2179
doi:10.1017/S1431927615011678 Paper No. 1088
Microsc. Microanal. 21 (Suppl 3), 2015

Super-X XEDS STEM Tomography of γ′ Precipitates in the LSHR Nickel Superalloy

S.J. Kuhr[1], J.M. Sosa[1], D.E. Huber, H.L. Fraser[1]

[1.] Center for the Accelerated Maturation of Materials, The Ohio State University, Columbus

Electron tomography has been proposed to be advantageous for reconstructing sub-micron precipitates in nickel superalloys. However, not every type of electron image provides the necessary contrast for high fidelity tomographic reconstruction. The γ′ precipitates in Low Solvus High Refractory (LSHR) form coherently with the matrix and exhibit limited atomic number contrast relative to the matrix when imaged with traditional transmission (TEM) and scanning transmission electron microscopy (STEM) bright field/dark field imaging. Previous research has shown that energy filtered TEM (EFTEM) has displayed successfully γ′ morphologies based off the Cr-$L_{3,2}$ edge[1,2]. However, EFTEM is hindered by poor signal to noise during image collection requiring prohibitive acquisition times. X-ray energy dispersive spectroscopy (XEDS) in STEM offers a robust method to collect a wide range of compositional information and enables spectral image (SI) collection that permits post-processing analysis of multiple elemental species. The primary objective of the work to be presented was to use Super-X™ XEDS in STEM to acquire compositional maps of LSHR to produce tomographic reconstructions that accurately depict γ′ precipitates within a dual-microstructure heat treatment (DMHT) gradient [3] as shown in Figure 1(a). Chromium is a significant component of the LSHR alloy at 12.3 wt% and segregates to the γ matrix, therefore chromium XEDS SI can provide strong contrast between the γ′ precipitates and the chromium-rich matrix. The XEDS SI shown in Figure 1(b) was used for the full precipitate reconstruction as displayed in Figure 1(c). The DMHT gradient contains a range of intricate γ′ morphologies that have proven difficult to characterize with two-dimensional methods. Three-dimensional characterization not only provides the morphology of the γ′, but will also permit more accurate microstructural metrics for inputs for integrated computational materials (ICME) models.

Images for Super-X XEDS tomographic reconstructions were acquired using a FEI Titan™ G2 60-300 S/TEM equipped with Super-X™ technology. The Super-X™ collection system is composed of four silicon drift detector (SDD) covering a 0.7 srad collection angle providing for fast and efficient XEDS SI collection with nanometer scale spatial resolution. A tomographic tilt series was acquired from needle-shaped LSHR specimens fabricated using a Helios™ dual-beam FIB. Using a needle shape geometry and tilting along the longitudinal axis avoids projected thickness variations that usually occur with conventional thin foils. SIs were collected with acquisition times of 300s/image and 20 μs/pixel dwell times. The tilt series was collected from -62° to +62° at 2° increments.

Due to the limited volume contained in the extracted needles, additional characterization techniques were applied for a more complete γ′ reconstruction. To this end, FIB serial sectioning was performed using images formed from secondary electron (SE) contrast [4]. Successive layers of the samples were removed via ion milling and subsequently SE imaged. Figure 2(a) displays the reconstructed volume of precipitates after image processing and feature extraction using MIPAR™ [5]. Figure 2(b) shows the non-edge touching precipitates of the reconstructed volume. Both types of acquired data required rigorous image processing to account for subtle changes in contrast and image translation during data collection.

The reconstructions produced by FIB revealed an apparent interconnected nature of the 200nm γ′ precipitates and also provided an ability to isolate a single precipitate. As this reconstruction was based on SE contrast, it provided no information about compositional differences between the precipitates and the matrix of the material. Conversely, the Super-X XEDS reconstruction offered compositional information as well as the ability to reconstruct both size scales of γ′ precipitation in the LSHR nickel superalloy. XEDS/STEM acquisitions were able to capture particles approximately 10 nm in size. It was determined that FIB serial sectioning provided a larger number of precipitates to analyze and the Super-X™-XEDS reconstruction offered composition and precipitate morphology information with nanometer scale spatial resolution. From the observations made in this study, it was apparent that both reconstruction techniques provided valuable metrics that could be useful with ICME models.

References:

[1] G. B. Viswanathan, *et al*., Acta Materialia **53** (2005), p. 3041-3057.
[2] P.M. Sarosi, *et al*., Ultramicroscopy **103** (2005), p. 83-93.
[3] J. Gayda, *et al*., Superalloys (2004), p. 323-329.
[4] M.D. Uchic, *et al*., Ultramicroscopy **109** (2009), p. 1229-1235.
[5] J.M. Sosa, *et al*. Integrating Materials and Manufacturing Innovation **3** (2014), p. 18.

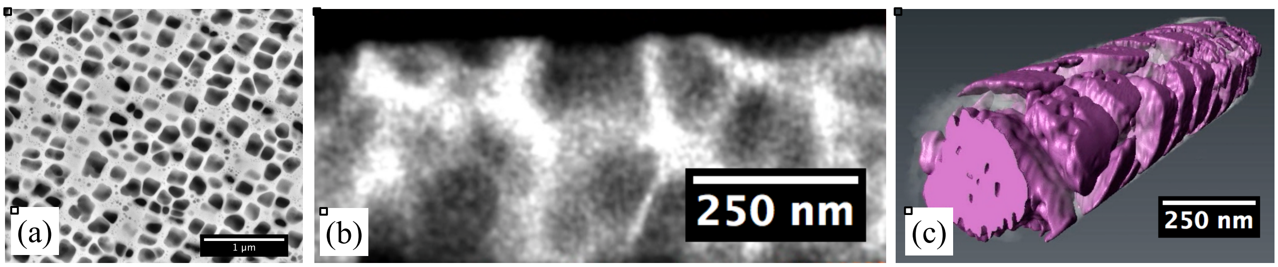

Figure 1. (a) High resolution scanning electron microscope image of LSHR after a γ′ etch (b) Titan™ chromium EDS map in gray scale (c) Full rendering of Titan™ γ′ data

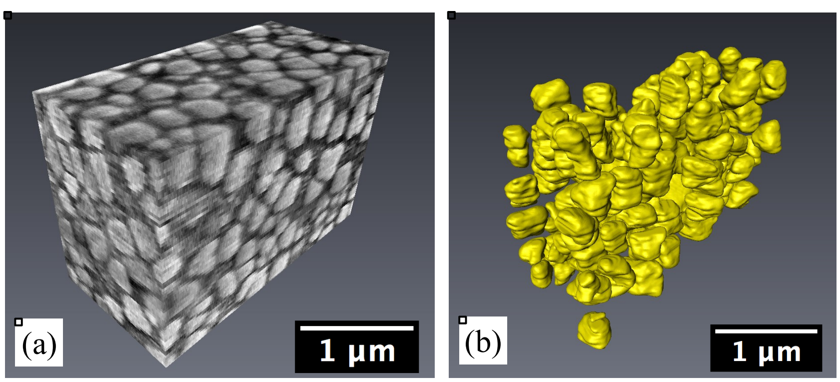

Figure 2. (a) γ′ volume element collected using serial sectioning performed in the DualBeam™ FIB (b) Reconstruction of non-edge γ′ precipitates