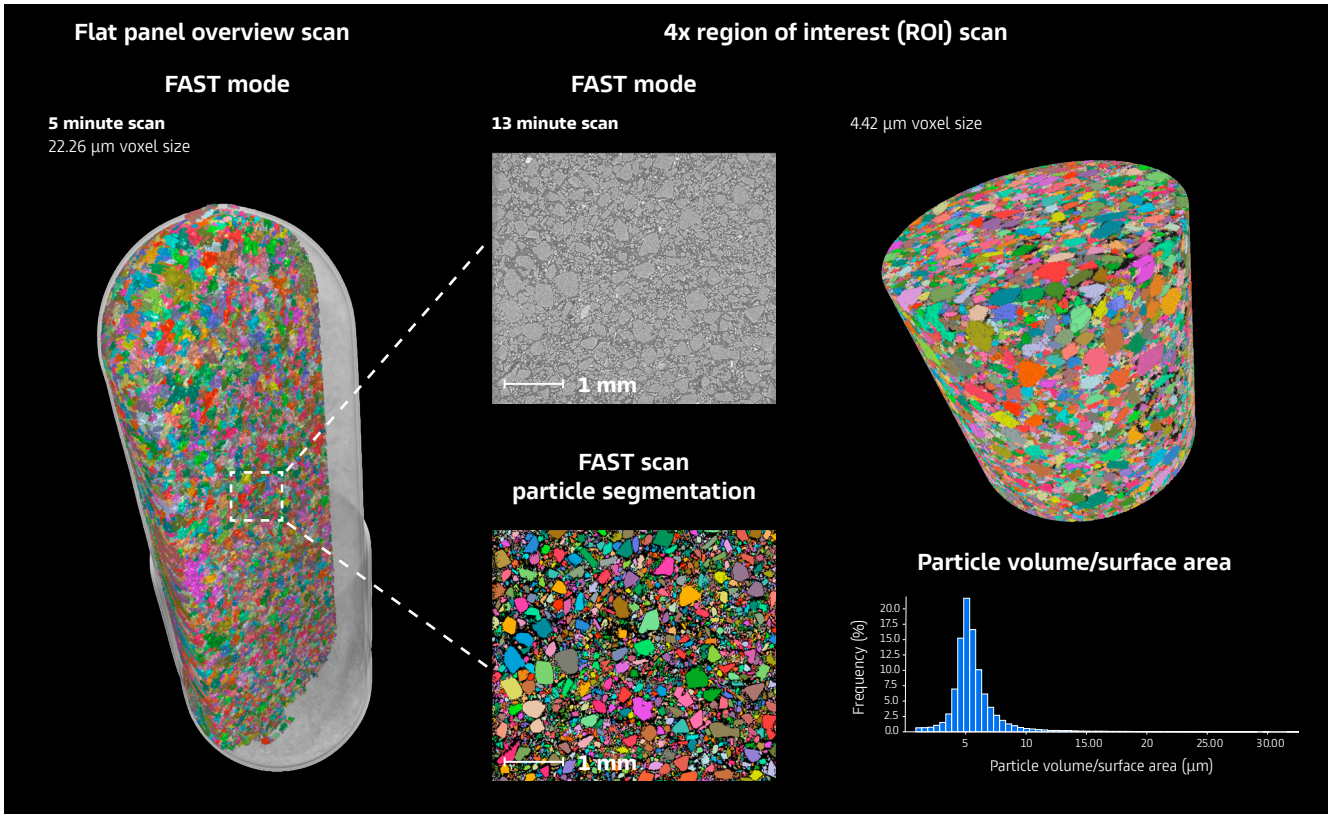


Figure 3: Particle segmentation of capsule powders from an overview flat panel scan and a subsequent higher resolution interior region of interest scan using the 4x objective. Both scans were collected using FAST mode. Object-based segmentation is achievable across a range of size fractions, with the 4x pushing the resolution limits for quantitative analysis of every particle.

images at the multi-scale and utilizing the higher resolution scanning on the 4x allows smaller size fractions to be studied with clarity. Powder datasets often require particle-level analysis, where individual particles must be separated from one another and from the surrounding background. This type of analysis depends strongly on image sharpness and boundary clarity. Figure 3 visualizes the benefits of sharp images for particle segmentation. Particle size distributions can easily be skewed by poor segmentation due to blurred boundaries. Without FAST mode, segmentation of loose powder datasets may require extensive filtering, denoising, image enhancement, or manual correction to produce usable particle boundaries. With FAST mode, loose powders can be imaged with reduced motion artifacts, producing cleaner boundaries between particles. This improves the reliability of automated or semi-automated segmentation and supports more quantitative analysis of powder structure.

Application areas

Pharmaceuticals: In pharmaceutical development and manufacturing, powders and compacted powder products are everywhere. X-ray microscopy can image and inspect tablets, granules, excipients, active pharmaceutical ingredient distributions, porosity, and internal defects. Because XRM is non-destructive, it can be applied to the entire range of materials from raw material powders to finalized or packaged products without cutting or otherwise damaging the sample.

