Extended Range Ultra Small-Angle X-ray, Small-Angle, and Wide-Angle Scattering for Materials Characterization at 9ID Beamline

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Development of new high-performance materials, e.g., new alloys, ceramics, or polymer materials, is critical for advances in energy production and utilization as well as materials future for everyday life. These materials often exhibit complex microstructures spanning multiple length scales that control their performance. In this context, it is important to simultaneously characterize, ideally in situ or in operando, various facets of the microstructure – for example precipitate shape and size, together with their phase and chemical composition. Advanced Photon Source (APS) with NIST has developed and optimized a combined Ultra-Small, Small, & Wide-Angle X-ray Scattering (USAXS/SAXS/WAXS) facility currently located at sector 9ID beamline. [1] Data spanning over 5 decades in microstructural size can be collected sequentially in 4 to 6 minutes, from the same volume during one in-situ experiment. This facility is available through APS general user program to world-wide user community at the APS sector 9ID (http://usaxs.xray.aps.anl.gov).

Presentation will discuss wide range of USAXS/SAXS/WAXS instrument application. As an example, we will present characterization of complex metallic materials microstructures formed by additive manufacturing (AM) methods. AM of metals is based on a layer-by-layer additive process, in contrast to traditional manufacturing processes that often require labor-intensive and costly subtraction or forming.[2] AM technologies provide great flexibility in manufacturing parts with complex geometrical shapes and can significantly reduce manufacturing lead times and associated cost. Thus, AM is fast becoming an attractive option for the fabrication of increasingly complex and high-valued metal components in the aerospace, oil & gas, automobile, electronics, and biomedical industries. Unfortunately, elemental segregation can present a ubiquitous problem for AM parts due to solute rejection and redistribution during the rapid solidification process. Using a series of USAXS/SAXS/WAXS *in situ operando* studies, together with electron microscopy & thermodynamic modeling, we show that in one AM Ni-based superalloy, Inconel 625, deleterious δ-phase precipitates grows on much shorter time scales than in the corresponding wrought alloys (i.e., hours *versus* hundreds of hours)[3-5] and we offer a mitigation strategy for producing AM 625 with homogeneous composition and reproducible microstructure.

[1] J. Ilavsky, F. Zhang, R. N. Andrews, I. Kuzmenko, P. R. Jemian, L. E. Levine and A. J. Allen; *J. Appl. Cryst.* **51**(3), 867-882 (2018).

[2] I. Gibson, D.W. Rosen & B. Stucker. *Additive manufacturing technologies*, Springer, 2010.

[3] Y. Idell, L.E. Levine, A.J. Allen, F. Zhang, C.E. Campbell, G. Olson, J. Gong, D. Snyder & H. Deutchman; *JOM*, **68**, 950-959, (2016).

[4] F. Zhang, L.E. Levine, A.J. Allen, C.E. Campbell, E.A. Lass, S. Cheruvathur, M.R. Stoudt, M.E. Williams & Y. Idell; *Scripta Mater.,* **131,** 98-102 (2017).

[5] F. Zhang, L.E. Levine, A.J. Allen, M.R. Stoudt, G. Lindwall, E.A. Lass, M.E. Williams, Y. Idell & C.E. Campbell; *Acta Mater.,* 152, 200-214 (2018).

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