doi:10.1017/S1431927622007516
Microsc. Microanal. 28 (Suppl 1), 2022

In-situ TEM Laser Heating for Manipulation of Cooling Rates and Observation of Precipitate Dissolution Kinetics

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Using in-situ TEM to observe materials at high temperatures is a popular way to analyze material behavior in extreme environments. The development of in-situ laser heating enables determination of mechanisms of microstructural evolution during heat processing such as welding, annealing and laser processing, such as many metal additive manufacturing processes. All of these techniques include high heating and cooling rates creating far-from-equilibrium conditions. In-situ laser TEM allows site-specific heating of the sample and has previously been used to synthesize nanowires, increase grain size, and initiate phase transformations in metals [1, 2]. The I^3TEM facility at Sandia National Laboratories is equipped with a specimen drive infrared (IR) laser with a maximum power of 20 W, enabling high temperatures and fast cooling rates to be applied to samples in single and double tilt, as well as heating, cryogenic, and other environmental TEM holders. Combining in-situ laser heating with an environmental holder such as a low temperature cryogenic holder enables the manipulation of cooling rates beyond those reached in environmental holders alone, resulting in observation of the differences in microstructural evolution induced by widely varying cooling rates.

As an exemplar, a stainless steel 304L VAR alloy was used to observe differences in dissolution kinetics of boride precipitates at high temperatures with varying cooling rates. The alloy examined is known to contain delta ferrite stringers associated with boride precipitates, the latter of which is known to dissolve during annealing. Observation of the dissolution of boride precipitate using in-situ laser TEM (Figure 1) is dependent on the reflectivity of the material, the laser power, and the stability of the temperature reached. The determination of the temperature of the sample in in-situ laser TEM experiments is not trivial due to the reflectivity of different materials in the sample; therefore, selected area diffraction (SAD) patterns were used to attempt to determine expansion of the austenite lattice which, when combined with the austenitic coefficient of thermal expansion (CTE) for 304, would result in the temperature of the sample. However, degradation of the diffraction pattern during laser exposure, particularly at high laser powers, results in unreliable temperature calculation. A FLIR camera to measure IR light emitted by the sample is currently being incorporated to measure the temperature reached.

To observe the difference in behavior of the boride precipitate at varying cooling rates, the in-situ laser was combined with heating and cryogenic TEM stages. Use of a heating stage leads to a slower cooling rate after high temperature is reached, enabling closer observation of microstructural kinetics similar to those achieved during furnace cooling, while cooling the sample to cryogenic temperatures during laser exposure enables observation of kinetics similar to those seen during rapid solidification processes such as laser additive manufacturing [3].

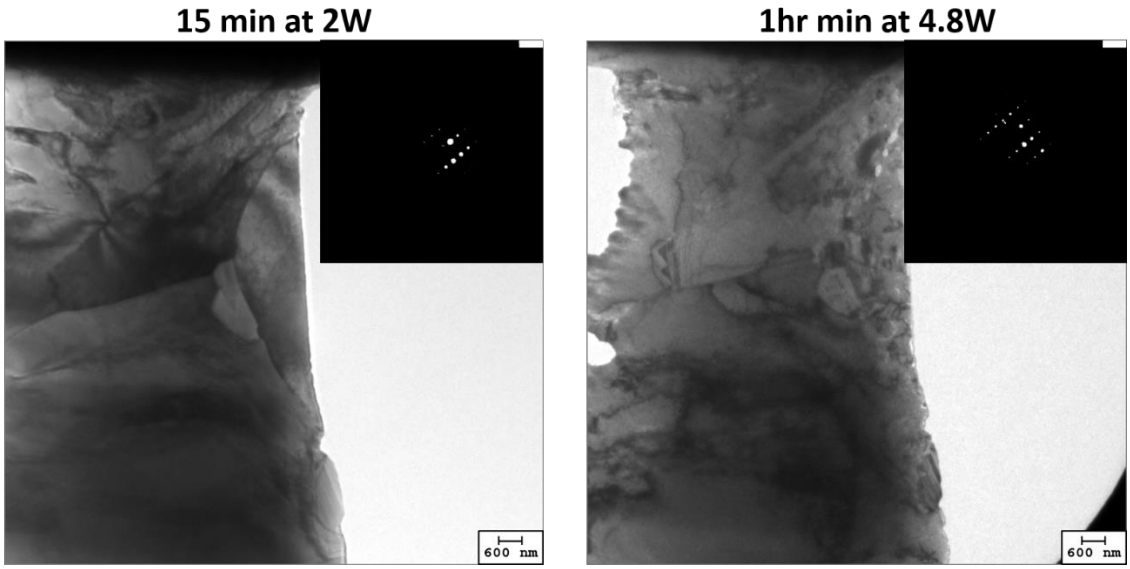

Figure 1. Stainless steel 304L VAR after 15 minutes at 2 W (left) and after 1 hour at 4.8 W laser power. The borides are both still present while the austenite matrix and delta ferrite stringer are degraded. The diffraction patterns in both states are inserted.

References:

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[2] Y Wu et al., Microscopy and Microanalysis **24**(6) (2018), p. 647.
[3] The authors would like to acknowledge Daniel Perry and Damion Cummings at Sandia National Laboratories for preparation of FIB lift-outs used in the above experiments. Shen Dillon and Keith Coffman are gratefully acknowledged for use of their strain calculation MATLAB code.
This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.