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The effect of cast microstructure and crystallography on rafting, dislocation plasticity and creep anisotropy of single crystal Ni-base superalloys

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ABSTRACT

In the present work we investigate three mechanical and microstructural aspects of high temperature and low stress creep of the single crystal superalloy LEK 94. First, we compare the tensile creep behavior of specimens loaded in precise [001] and [110] directions and show that tensile creep specimens with precise [110] directions show significantly lower minimum creep rates. However, small deviations from precise [110] orientations result in a significant increase of creep rate. Second, we use a novel SEM technique to measure dislocation densities. We show that after short periods of creep, dislocation densities in dendritic regions are always higher than in interdendritic regions. This finding is probably associated with wider γ-channels, higher concentrations of W and Re and higher misfit stresses in the γ-channels of dendrites. Finally, we show that internal stresses associated with solidification can drive complex rafting processes during high temperature exposure, which differ between dendrite cores and interdendritic regions.

1. Introduction

Technical Ni-base superalloy single crystals (SXs) are used to fabricate turbine blades for aero engines and gas turbines for energy production e.g. [1–6]. SXs are produced using vacuum induction melting in combination with a Bridgman type of solidification process [e.g. 2,4,6]. During solidification, the [001] direction is the natural growth direction of dendrites. However, scatter in process parameters and the geometric complexity of turbine blades often result in deviations between the longitudinal axis of a blade and the natural [001] solidification direction. In SX technology one must accept a certain crystallographic deviation range from the [001] direction in order to keep processing costs at a reasonable level. Deviations between 12 and 15° from [001] [4,6] are considered as acceptable. However, there is only limited information available on how much such deviations affect the creep behavior of SXs, most of the published experimental results stem from the low temperature high stress creep regime. For a mechanistic interpretation of creep, this knowledge is required. One objective of the present work is to provide this type of information for tensile creep specimens oriented precisely in [110] directions and for specimens with tensile axis which deviate from [110].

Second, we are interested in differences in dislocation densities between interdendritic regions (ID) and dendrite cores (D). Chemical and microstructural differences between these two regions result in a large scale heterogeneity which is an inherent feature of SX microstructures. The spatial arrangement of dendrites is schematically illustrated in Fig. 1a [7,8]. Fig. 1b shows a micrograph which was taken perpendicular to the [001] solidification direction (see Fig. 1a) using secondary electron (SE) contrast in the scanning electron microscope (SEM). A typical average spacing between the centers of individual dendrites is of the order 400 μm [7,8]. It is well known that heavy elements like Re and W have a tendency to segregate to the dendrites while there is a higher concentration of light elements like Al and Ti in the interdendritic regions [7,9–12].

Multiple step heat treatments are required to homogenize SX alloys with respect to the chemical differences between D and ID regions after solidification, e.g. [7]. During the precipitation heat treatment of SXs, a fine γ/γ′-microstructure forms where γ′-cubes (typical cube edge length: 0.5 μm, typical volume fraction: 70%, $L1_2$ crystal structure, chemical composition: $Ni_3Al(Ti,Ta)$ are coherently precipitated in a γ-matrix, e.g. [4–6].

The face centered cubic (fcc) γ-channels, which separate the γ′-cubes, have a typical width of 0.1 μm. Their volume fraction in the γ/γ′-microstructure is close to 30%, e.g. [4–6]. Fig. 2 shows a

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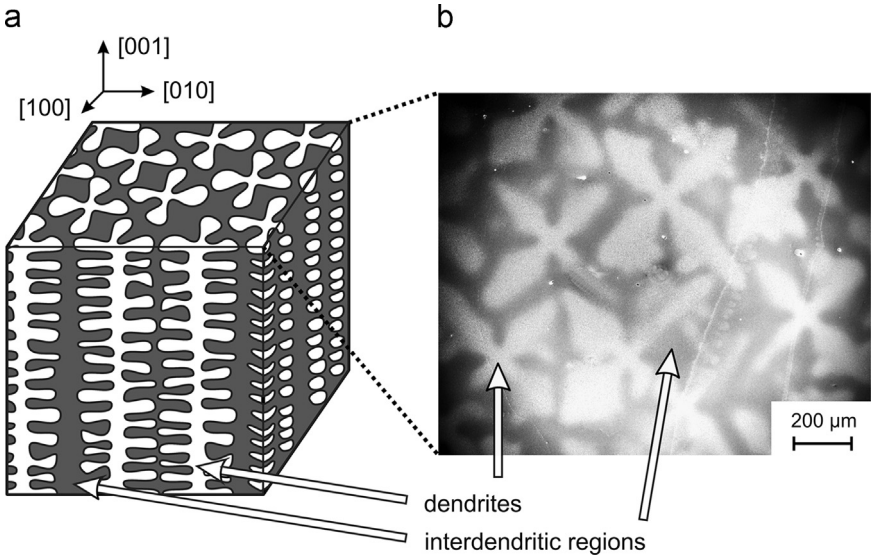

Fig. 1. Cast microstructure. Dendrites and interdendritic regions are highlighted by arrows. (a) Schematic illustration of dendrites parallel to the [001] solidification direction. (b) Secondary electron scanning electron micrograph taken perpendicular to the solidification direction (LEK 94, for details see [8]).

