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Quantification of the coarsening kinetics of γ' precipitates in Waspaloy microstructures with different prior homogenizing treatments

V. Siva Kumar G. Kelekanjeri [a], Lewis K. Moss [a], Rosario A. Gerhardt [a,*], Jan Ilavsky [b]

[a] School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA 30332, USA
[b] X-ray Operations Division, Argonne National Laboratory, Argonne, IL 60439, USA

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Abstract

We report on quantification of the γ' precipitate population, and its coarsening behavior, in controlled Waspaloy microstructures synthesized to possess γ matrix grain sizes ranging from 13 to 89 μm. The grain microstructures were produced by initial solution-treatments at 1045, 1090 and 1145 °C. The γ' precipitates were obtained by aging at 779 and 796 °C for times ranging from 0.1 to 263.5 h. Specimen characterization was conducted via optical microscopy and scanning electron microscopy, DC four-point probe resistivity and ex situ ultra-small-angle X-ray scattering (USAXS) experiments at each aging time. The γ' size distribution, obtained from the USAXS analysis, transformed from an initial unimodal to an eventual bimodal distribution with continued aging. The overall coarsening kinetics, although non-steady state, followed $t^{1/3}$ behavior, when the primary γ' radius was used as the quantifying precipitate dimension. The coarsening rate constants were primarily determined by the aging temperature used, while the influence of prior homogenizing treatments was minimal to non-existent. A generic correlation was found to exist between a newly proposed figure-of-merit of scattering, η based on the USAXS-derived γ' precipitate distribution(s) and the measured electrical resistivity.

Keywords: Nickel-base superalloy; Precipitate coarsening; Microstructure; Four-point probe resistivity; Ultra-small-angle X-ray scattering

1. Introduction

Nickel-base superalloys are an important class of metallic alloys that find their use in land- and air-based turbine engine applications and are of great technological importance. The key aspect of their microstructure that facilitates their exceptional strength retention properties at elevated temperatures is the presence of a finely dispersed γ' precipitate phase, which is coherent with the matrix (γ) phase [1]. However, the high interfacial energy associated with the nanometer-sized γ' precipitates provides enough driving force for coarsening when exposed to high temperatures during service [2]. Precipitate coarsening in superalloys is known to proceed via volume-controlled diffusion of solute elements such as Al and Ti in the Ni-rich matrix (γ) phase. There exist several reports in the literature on both precipitation studies in nickel-base superalloys [3–6] and those that pertain to microstructural changes that result upon prolonged thermal exposure or aging [3,7–9]. In spite of these advances, a lifetime prediction model based on γ' microstructural evolution has not yet been developed. In order to develop a quantitative non-destructive method that could be used for lifetime prediction, it will be necessary to first break up this problem into many steps due to the complexity of the problem. This study is the first in a series of investigations designed to develop quantitative correlations between the γ' precipitate population(s), as determined from microscopy, small-angle scattering measurements, and electrical resistivity measurements. A brief summary of the relevant literature is given below.

Non-destructive evaluation (NDE) techniques present a fast and efficient means of specimen analysis, provided a connection can be made between the microstructural state

* Corresponding author. Tel.: +1 404 894 6886; fax: +1 404 894 9140.
E-mail address: rosario.gerhardt@mse.gatech.edu (R.A. Gerhardt).

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of the alloy and the measured parameters. Scattering-based techniques such as small-angle X-ray or neutron scattering (SAXS or SANS) and electrical resistivity measurement are specific examples of NDE techniques that have been used for studying superalloy systems [4–6,10,11]. Del Genovese et al. [10] reported on quantitative microstructural characterization of a modified Ni–Fe superalloy, DT 706, via ex situ small-angle neutron scattering (SANS) experiments. SANS spectra acquired from differently heat treated specimens were analyzed to fingerprint the different maxima in the spectra to the corresponding microstructural entities (γ', γ'') with the aid of supplementary electron microscopy studies. The SANS spectra were further fitted by making basic assumptions about the precipitate shape and the distribution type to obtain quantitative microstructural indicators such as the size distribution, interparticle distance and the volume fraction of precipitates. Ratel et al. [5] reported in situ SANS investigations of morphological evolution of γ' precipitates as a function of time and temperature in single-crystal SC16 superalloy. SANS spectra collected at different stages below the solvus temperature were modeled by assuming both spherical and cuboidal precipitate populations and subsequent inferences were drawn regarding the size and shape transition of precipitates [5].

