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Particle-associated misorientation distribution in a nickel-base superalloy

C.G. Roberts,[a,*] S.L. Semiatin[b] and A.D. Rollett[a]

[a] *Department of Materials Science and Engineering, Carnegie Mellon University, 5000 Forbes Avenue, Pittsburgh, PA 15213, USA*
[b] *Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson Air Force Base, OH 45433-7817, USA*

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An approach was developed to quantify the relationship between the location of carbide particles and grain boundaries during the annealing of a typical nickel-base superalloy, Waspaloy. The average grain size increased during supersolvus heat treatment at 1100 °C for 1 h despite an absence of carbide coarsening and changes in the global texture. It was determined that the spatial distribution of carbides which developed during annealing was correlated to the grain-boundary misorientation distribution.

Keywords: Nickel; Misorientation; Texture; Grain growth; Zener pinning

Inert particles, such as borides and carbides, influence the microstructural stability and thus mechanical properties of metallic alloys. Generally, the addition of a small volume fraction of precipitates results in grain-size refinement [1]. In early seminal work by Zener, for instance, the effect of particle size and volume fraction on the maximum (stable) matrix grain size was quantified [2]. The driving force for grain growth is the reduction of the total grain-boundary area [3]; in multiphase materials, the particle–grain boundary interaction leads to a retardation in grain growth. When the driving force for growth and the retarding force of particles achieve a balance, a maximum (stable) grain size is achieved, as predicted by Zener. Relatively little attention has been paid, however, to the effect of variable or anisotropic properties, such as grain-boundary energy, on the stable grain size.

Numerous alloys [4–8] utilize the pinning phenomenon introduced by dispersed and precipitated second phases to control grain size and thus obtain desired mechanical properties for a given application. In particular, nickel-base superalloys contain various alloying elements which are responsible for phases such as γ, γ', carbides and borides. A number of such materials contain large volume fractions of fine γ' precipitates which essentially prevent gamma grain growth below the γ' solvus. Specific types of carbides include MC, $M_{23}C_6$ and M_7C_3 [9]. The primary equilibrium phases MC and $M_{23}C_6$ have a tendency to form on grain boundaries [10], presumably because of heterogeneous nucleation. Temperatures above the γ' solvus must be used for substantial grain growth; the upper limit on grain size is thus controlled by the inert carbide particles.

The objective of the present work was to characterize and interpret microstructure evolution during supersolvus annealing of a typical γ–γ' superalloy, Waspaloy. Specifically, the correlation between carbide location and grain-boundary disorientation distribution was established. The concept of the particle-associated misorientation-distribution function (PMDF) was introduced to quantify such correlations.

The nickel-base superalloy Waspaloy was used in the experiments. The initial ingot-metallurgy material had been vacuum-induction melting (VIM) and vacuum-arc remelted (VAR). Following casting, the ingot was homogenized and subjected to a series of supersolvus and subsolvus hot working operations to produce a wrought (recrystallized) microstructure. It was received as 106-mm-diameter billet with the measured composition shown in Table 1.

Slices approximately 12 mm thick were cut from the billet using a band saw. Subsequently, small samples measuring 10 mm × 10 mm were removed via electric-discharge machining from the central region of each disk at a location not exceeding 5/8 of the radius. The

* Corresponding author. Tel.: +1 412 258 8692; e-mail: croberts@andrew.cmu.edu

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Table 1. Typical composition of Waspaloy

Element (wt.%)	Al	B	C	Co	Cr	Fe	Mo	Ni	Ti	Ta	W	V	Zr
Measured	1.34	0.007	0.036	13.45	19.75	0.79	4.25	Bal.	2.98	0.01	0.04	0.03	0.05
Nominal	1.40	0.01	0.05	13.00	19.50	1.00	4.30	Bal.	3.00	n/a	n/a	n/a	0.07

microstructure and PMDF were determined for both an as-received sample and a sample given a heat treatment for 1 h at 1100°C, a temperature approximately 65 °C above the γ' solvus [11]. Each specimen was ground and polished using standard methods. The final polishing step used an attack polish with a 10:1 diluted Kallings etchant and a suspension of 0.05 μm Al_2O_3. This method provided a surface with lightly etched grain boundaries and intact carbides.