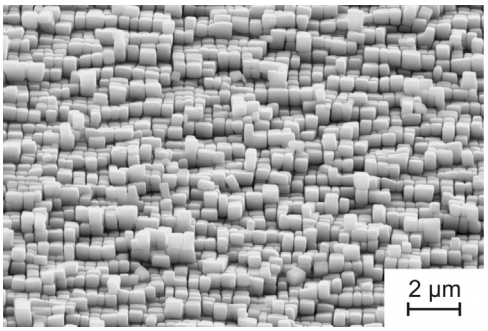

Fig. 2. Secondary electron SEM micrograph of free standing γ′-particles obtained after an electrochemical treatment which selectively etches out the γ-phase.

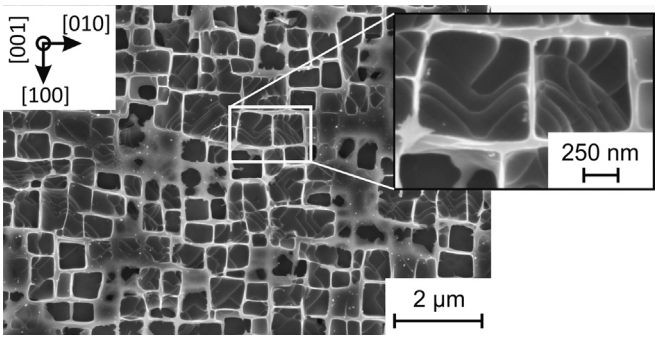

Fig. 3. Interfacial dislocation traces at γ/γ′-interfaces after etching out of the γ′-phase.

large area of free standing γ′-particles which was obtained after selective electrochemical etching of the γ-phase.

In SXs there is often a negative misfit between the γ- and the γ′-phases. A negative misfit refers to a microstructure where the $L1_2$ γ′-phase has a smaller lattice constant than the fcc γ-matrix. In the absence of an external stress this misfit can result in local stresses as high as 500 MPa, e.g. [4,13,14]. In service, Ni-base superalloy single crystals must withstand mechanical loads at temperatures above 1000 °C, where their microstructure is not stable. Under these conditions of high temperature and low stress creep, rafting, the directional coarsening of the γ′-phase occurs [e.g. 15–20]. During high temperature exposure and in the early stages of creep, dislocation networks form around the γ′-cubes in the γ-channels close to the γ/γ′-interfaces, e.g. [19–23]. Later in creep, cutting processes occur where γ-network dislocations enter the γ′-phase, e.g. [20,24,25].

The density of the dislocations in the dislocation networks is an important parameter for a physically sound interpretation of creep based on the evolution of dislocation substructures during high temperature plasticity. Traditionally, dislocation densities were determined using transmission electron microscopy (TEM), e.g. [24,26]. Recently, Epishin and Link [27] have shown that conventional secondary electron scanning electron microscopy (SEM) can be used to study dislocation configurations at γ/γ′-interfaces. Agudo et al. [19] have explained this finding (see Fig. 3 of [19]). It requires that γ′-particles are etched out during the metallographic preparation of the specimen. As a result, after etching only γ-phase regions remain. The heterogeneous γ-phase surface consists of bright high standing γ-ligaments representing γ-channels between etched out γ′-particles. These ligaments are parallel to the electron beam. Darker regions between the ligaments represent γ-phase from channels below the etched out γ′-particles (perpendicular to the electron beam). Agudo et al. [19] have shown that traces of dislocations are visible in these darker regions. Assuming that each γ/γ′ interface dislocation is associated with a trace which can be detected in the SEM, dislocation densities can be evaluated using Ham's method [28]. Agudo et al. [19] have shown that this type of SEM based dislocation density measurements yield similar results as conventional TEM measurements. The Epishin/Link-SEM method, however, has two important advantages as compared to traditional TEM assessments. First, in the SEM, it is easy to differentiate between dendritic and interdendritic regions and second, larger specimen regions can be screened for dislocations. An example is given in the SE SEM micrograph of Fig. 3, where dislocation traces can be seen in a large region $> 70\ \mu m^2$. The area highlighted with a white rectangle is shown at a higher magnification to document the details of dislocation traces. Agudo et al. [19] presented graphs where dislocation densities were plotted as a function of time and as a function of accumulated creep strain. These graphs compiled dislocation densities measured by TEM and by SEM, Fig. 4. The wide error bars in Fig. 4 indicate that dislocation densities generally show large scatter. This particularly holds for TEM data,