Besides SANS, microstructural evolution in precipitation-hardened alloys has also been studied quantitatively using the SAXS technique. Ping et al. [12] reported on quantitative microstructural evolution studies in 13Cr–8Ni–2.5Mo–2Al precipitation-hardened stainless steel using a combination of TEM, SAXS and three-dimensional atom probe technique. SAXS was used for the determination of the average radius, number density and volume fraction of β-NiAl precipitates in the temperature range from 450 to 620 °C. The authors reported that at temperatures between 510 and 550 °C, determination of precipitate radius through SAXS was difficult because of the overlap in the scattering profiles from β-NiAl precipitates and carbides. The SAXS technique has also been used extensively for conducting quantitative precipitation studies in Al–Zn–Mg–Cu [13] and Al–Mg–Si [14] alloys and also for investigating the effect of welding in precipitation-hardened alloys [15].

The resistivity technique is based on the principle that the measured electrical resistivity is influenced by the presence of defects, dissolved impurities or a secondary phase present as a dispersion, all of which can affect conduction electron scattering. White et al. [4] studied the kinetics of γ' nucleation and growth mechanisms in Nimonic PE 16 superalloy via four-point resistivity measurements. The precipitate growth was found to follow a $t^{1/2}$ behavior instead of the usual $t^{1/3}$ law, which they attributed to the effect of quenched-in excess vacancies from the solution-treatment. Kelekanjeri and Gerhardt [6] investigated the resistivity behavior in Waspaloy subjected to aging at 600, 725, 800 and 875 °C. The observed resistivity variations were discussed in terms of nucleation-growth, a first coarsening stage and a second coarsening stage. While the effect of γ' distribution was found to dominate the first two stages, the resistivity, during the second coarsening stage, is speculated to have been influenced primarily by the solid-solution impurities. Whelchel et al. [16] investigated the coarsening kinetics of specimens from the above aging sets using conductivity and hardness measurements. An interesting finding of this work was that for a given aging set, the initial conductivity minimum and the hardness maximum occurred at widely different aging times and therefore corresponded to distinctly different values of critical γ' radius. This is probably due to differences in the physical mechanisms that dictate the final measured response in both conductivity and hardness measurements.

The present paper describes the synthesis of controlled Waspaloy microstructures of varying matrix (γ) grain size and γ' precipitate size distributions with the objective of developing quantitative correlations between the precipitate populations present and the measured resistivity. This study focuses on the coarsening aspect of microstructural evolution upon aging at 779 and 796 °C for times ranging from 0.1 to 263.5 h. Specimen characterization was conducted via a combination of ultra-small-angle X-ray scattering (USAXS) and DC four-point probe electrical resistivity measurements. Additionally, the specimens were also characterized via scanning electron microscopy to enable specific correlations with the scattering and resistivity experiments. The results of these investigations are subsequently discussed to gain insights into the kinetics associated with the microstructural evolution induced upon progressive aging. Finally, this paper provides the first link between the measured resistivity and the average precipitate size distributions in Waspaloy, as determined from analysis of USAXS spectra.

2. Experimental procedure

2.1. Heat treatments

Heat treatments were conducted with the objective of varying the microstructural parameters systematically, viz. the γ matrix grain size and the γ' precipitate size distribution. Industrial grade Waspaloy obtained from Fry Steel was the starting material for the heat treatment experiments. The elemental composition of the alloy is given in Table 1. Heat treatments were conducted in a Carbolite model CTF 12/65/500 horizontal tube furnace under flowing argon. A type N thermocouple was used for external temperature monitoring, which was approximately positioned at the furnace center and below the specimen tray. All of the heat treatments conducted in this investigation consisted of three steps: a ramp-up at 5.5 °C min^{-1}, a dwell and a rapid quench at the conclusion of the dwell duration. The quench was conducted by manually removing the specimen bar from the furnace and immersing it in a 5 wt.% brine solution at ~50 °C. After slicing the specimens, the previously heat treated bars were reinserted into the fur-