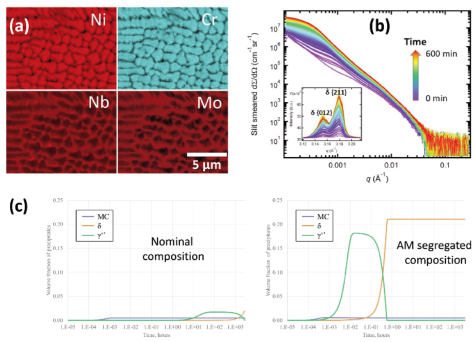
The Advanced Photon Source

Putting the Heat on an Additive-Manufactured Alloy



(a) Energy dispersive x-ray spectrometry compositional mapping of as-built AM IN625 alloy clearly demonstrates severe elemental microsegregation introduced by rapid cooling and solidification in the AM build process. (b) Simultaneous synchrotron USAXS and XRD data reveal an unexpected, fast formation of deleterious delta phase in AM 625 and its morphological and structure transformation kinetics during a stress-relief heat treatment at 870° C. (c) Thermodynamic calculations unequivocally show that the elemental microsegregation (right panel) leads to a much accelerated (> 1 × 10^2) formation of precipitate phases comparing with the nominal composition (left panel).

As the techniques of additive manufacturing (AM), or “3-D printing,” become more versatile and applicable for diverse purposes, they sometimes pose unique challenges not present with more traditional manufacturing methods. For instance, in additive-manufactured metals, microstructural defects that lead to reduced strength and stress resistance can form, an issue commonly addressed by post-build heat treatment. Researchers used the U.S. Department of Energy’s Advanced Photon Source (APS) to examine the AM alloy Inconel 625 (IN625) in an effort to better understand the effects of heat treatment on AM alloy microstructure and phase evolution.

A widely-used superalloy in many industries where its high strength and corrosion resistance are critical advantages, IN625’s limited machinability makes it attractive for AM techniques. The investigators used electron microscopy and thermodynamic modeling, and *in situ* synchrotron small-angle and ultra small-angle x-ray scattering (USAXS) and x-ray diffraction (XRD) techniques for experiments at the X-ray Science Division (XSD) 11-BM-B powder diffraction beamline and the XSD 9-ID-C USAXS facility.

The AM principle of using a laser or electron beam to build up material layer by layer is versatile, but it is also a volatile and somewhat unpredictable process at the microscale, involving complex repeated cycles of heating and cooling that inevitably result in a certain degree of microstructural inhomogeneity. One effect often seen in AM metals is elemental segregation, which can create defects including residual stress and nonequilibrium phase structure. The unexpected side effects of these post-build heat treatments were the focus of the current work, which includes some of the first detailed *in situ* investigations of the phenomenon.

The *in situ* x-ray scattering and diffraction techniques at the APS USAXS facility revealed the actual formation of undesired platelet-shaped delta-phase precipitates. These methods also allowed both time- and temperature-dependent studies of the phase evolution kinetics within the same sample volume, including enough data for thermodynamic modeling. Across the entire temperature range of the heat treatment from 800° C to 870° C, the delta-phase precipitates grew very rapidly, beginning less than five minutes after the commencement of heat treatment, suggesting a very low nucleation barrier. The growth kinetics was found to be at least 100 times faster than with conventional alloys of similar composition.

Applying a second heat treatment at 1150° C for 1 h succeeded in removing the delta-phase precipitates, resulting in a single-phase face-centered cubic structure. But this second heat treatment also promoted grain growth that could weaken the alloy's mechanical strength. The research team is continuing to explore, in greater detail, the benefits and adverse effects of this homogenization heat treatment.

Although the current study was confined to AM IN625, the investigators note that their general findings are applicable to many other AM alloys, since phenomena such as elemental segregation with undesired phase precipitates are an almost inevitable byproduct of AM technology. Extending the multifaceted approach taken in this work to other AM alloys will lead to a better understanding of their phase evolution in comparison with their wrought counterparts, and ultimately to new strategies to avoid or mitigate microstructural defects of additive-manufactured metal alloys.

— *Mark Wolverton*

See: F. Zhang et al., “Effect of heat treatment on the microstructural evolution of a nickel based superalloy additive-manufactured by laser powder bed fusion,” Acta Materialia **152**, 200 (2018). DOI: 10.1016/j.actamat.2018.03.017

Call for APS General-User Proposals

The Advanced Photon Source is open to experimenters who can benefit from the facility’s high-brightness hard x-ray beams.

General-user proposals for beam time during Run 2019-1 are due by Friday, October 26, 2018.

Information on access to beam time at the APS is at http://www.aps.anl.gov/Users/apply_for_beamtime.html or contact Dr. Dennis Mills, DMM@aps.anl.gov, 630/252-5680.

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