

Recent Advancements in 3D X-ray Microscopes for Additive Manufacturing

Leah Lavery¹, William Harris¹, Hrishi Bale¹ and Arno Merkle¹.

¹ Carl Zeiss X-ray Microscopy, Pleasanton, CA, United States of America.

Three-dimensional X-ray microscopy (XRM) is a powerful sub-surface imaging technique that reveals tomography of three-dimensional microstructure from a range of materials, non-destructively. The non-destructive nature of X-rays has made the technique widely appealing, with the potential for characterizing sample changes in “4D,” delivering 3D microstructural information on physically the same sample over time, as a function of sequential processing conditions or experimental treatments. This has led to a new generation of functional studies with applications and is in a state of rapid expansion [1]. Recently, laboratory-based X-ray sources have been coupled with high resolution X-ray focusing and detection optics from synchrotron-based systems to acquire tomographic datasets with resolution down to 50 nm across a great span of sample dimensions [2]. Additionally, the technique of laboratory based X-ray diffraction contrast tomography has recently become available; allowing the nondestructive routine characterization of 3D crystallographic information on polycrystalline materials in a commercial laboratory X-ray microscope (ZEISS Xradia 520 Versa). Known as laboratory diffraction contrast tomography (LabDCT), this imaging modality will open the way for routine, non-destructive studies of time-evolution of grain structure to complement destructive electron backscatter diffraction (EBSD) end-point characterization. This talk will explore both the implementation of optics in nanoscale and sub-micron laboratory XRM architectures and review in detail several leading applications examples for the field of additive manufacturing including the ability to track changes, in grain size and orientation, over time, e.g. ‘4D’ time lapse studies using LabDCT. XRM for tomography using a laboratory source was used to characterize porosity in additive process control study for various steel input materials and Ti-6Al-4V built with Arcam SEBM. For additional process and material qualification using LabDCT, an example of this capability was used to follow the sintering of copper particles through a series of time-lapsed DCT measurements. XRM tomographic data provides multi-scale imaging and visualization for a wide variety of AM materials, even creation of accurate 3D-printed models in biomimetic studies [4-5]. XRM can provide accurate 3D internal structural information critical to aid computational design of next-generation materials.

Additive manufacturing (AM) techniques can produce complex 3D structures. The reliability and performance of the produced parts relies heavily on the resultant microstructure. Frequently the material performance can be quite sensitive to its discrete, complex pore structure, thereby motivating the need to investigate and understand the morphology of these materials in 3D at the appropriate pore scale. XRM provides a means to perform non-destructive 3D characterization of complex even anisotropic geometries. Beyond imaging, quantification of volumetric properties such as porosity can be calculated from the reconstructed dataset. This is highly dependent on the spatial resolution of the imaging system. Porosity is an important property because for materials destined for high stress applications should be fully-dense to avoid failure in service. XRM has been used to characterize porosity in metal additive process control studies [3]. Figure 1 shows a sintered powder steel cylinder. From the 3D reconstructed dataset (inset), a virtual 2D cross-section (left) reveals several regions of non-sintered volume. In addition to the non-sintered volume, several micron-size cracks were discovered.

Through the incorporation of post-sample optical magnification detector technology, sub-micron XRM

has recently extended the application scope of laboratory-based microCT by extending resolution and contrast. As illustrated in Figure 1, interior (local) tomography is routinely performed, as ZEISS Xradia Versa XRM architecture has been designed to maintain sub-micron spatial resolution for a variety of working distances and sample sizes/geometries. System is capable of imaging at high resolution at large working distance, opening up opportunities for interior tomography of large samples and *in situ* experiments with large environmental cells. Furthermore, new contrast modes have emerged based on dual energy absorption or diffraction (LabDCT).

Soft materials, such as biocompatible photopolymers, consistently pose challenges in generating contrast by several techniques, X-ray absorption included. We demonstrate the application of phase contrast techniques on such materials. The tunable contrast enhancement mechanism (propagation-based) within combines optimized absorption contrast, for enhanced imaging of soft materials (low-Z) such as scaffold or implant materials. Porosity in these materials is often engineered. [5]

Additive manufacturing techniques can produce complex anisotropic 3D structures. The reliability and performance of the produced parts relies heavily on the resultant microstructure from often-proprietary feedstock. Materials designed for high-stress applications can be quite sensitive to discrete, complex pore structure, therefore the need to investigate and understand the morphology in 3D at the appropriate pore scale to avoid premature failure. X-ray microscopes provide internal structural information critical to the evaluation of additive manufacturing materials as well as aid in the design and processing of next generation materials.

References:

- [1] A. Merkle and J. Gelb. *Microscopy Today* **21**, (2013) pp. 10-15.
- [2] A. Tkachuk, *et al.*, *Z. Kristallogr.* **222**, (2007) pp. 650-655.
- [3] S. MacDonald *et al.*, *Scientific Reports*, **5** (2015) 14655.
- [3] J. Slotwinski, *et al.* *J. Res. Natl. Inst. Stan.* **119**, (2014) pp. 494-528.
- [4] L. Wen, *et al.* *J. Exp Biol* **217**, (2014) pp. 1656–1666.

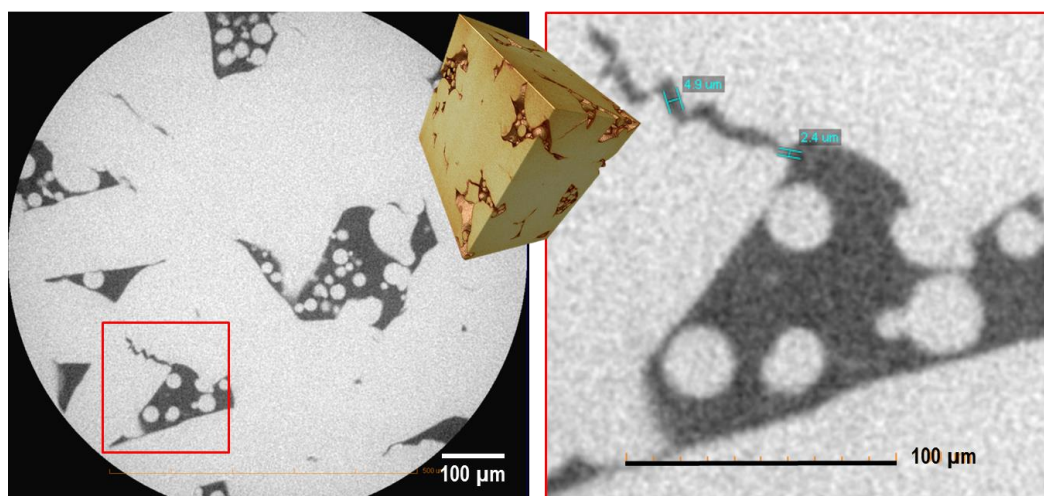


Figure 1 From the 3D reconstructed dataset (inset), a virtual 2D cross-section (left) reveals several regions of non-sintered powder steel volume. In addition to the non-sintered volume, several micron-size cracks were discovered. Sample courtesy of NIST.