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3D ChemiSTEM™ Tomography of Secondary and Tertiary γ' Precipitates in Ni-based Superalloys

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Nickel based alloys have brought significant advancements to the aerospace and power industries due partly to their excellent mechanical strength, surface stability, creep and corrosion resistance at elevated temperatures. Indeed, mechanical properties of nickel based alloys are influenced greatly by the size, distribution and volume fraction of secondary and tertiary γ′ (Ni_3Al) precipitates [1]. The ability to accurately quantify such precipitates is critical to refine predictive models [2], yet has proven difficult due to precipitate size (5 – 200nm) and inadequate imaging techniques. Recent improvements in energy x-ray dispersive spectroscopy (EDS), however, now allows for high fidelity chemical mapping with ~1 nm resolution.

For this work, a FEI Titan 60-300 ChemiSTEM™, equipped with a quad-detector SDD for rapid EDS signal acquisition, was used to collect a tomographic tilt series of EDS spectral images containing secondary and tertiary γ′ precipitates. One distinct advantage of the ChemiSTEM™ technology is the ability to collect signal on the order of image acquisition times. For this particular work, dwell times were 20 us/px, with a live time of 600 s. The tomographic tilt series was collected along a single axis from -30° to +30° in 2° increments. In order to collect adequate signal, beam currents of ~2 nA were used. Beam amperages of this magnitude pose a serious threat to the stability of most any material; however, when repeated imaging of the same local area is required, as with tomography, beam damage manifests as a prohibitive factor. One objective of this work was to explore methods of reducing the number of images necessary for accurate tomographic reconstruction and will be discussed.

Following collection, EDS maps of salient alloy components were extracted from the spectral data for each tilt angle using a standardless Cliff-Lorimar quantification. The HAADF image and corresponding EDS maps representing Cr, Ni, and Al, collected at +10° tilt, are shown in Figure 1 (a-d). The Cr EDS map, Figure 1b, exhibited strong contrast between the γ and γ' phases and therefore, Cr was chosen as the chemical signal on which to base the tomographic reconstruction.

The aligned data suggested that either a second tilt axis, β, was non-zero, or that the sample was not level within the specimen holder. As a result, the tilt axis appeared to shift orthogonally as the sample tilted from one side of 0° to the other. To correct this, non-rigid cubic b-spline image registration [3] was performed between the 0° and 12° Cr maps. Such registration was then used to quantify the tilt-axis' departure from vertical (within the image), as shown in Figure 2. Each Cr map was rotated uniformly by this departure to correct for the sample's non-zero β-tilt. To the best of the author's knowledge, this is the one of the first applications of non-rigid image transformation for the purpose of quantifying a tomographic tilt-axis.

Currently, a variety of reconstruction techniques, such as back-projection, weighted back-projection, ART, and SIRT exist for tomographic reconstruction; however, all are dependent on the size of the missing wedge. In an attempt to address the issue of beam damage and sample stability, the efficacy of the aforementioned techniques was explored and will be presented.

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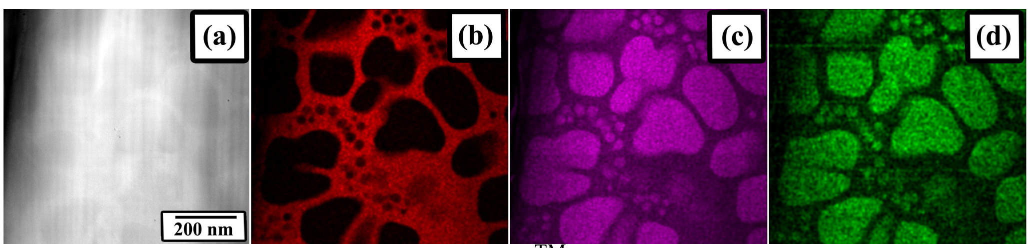

Figure 1. (a) A HAADF map, and ChemiSTEM™ maps of (b) Cr, (c) Ni, and (d) Al of low-solvus high-refractory (LSHR) Ni-based superalloy acquired at +10° α-tilt

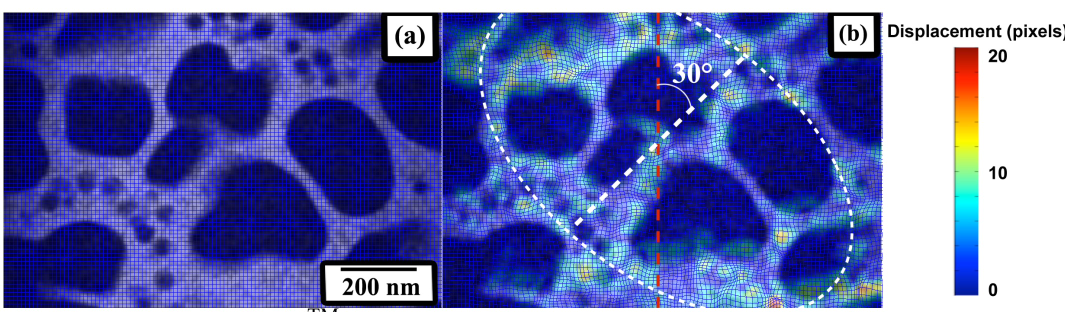

Figure 2. A Cr ChemiSTEM™ map at 12° (a) before and (b) after non-rigid transformation which demonstrates tilt-axis quantification by measuring the orientation of an ellipse fit around the domain of transformation displacement maxima

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