Electron Microscopy Technique for Imaging Ultra Fine Scaled γ′ Precipitates in Nickel Based Superalloys

Introduction

Nickel Based Superalloys are used extensively in gas turbine engines because of their desirable mechanical properties at elevated temperatures and under load bearing conditions.

Since many of the sought after attributes of this alloy system are primarily controlled by the γ′ precipitates, it is essential to accurately quantify their size, volume fraction, and morphology so that this data may be incorporated into microstructural evolution and mechanical property modeling.

Research Goals

For disk applications, the cutting edge microstructure consists of a bimodal distribution of secondary and tertiary γ′ (Ni_3Al,Ti) precipitates of the following size scale:

Secondary γ′ (100-250 nm)
Tertiary γ′ (5-30 nm)

Imaging of such *ultra fine-scaled precipitates* proves to be a great challenge under conventional TEM and STEM characterization techniques.

Therefore, the objective of this work was to optimize an imaging technique that can be utilized to rapidly *and* accurately acquire images at a ***nano-scaled level of resolution.***

Characterization Methodology

Energy **F**iltered **T**ransmission **E**lectron **M**icroscopy **(EFTEM)** is an analytical characterization technique used to produce elemental maps. Images are formed from electrons scattered at characteristic elemental ionization edges. Degrading artifacts and inelastically scattered electrons at all other energy levels are then filtered out and thus do not contribute to the image construction. The result being a high resolution chemical map. In Ni-based Superalloys, the γ matrix is enriched in elements such as Cr, Mo, Co, W, and Zr while the γ′ precipitates are comprised of Ni_3Al,Ti. Due to the differences in ionization edge potentials it is possible for the EFTEM imaging technique to ***distinguish γ′ from the γ matrix.***

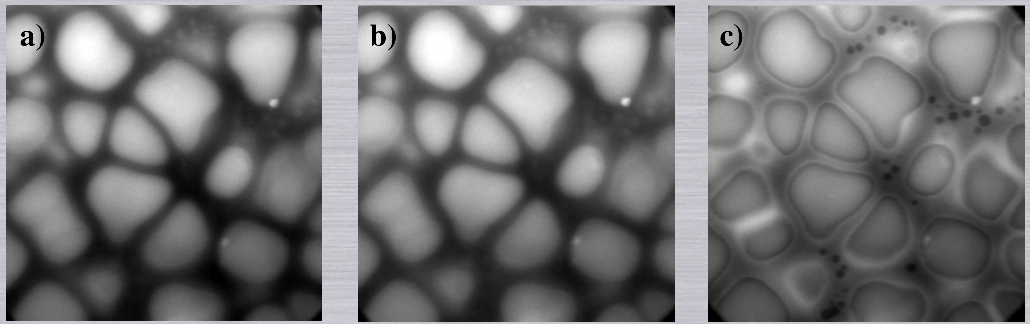

Figure 1. EFTEM micrographs of the Pre and Post Cr $L_{2,3}$ ionization edge at **a)** Pre Edge 1 [495eV] **b)** Pre Edge 2 [565eV] and **c)** Post Edge [585eV].

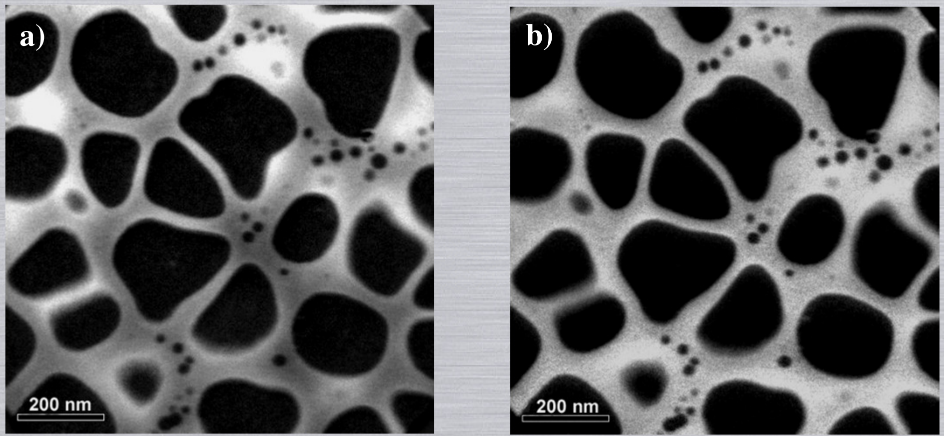

Figure 2. High resolution EFTEM micrographs that clearly resolve the ultra fine scaled, bimodal distribution, of γ′ precipitates in Nickel Based Superalloy René 104. **a)** Elemental Cr Map and **b)** Cr Jump Ratio Map

EFTEM imaging performed on a FEI Tecnai TF20 Transmission Electron Microscope

Experimental Results

Superalloy René 104 was used to substantiate the EFTEM imaging technique. Thin TEM foils were prepared by electropolishing in a solution consisting of 10%$HClO_4$ and 90% Methanol at -40°C/15V.

The Cr $L_{2,3}$ ionization edge (575eV) was used to obtain Pre and Post Edge images under an acquisition time of *40 seconds*.

(Figures 1 a-c)

The elemental Cr map was then created by subtracting the two Pre Edge images from the Post Edge image, thereby yielding an edge intensity map with the brighter region being that enriched in Cr.

(Figure 2a)

A higher resolution Cr jump ratio map can also be constructed by subtracting Pre Edge Image 2 from the Post Edge image.

(Figure 2b)

Technical Significance

The EFTEM imaging technique has proven to be a superior method for rapidly acquiring high resolution images of γ′ precipitates in Ni-based Superalloys down to a size scale of ~5nm.

The size, volume fraction, and morphology can then be *accurately* characterized using quantitative image analysis techniques.

Raymond R. Unocic*
Peter M. Sarosi**
Michael J. Mills***

Department of Materials Science and Engineering
The Oho State University
Columbus, OH 43210

***Graduate Research Associate**
****Post Doctoral Researcher**
*****Professor**

Contact Information

477 Watts Hall
2041 College Rd
Columbus, OH 43210

unocic.4@osu.edu
sarosi.2@osu.edu
614-688-3409

mills.108@osu.edu
614-292-7514