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In Situ Synchrotron and Neutron Characterization of Additively Manufactured Alloys

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Additive manufacturing (AM) of metals and alloys represents a suite of emerging manufacturing processes that fabricate parts layer-by-layer following a three-dimensional digital model. Since its inception, AM has proven effective for concept modeling and rapid prototyping, and during the last decade, has started to realize its revolutionary potential to impact the manufacturing industry for the production of customizable, high-value parts with complex shapes and geometries in, e.g., the aerospace, automotive, medical, and military sectors. As one of the most rapidly growing and heavily invested components of the manufacturing industry, AM is currently undergoing rapid advancement, with new and innovative concepts and applications constantly being developed.

Despite the growing use of AM, a number of challenges, such as the lack of established qualification procedures and thorough understanding of the nonequilibrium fabrication processes, continue to impede its more widespread adoption. In response to these challenges, the most recent quadrennial Strategy for American Leadership in Advanced Manufacturing, released by the US National Science and Technology Council in 2018, set out four strategic goals for AM development, including to: continue advancements in process control and process monitoring to secure AM technologies as viable production alternatives, develop new methods to measure and quantify the interactions between material and processing technology to better understand the material–process–structure relationship, establish new standards to support the representation, presentation, and evaluation of AM data to ensure part quality and reproducibility, and expand research efforts to establish best practices for applying computational technologies to AM, including simulation and machine learning.[1]

Each of these goals emphasizes the essential roles of measurement science and standardization in the continued growth of AM technologies. At the same time, it is important to acknowledge that critical measurement science barriers, challenges, and gaps exist. Such barriers include limited in situ monitoring and measurement capabilities needed to provide robust data to characterize material deposition, detect build anomalies, and provide feedback control; and also a limited proper understanding of post-build processing that can effectively and reproducibly control materials properties to enable part qualification and certification. Because of the rapid layer-by-layer fabrication of AM, these measurement science challenges usually encompass several orders of magnitude in the spatiotemporal domain and require us to search deep into our materials characterization arsenal and invent new characterization techniques as required.

Synchrotron radiation sources and neutron facilities worldwide, due to their powerful and versatile materials characterization capabilities, have joined this quest and become an increasingly vital component. Synchrotrons can generate high-energy x-rays (on the scale of 100 keV), which allow deep penetration into AM alloys due to the significantly reduced photo-absorption cross section. This enables measurements of bulk AM materials in complex sample environments, such as high-temperature furnaces or mechanical testing stages. Synchrotron radiation also has extremely low emittance and high photon flux, enabling measurements

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with high spatial resolution, such as those aimed at resolving the local strain and texture of the highly heterogeneous AM materials. The high synchrotron photon flux and the availability of fast, pixelated, and efficient x-ray detectors, when combined, can provide sub-millisecond time resolution that proves highly effective for capturing melt pool evolution and phase transformations during/after solidification in AM processing. Neutrons, because they are uncharged and only interact with atomic nuclei via the strong force, possess an even greater capability to penetrate metallic materials than high-energy x-rays and enable measurements of voids, phases, and residual stresses in large, industry-relevant AM parts.

Because of these unique capabilities, many state-of-the-art x-ray and neutron characterization techniques have been applied to understand the processing, structure, and properties of AM metals. To provide a forum for scientific discourse between AM researchers and synchrotron and neutron scientists, The Minerals, Metals, and Materials Society (TMS) Advanced Characterization, Testing, and Simulation Committee and the Additive Manufacturing Committee sponsored a 4-day symposium at the 149th TMS Annual Meeting and Exhibition (San Diego, CA, February 2020). A broad range of scientific and engineering topics were discussed, with a focus on in situ/*operando* characterization of AM processing, nondestructive evaluation and quantification of AM parts, microstructural evolution during post-build processing, and the role of precise measurements in AM modeling and benchmarking, particularly those targeted at applications in an industrial setting. This *JOM* special topic includes five examples that highlight the potential of synchrotron and neutron techniques to lead to major breakthroughs in understanding AM materials at a fundamental level and to contribute to the development of AM technology and industrial innovation.

High-speed synchrotron x-ray imaging and diffraction are among the most hotly pursued areas in AM research. These techniques take full advantage of the spatial and temporal resolution provided by the intense and tunable hard x-ray synchrotron sources and can capture the subsurface dynamics, in contrast to traditional visible-light and infrared imaging that are limited to the surface and above. In the imaging mode, equipped with a kilohertz or faster x-ray detector, these techniques have revealed many important transient behaviors related to laser powder-bed fusion (L-PBF), arguably the most prevalent AM fabrication method, including keyhole formation and morphology, powder spreading dynamics, melt-pool flow dynamics, etc. In a paper entitled "Study of Powder Gas Entrapment and Its Effects on Porosity in 17-4 PH Stainless Steel Parts Fabricated in Laser Powder Bed Fusion," Wu et al.[2] designed an elegant study in which they intentionally varied the level of gas entrapped in 17-4 PH stainless-steel powder and performed in situ high-speed x-ray imaging, dynamic x-ray radiography, and x-ray computed tomography measurements to evaluate the extent to which the gas initially entrapped in the powder affected the porosity of parts fabricated by L-PBF. They found that approximately 30% of the gas entrapped in the powder was transferred to the as-built part and established that the level of gas typically entrapped in commercial AM powders does not lead to an appreciable net-part porosity increase in L-PBF processes.