FAST mode may be especially useful when imaging loose powders or fragile granular structures that would otherwise require embedding. By avoiding resin or epoxy, researchers can preserve the native structure and contrast of pharmaceutical powder systems; for example, in Figure 4, FAST mode enables the imaging of loose granules in situ within their capsule,

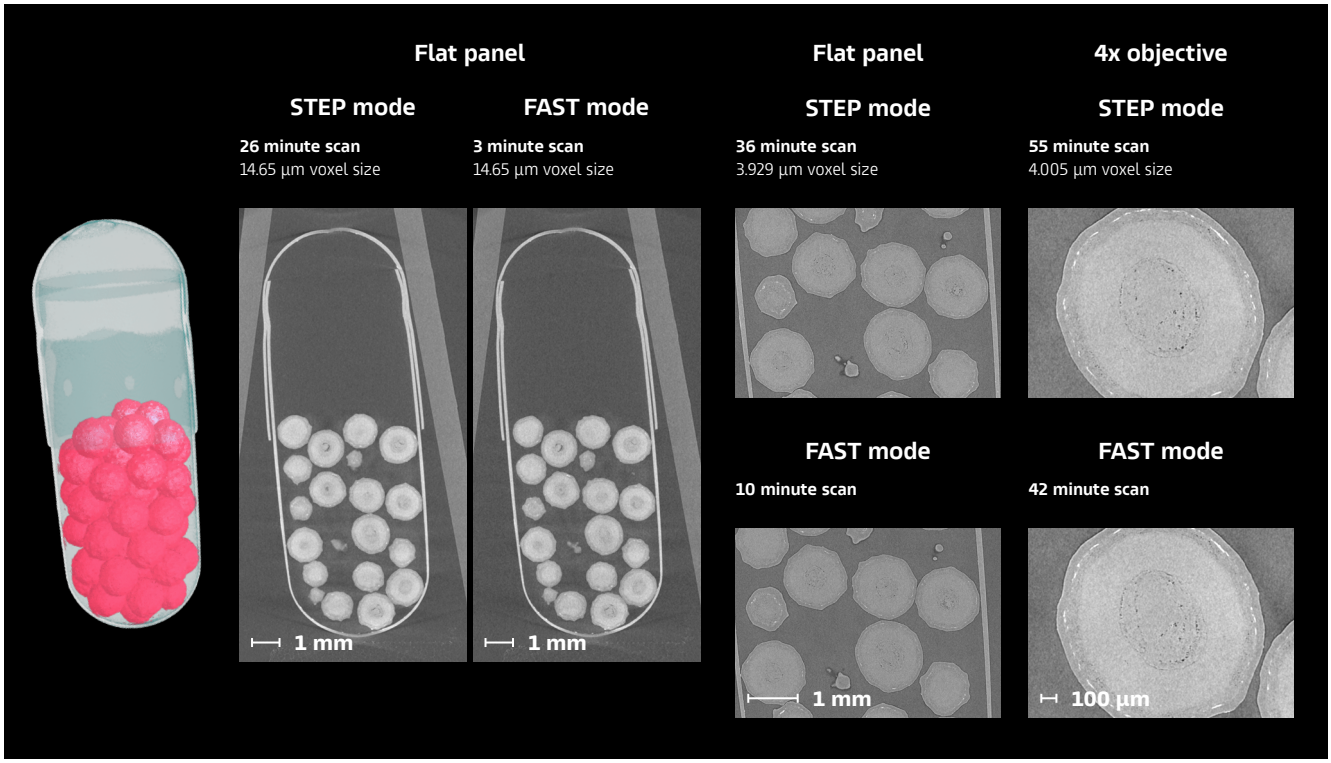


Figure 4: Comparisons of STEP vs FAST mode using the flat panel and 4x for a slow release microgranule capsule. Scans are significantly quicker using FAST mode, which can help alleviate particle movement in capsules like this where there is no particle packing and almost 50% empty space.

which limits particle movements and resists the need to remove the particles from the capsule or further packing, a process that could result in sample damage.

Geosciences: Geological materials such as sediments, crushed rocks, and particulate mineral samples are often heterogeneous and structurally complex. Non-destructive imaging allows these materials to be analyzed without polishing, sectioning, or altering the original particulate arrangement.

FAST mode can help preserve the structure of loose geological particulates. Embedding loose geological samples in epoxy resin reduces X-ray absorption throughout the sample, reducing contrast and image quality in regions where it matters most. Figure 5 shows overview and high-resolution reconstructions of uncompacted, loose sand. Despite the considerable space between neighboring sand particles, the individual particles stayed in place throughout scanning, resulting in high contrast, segmentable boundaries. In the higher resolution 4x scan, the internal structure of each sand particle becomes clear. This type of imaging would not be possible with blurred projection images, which smooth out the contrast of the particle's interior. This is valuable when analyzing grain morphology, packing, mineral distribution, pore networks, or sediment microstructure.

Materials science: Powders play a major role across materials science, often as precursors for manufacturing. Additive manufacturing, powder metallurgy, ceramic casting, and battery preparation all require powder processing. One example is the nylon-glass mixture shown in Figure 6. These precusors are commonly used in additive manufacturing of lightweight polymer composites, where glass microballoons help reduce component weight while maintaining mechanical performance.

