Anomalous USAXS Investigation of Ni-based Superalloys

P.R. Jemian,[1] R.A. Gerhardt,[2] G.G. Long[3]

[1]University of Illinois at Champaign-Urbana (UICU), Urbana, IL, U.S.A.

[2]Georgia Institute of Technology, Atlanta, GA, U.S.A.

[3]National Institute of Standards and Technology (NIST), Gaithersburg, MD, U.S.A.

Introduction

Commercial nickel-based superalloys have been used in high-temperature applications for a number of years because their microstructure contains coherent intermetallic γ' precipitates, such as Ni_3Ti or Ni_3Al, that provide stability under applied loads and thermal cycling [1]. However, long-term exposure at high temperatures can result in coarsening of the precipitates as well as dissolution and reprecipitation. Since the stability of these alloys at their operating temperature is a key concern, understanding the evolution of the precipitate population might be the key to understanding the mechanical behavior of components that utilize these types of materials. It is therefore desirable to develop a methodology for detecting changes in the precipitate population in a nondestructive way so that life prediction models can be modified to account for these changes.

Waspaloy® [2], a Ni-based commercial superalloy, is used to fabricate aircraft engine turbine blades. We used anomalous ultrasmall-angle x-ray scattering (A-USAXS) to study the γ' populations in a series of Waspaloy samples as a function of long-term thermal exposure up to 12,623 hours. The use of A-USAXS was necessary to isolate the contributions to the scattering from the γ' population from those of the other precipitates, such as grain-boundary carbides.

Methods and Materials

In previous work [3, 4], Waspaloy samples exposed to a temperature of 1200°F (649°C) for times ranging from 0 to 12,623 hours had been characterized by using impedance spectroscopy, microhardness measurements, x-ray diffraction, and quantitative stereology. Table 1 shows the nominal alloy composition of Waspaloy. The scanning electronic microscopy (SEM) replica photomicrographs shown in Fig. 1 depict the microstructure in the baseline samples and the samples that were aged for the longest time.

Samples were prepared for the A-USAXS measurements by slicing thin sections from the aged material and then thinning those sections further by mechanical polishing as much as possible. The optimum thickness for the A-USAXS measurements near the Ni K edge is ~8 μm. The thickness of the samples measured by x-ray transmission was between 20 and 60 μm.

TABLE 1. Nominal composition of Waspaloy (wt.%) [2].

Ni	Cr	Co	Mo	Ti	Al	Fe	Zr	C	B
Balance	19.5	13.0	4.3	3.0	1.40	1.0	0.07	0.05	0.01

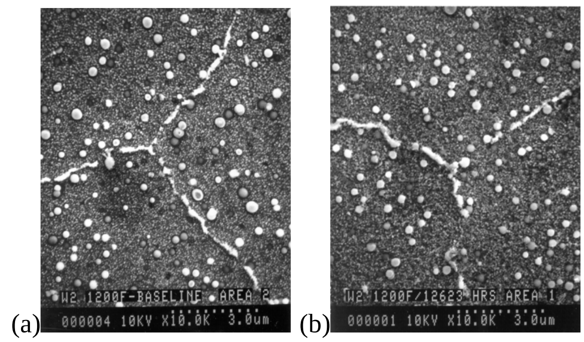

FIG. 1. SEM replica photomicrographs of precipitates in Waspaloy. (a) Baseline sample. (b) Sample aged for 12,623 hours at 1200°F (649°C). Each image width is ~8 μm.

The scattering measurements were performed at UNI-CAT beamline 33-ID at the APS [5]. In A-USAXS, a series of measurements is made at each of several energies below the absorption edge of the element of interest. X-rays at energies near the Ni K edge (8.333 keV) were chosen to enhance the contrast of scattering from the γ' precipitates. The calculated scattering contrast of Ni_3Al γ' precipitates in Waspaloy is shown in Fig. 2. Each separate USAXS measurement, at the energies indicated in Fig. 2, spanned the Q range from 0.0001 to 0.1 $Å^{-1}$ with several hundred data points, where $Q = (4\pi/\lambda)\sin\theta$, λ is the incident wavelength, and 2θ is the scattering angle. The x-ray beam size was 0.6 × 2.0 mm for most samples. For a few smaller samples, it was necessary to reduce the beam size to 0.6 × 0.7 mm. X-ray radiography was used, and care was taken to avoid examining areas with cracks or pinholes.

Results

Figure 3 shows the A-USAXS measurements for a sample aged 50 hours at 1200ºF. The variation of

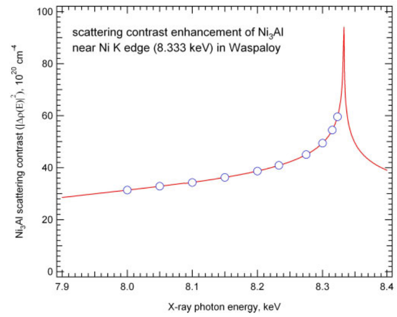

FIG. 2. Scattering contrast density $|\Delta\rho(E)|^2$ calculated for Ni_3Al γ' precipitates in Waspaloy. Energies for each USAXS experiment are indicated by circles. The variation with energy is due to the anomalous dispersion of nickel.

intensity with energy at a constant Q between 0.001 and 0.01 is consistent with the change in the scattering contrast of the Ni_3Al γ' precipitates as shown in Fig. 2. This consistent variation of scattered intensity with x-ray energy was observed in all the other samples. The three curves show features consistent with three different population distributions: primary γ' precipitates, secondary γ' precipitates, and grain-boundary carbides.

Analysis of the A-USAXS data collected so far is in progress. We will characterize the size, shape, and distribution of all phases present in the scattering data.

We will continue to measure A-USAXS from aged Waspaloy samples for different aging times and temperatures and will also investigate other superalloys. We will then correlate our A-USAXS determinations of the size, shape, and distribution of all phases present with complementary electrical characterization measurements and microscopy. All of these techniques together have the potential to provide statistically significant results that could be used to develop structure-property correlations that would facilitate a better understanding of the kinetics of degradation in this important class of structural materials. The experimental results will be complemented by modeling the electrical properties by using effective medium, percolation, and finite element models.

Discussion

We are encouraged by our initial measurements of A-USAXS from aged Waspaloy samples at x-ray photon energies near the Ni K edge. The samples investigated so far all show the intensity variation expected for scattering contrast variation due to Ni_3Al γ' precipitates. This variation will be instrumental in separating the scattering

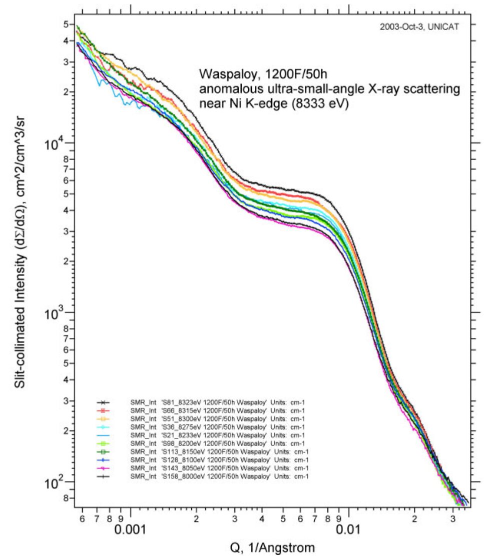

FIG. 3. A-USAXS data near the Ni K edge for a Waspaloy sample aged 50 hours at 1200ºF.

of the Ni_3Al γ' precipitates from the other precipitation in the alloy.

Acknowledgments

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