In addition to L-PBF, directed energy deposition (DED) represents another mainstream AM fabrication technology. DED offers the flexibility to fabricate functionally graded materials by using multiple nozzles. With its larger melt pool, DED often has a faster build rate when compared with L-PBF. However, due to the powder–laser interaction, the powder flow in a DED process introduces constant changes in the laser attenuation, leading to fluctuations in the resulting microstructure and thus making it essential to monitor the influence of process parameters such as the powder flow rate, melt pool dynamics, and porosity formation. In a paper entitled "In Situ Observations of Directed Energy Deposition Additive Manufacturing Using High-Speed x-ray Imaging," Wolff et al.[3] demonstrate the influence of powder flow rates using two separate DED systems, one with high powder flow rates ($\sim$ 6 m/s and $\sim$ 10 m/s) to represent industry-scale DED processing and the other with low powder flow rates ($\sim$ 1.5 m/s) to enable individual powder tracking. One of their main findings was that powder flow was the main reason for melt pool fluctuations in DED, in contrast to keyhole instability as found for L-PBF. A minimal threshold for the powder mass flow rates of between 10 mg/s and 50 mg/s was also established for their setups to prevent keyhole formation.

AM processing is highly localized and transient. To control the build quality, build parameter optimization, which is often conducted through a combination of experiments and modeling, is critical. Due to the complexity of the key physical phenomena involved in AM, the validation data are multidimensional in nature, and the data completeness and fidelity are critical. Gould et al., in "In Situ Analysis of Laser Powder Bed Fusion Using Simultaneous High-Speed Infrared and x-ray Imaging"[4] presented an experimental framework that combines high-speed infrared imaging and x-ray imaging to simultaneously monitor the surface behavior and the melt pool dynamics in situ. They demonstrated the effectiveness of this combined methodology for detecting vapor plume dynamics, calibrating the emissivity of different materials at their melting temperatures, and connecting defect formation with thermal signatures.

While high-speed x-ray imaging work has elicited much excitement, high-speed x-ray diffraction has the potential to reveal the oftentimes unexpected phase-transition pathway during rapid solidification in metallic AM parts and to advance understanding of the phase landscape of these advanced materials. In a paper entitled "In Situ Characterization of Phase Evolution in Ni Alloy 718 during Laser Melting Using High-Speed x-ray Diffraction", Oh et al.[5] demonstrated the transient behaviors of different phases before, during, and after the melting process in Inconel 718, a popular choice for AM processing due to its superior performance even at temperatures as high as 923 K. At a cooling rate of $\sim 10^4$ K/s, the authors found continuous formation of γ' and γ'' phases during cooling until the temperature reached approximately 923 K. The final volume fractions of γ' and γ'' were estimated to be 11% and 3%, respectively. Interestingly, even though both MC carbide and δ intermetallics were present prior to melting, after melting, MC carbide and Laves phase were identified, suggesting that the formation kinetics of different intermetallics may be related to the cooling speed.

Finally, Capek et al.[6] presented a very interesting study on Inconel 718 under uniaxial tensile loading using in situ neutron diffraction. Two conditions of Inconel 718 were used: as-built and after thermal annealing at 1123 K for 8 h. While the as-built Inconel 718 consisted of γ, γ'', and Laves phases, γ'' was found to completely transform to δ phase in the annealed sample. These δ precipitates serve as dislocation barriers, strengthening the annealed alloy by $\sim$ 20% and making it less ductile. Through a load balance analysis, this work also suggests that the δ precipitates in the annealed sample can carry significant load, elucidating the role of different fractions and types of precipitates in the mechanical performance of AM Inconel 718.

With these five selected examples, we hope that this special topic will provide the audience of *JOM*, particularly those with a strong interest in AM, with a flavor of the unique capabilities and exciting opportunities provided by synchrotron sources and neutron facilities worldwide. We truly believe that digital fabrication exemplified by additive manufacturing has the potential to transform the industrial landscape and our daily lives. While many critical questions remain to be answered and technical challenges remain to be addressed, we are convinced that these advanced x-ray- and neutron-based characterization techniques will be essential to materialize the revolutionary potential of additive manufacturing.

To read or download any of the papers, follow the URL http://link.springer.com/journal/11837/73/1/page/1 to the table of contents page for the January 2101 issue (vol. 73, no. 1).

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