Nylon powder containing glass microballoons presents a challenging imaging case due to the wide distribution of particle sizes and the presence of low-density hollow inclusions within a densely packed powder. Using the 4x objective, FAST mode on

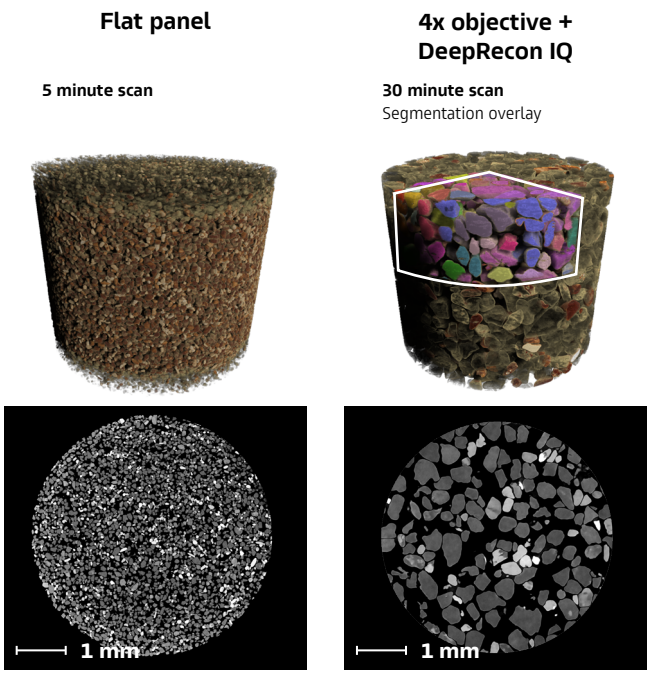


Figure 5: Unpacked (loose) sediment from a California beach. Interior sand particulate structure can be seen in the high-resolution 4x scan.

VersaXRM 5 Insight produced superior image quality compared to the equivalent STEP mode scan, delivering improved contrast and sharper definition of particle boundaries and microballoon features throughout the reconstructed volume. Enhanced visualization enables more confident characterization of powder morphology and particle distribution while maintaining the throughput advantages required for efficient powder analysis workflows.

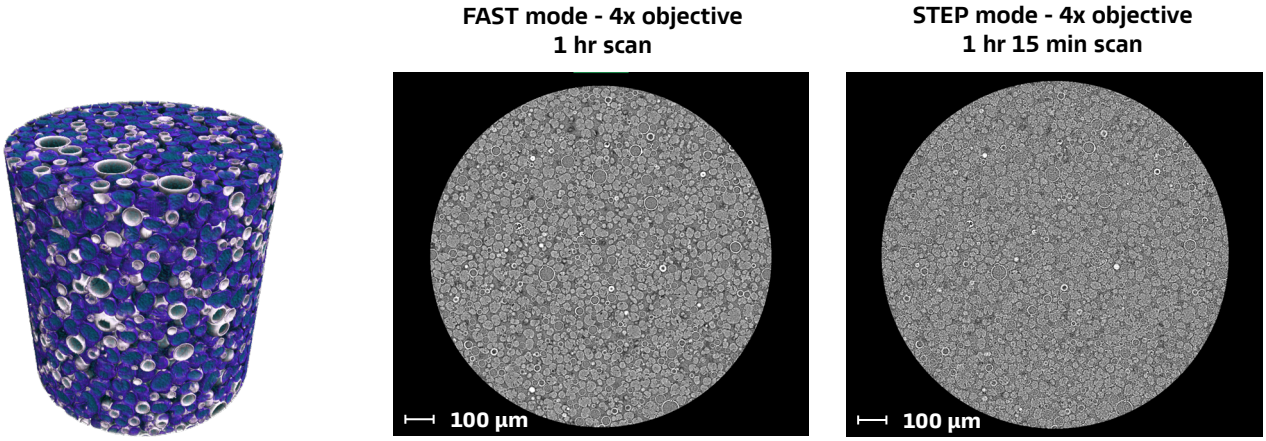


Figure 6: Nylon powder blended with dense glass microballoons for additive manufacturing. The 3D rendering on the left shows the nylon particles and glass microballoons, while the image on the right compares FAST mode and STEP mode scans with 4x objective at 2 µm resolution. Sample courtesy of Dr. Nikhil Gupta, Professor at New York University Tandon School of Engineering.

Conclusion

Powder samples are essential across scientific and industrial fields, but they remain challenging to image using conventional X-ray microscopy workflows. Traditional step-and-shoot tomography can disturb loose particles during acquisition, producing motion blur and reducing the quality of reconstructed datasets. FAST continuous motion scanning on ZEISS VersaXRM 5 Insight provides a powerful alternative. By maintaining steady rotation during acquisition, FAST mode reduces the repeated acceleration and deceleration events that can cause loose powders to move. This enables sharper imaging of unembedded powders, leaving the sample in its natural state, and improves the quality of downstream segmentation.

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