Available online at www.sciencedirect.com
ScienceDirect
Acta Materialia 56 (2008) 4715–4723
Acta MATERIALIA
www.elsevier.com/locate/actamat

Mapping single-crystal dendritic microstructure and defects in nickel-base superalloys with synchrotron radiation

Naji S. Husseini [a,*], Divine P. Kumah [a], Jian Z. Yi [b], Christopher J. Torbet [b], Dohn A. Arms [c], Eric M. Dufresne [c], Tresa M. Pollock [b], J. Wayne Jones [b], Roy Clarke [a]

[a] *Applied Physics Program, University of Michigan, Ann Arbor, MI 48109, USA*
[b] *Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI 48109, USA*
[c] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

Received 15 January 2008; received in revised form 3 April 2008; accepted 20 May 2008
Available online 9 July 2008

Abstract

Solidification of single-crystal nickel-base superalloys introduces large-scale segregation of constituent elements and defects such as dislocations and mosaicity. By exploiting the energy tunability and interference capabilities of high-brilliance X-ray radiation, key structural features of the dendritic single crystals were mapped over large areas. Interference and diffraction of synchrotron X-rays revealed significant misorientations between individual dendrites in the as-solidified state. For the first time this mosaic structure was quantified for an array of dendrites and correlated with the density of "grown-in" dislocations whose density ranged from 10^7 to 10^8 cm^{-2}. Absorption contrast permitted simultaneous mapping of the distribution of refractory metal additives (e.g. rhenium and tungsten), which segregated preferentially to the dendrite cores with a linear composition gradient toward the interdendritic regions. The results demonstrate that synchrotron X-ray imaging is promising for in situ studies of single-crystal structure and defects in nickel-base superalloys.

Keywords: Superalloy; X-ray radiography; Microstructure; Dislocations; Lattice defects

1. Introduction

Operating conditions in air- and land-based turbine engines impose extremely demanding conditions on the engineering materials in critical components such as blades and vanes. These parts, which are typically composed of single-crystal nickel-base superalloys, operate under high stresses and near their melting point. Ni-base superalloys are typically composed of 50–60% Ni with small amounts of carbide-forming elements and other refractory metals [1]. These superalloys exhibit exceptional mechanical strength, toughness, and resistance to corrosion and oxidation at high temperatures, making them ideally suited for critical applications in severe environments.

A key factor in the extreme durability of Ni-base superalloys is the presence of a high volume fraction (>50%) of a cuboidal, intermetallic γ' phase (i.e., Ni_3Al), which is highly resistant to creep and fatigue crack propagation [1–3]. The submicron-scale γ' phase is coherent with the γ-Ni matrix (Fig. 1a). At a much larger length scale, the microstructure is characterized by $\langle 0\,0\,1\rangle$-oriented dendrites that grow parallel to the solidification direction [4,5] with core spacings on the order of 200–600 μm (Fig. 1b).

High-temperature mechanical properties are further enhanced by moderate concentrations (5–15%) of refractory metals (e.g., Mo, Re, Ta, W), but these additions can create problems during materials processing [6,7]. The thermodynamically driven segregation of the heavy elements can cause convective instabilities and subsequent breakdown of the single-crystal solidification front [5,7–10]. The residual segregation is difficult to eliminate with annealing and may degrade mechanical properties; thus,

* Corresponding author. Tel.: +1 734 764 7446; fax: +1 734 764 2193.
E-mail address: najihuss@umich.edu (N.S. Husseini).

1359-6454/$34.00 © 2008 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.actamat.2008.05.041