Large-area particle maps of the microstructure were constructed for both samples using a Philips XL-30 FEGSEM® scanning electron microscope (SEM) in the backscattered electron imaging mode. Such images provided optimal contrast between the carbides and the γ matrix so that the average particle size, volume fractions and spatial distributions could be readily extracted. To provide statistically meaningful data, a montage measuring 1 mm × 2 mm containing more than 800 particles was developed. Likewise, large area grain maps were generated using a light optical microscope in combination with chemical etching to darken the grain boundaries. Because of extensive grain growth during 1 h at 1100 °C, the average grain size of both samples were determined using the ASTM linear-intercept method on the large-area grain maps [12]. The twin boundaries were ignored in the grain-size calculations.

Orientation imaging was conducted using a Philips XL-40 FEGSEM® with TSL® electron backscatter diffraction (EBSD) OIM™ software. Scan areas measured approximately 390 μm × 570 μm for both samples. A relatively small step size (2 μm) was used to provide reasonable resolution for the initial microstructure and the annealed structure. Approximately 25 scans were obtained for both the as-received material and the annealed sample to obtain a statistically significant number of carbides on grain boundaries.

A novel method was developed to establish the relationship between carbide location and grain-boundary misorientation. Specifically, an algorithm was formulated to generate a pixel-to-pixel correlation between the SEM secondary electron image and the inverse-pole-figure map. A second routine searched and replaced the x- and y-coordinates with carbide information. Each particle was then determined to be either within the grain interior or on a boundary. If the carbide appeared on a boundary, the minimum grain misorientation (i.e. the disorientation) was determined and the respective bins were updated. A 5° bin width was used. A total of 250 (as-received) carbides and 325 (1100 °C/1 h) carbides contributed to the analyses. Figure 1 provides an example of registration between an SEM image and an orientation imaging microscopy inverse-pole-figure image. From such information, PMDFs were determined. In the present context, the term PMDF connotes the number fraction of carbides located on grain boundaries with a specific range of misorientation angles.

The grain structure of the as-received sample was found to be equiaxed with an average grain diameter of 38 ± 2.5 μm. The 1100°C/1 h sample also had an equiaxed grain structure; its average grain diameter was 200 ± 30 μm (Fig. 2). Applying Zener's analysis, the predicted pinned grain size was calculated to be (4/3) (1.2 μm/0.002) = 800 μm. Based on this calculation, the annealed sample (1100 °C/1 h) was thus at an intermediate stage, and additional grain growth would be expected for longer annealing times.

The absence of a spatial correlation of the carbides was confirmed using a two-point pair-correlation function (Table 2). In contrast to the random spatial distribution, the relationship between carbide location with respect to the internal grain boundaries was investigated. The expected value was based on a random spatial distribution of single-sized, spherical carbides. With these assumptions, the expected number fraction on boundaries (N_{gb}/N_{total}) was significantly lower than the observed fraction for both the as-received and the 1100°C/1 h samples (Table 2). Furthermore, the expected and observed number fractions both decreased

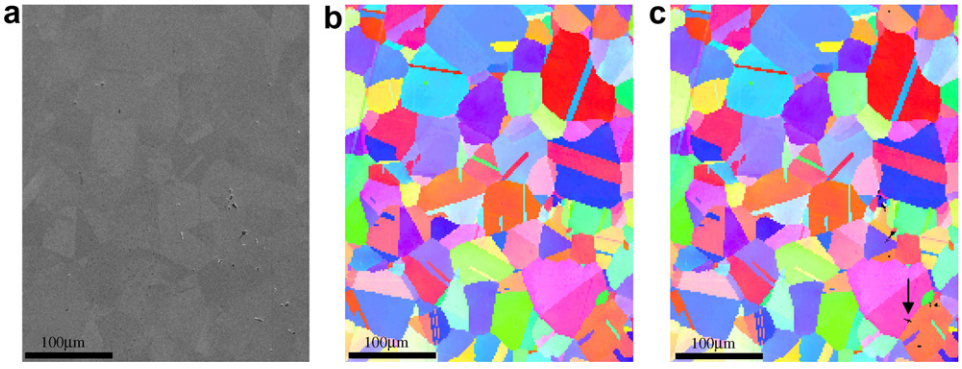

Figure 1. (a) High-angle secondary electron image with intact carbides. (b) Inverse-pole-figure image of same area as that shown in part (a). (c) Combined particle and grain inverse-pole-figure map (arrow indicates a particle on the grain boundary).