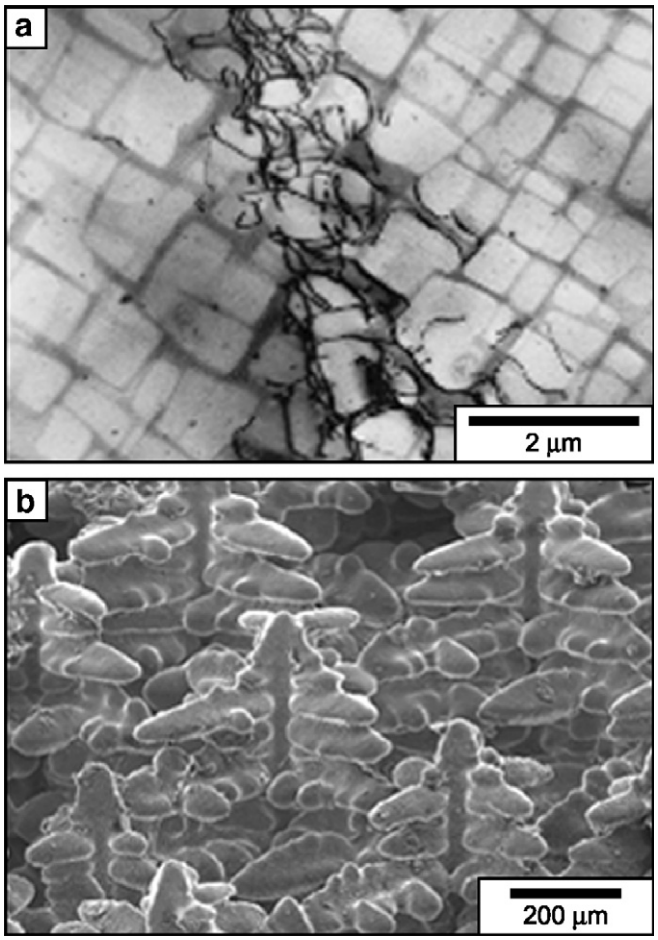

Fig. 1. (a) TEM image of the γ/γ' phases of two dendrites separated by a dislocation-rich interdendritic region; (b) SEM image of the dendritic microstructure growing upward along $\langle 001 \rangle$ with the interdendritic material removed while still liquid (image courtesy of McLean Echlin).

a detailed understanding of the distribution of heavy-element additives within the dendritic structure is crucial. The addition of carbon, appearing in solution or as metal carbides [1,11], is beneficial for single-crystal growth. Carbon also reduces the sensitivity of the structural properties to low-angle boundaries that may develop during solidification of the complex geometries characteristic of turbine blades [11].

In multicomponent superalloys, solidification occurs over a wide temperature range. The spacings between the dendrites depend on the cooling rate at the solid-liquid interface during withdrawal in the Bridgman process. Due to slight misalignments between neighboring dendrites and thermal stresses developed during solidification, the interdendritic regions with the last liquid to freeze typically contain a complex network of dislocations [3]. While these dislocations have been observed on a local scale by transmission electron microscopy (TEM) (Fig. 1a), the mosaic character of the dendritic microstructure has not been studied in detail, even though dislocation networks are known to influence primary creep behavior [3].

Due to the complexity of superalloys and their accompanying processing paths, knowledge of the microstructure across the relevant length scales is often lacking. Large-scale, three-dimensional characterization of microstructure is particularly challenging, and the suite of characterization tools is incomplete. Synchrotron X-ray radiography is a versatile tool that complements traditional characterization techniques: it can probe relatively large volumes to gain three-dimensional compositional and structural information. Synchrotron X-rays have already proved invaluable for studies of growth [12–15], strain [16,17], surface defects [17,18] and nanoscale precipitation [19], though predominately via diffraction and not typically in a transmission geometry. Here, we use the special characteristics of undulator-based synchrotron radiation, particularly high brilliance and energy tunability [20], to perform a quantitative mapping of single-crystal Ni-base superalloy microstructure, including the distribution of refractory elements and mosaic structure within the dendritic array.

2. Experimental design

2.1. Superalloy preparation

The René N5 superalloys had a nominal composition given in Table 1. Cylindrical bars were directionally solidified along the $\langle 001 \rangle$ crystallographic orientation by withdrawing a ceramic investment mold downward through a radiation baffle in a Bridgman-type high-temperature furnace. Thermal gradients and withdrawal rates were approximately 40 °C cm^{-1} and 20 cm h^{-1}, respectively. Small samples parallel and perpendicular to the dendrite-growth direction were machined into coupons with dimensions of approximately 2 mm by 2 mm via wire electrical discharge machining, and then polished to a variety of thicknesses between 100 and 300 μm.

2.2. Electron probe microanalysis calibration

The segregation characteristics of the constituent elements were analyzed with a Cameca CAMEBAX or a Cameca SX-100 Electron Probe Microanalyzer (EPMA) equipped with four wavelength-dispersive spectrometers (WDS). The acceleration voltage and beam current for these analyses were 20 kV and 100 nA, respectively. The WDS system was equipped with eight diffraction crystals of thallium acid phthalate (TAP), used for Al and Ta; lithium fluoride (LiF), used for Re and W, and pentaerythritol (PET), used for Co, Cr, Mo and Ni. X-ray counts were measured simultaneously for K_α (Al, Co, Cr), K_β (Ni), L_α (Re, Mo) and M_α (Ta, W) radiation. These counts were converted to concentration values using standard correction procedures calibrated from pure-element standards, except for Al, for which an 70Ni–30Al (wt.%) standard was used.