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Creep of a nickel-based single-crystal superalloy during very high-temperature jumps followed by synchrotron X-ray diffraction

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Abstract—Complex thermomechanical loadings at high temperature ($T \geqslant 950$ °C) on the AM1 single-crystal superalloy were studied by X-ray diffraction under synchrotron radiation. This technique enables in situ access to the evolutions of the lattice mismatch as well as the volume fraction and the dislocation density of the γ and γ′ phases during high-temperature non-isothermal creep loadings. It is shown that the large microstructure evolutions through the growth/shrinkage of γ/γ′ interfaces due to the γ′ volume fraction changes and the associated variations in the density of dislocations at these interfaces during temperature jumps are key parameters controlling the whole behavior (distribution of internal stresses and constitutive laws) of both phases. We propose an empirical constitutive law linking the strain rate of the γ′ phase to its stress state as well as a way to determine it. During our experiments, the plastic behavior of the γ′-rafts is related to the entry of dislocations with Burgers vectors perpendicular to the tensile axis and controlled by the overcoming of a threshold stress corresponding to the internal stress components perpendicular to the tensile axis.

Keywords: Nickel alloy; Synchrotron radiation; X-ray diffraction (XRD); Non-isothermal test; Creep deformation

1. Introduction

Nickel-based single-crystal superalloys are widely used for the manufacturing of turbine blades and vanes of the most advanced aeroengines and industrial gas turbines where high-temperature creep resistance is required for a wide range of temperature/stress regimes [1,2]. The high creep resistance of these alloys is induced by the precipitation of a high volume fraction (close to 70%) of the long-range ordered $L1_2$ γ′ phase which appears as cubes coherently embedded in a face centered cubic (fcc) solid solution γ matrix [3]. This volume fraction is modified during thermal changes, which have tremendous effects on the creep life [4], and depends on the temperature, the hold time and the applied load [5–7].

Several studies reported in the literature have been conducted on the isothermal creep behavior under various temperatures and loading conditions [8–11] as well as for different crystal orientations [12–15]. Three temperature/stress creep regimes can be distinguished for single-crystal superalloys depending on the alloy chemistry [16–19]. Low temperature creep regimes (~750 °C) are characterized by stable microstructures with a possible growth of the γ′ precipitates by Ostwald ripening processes and a plastic deformation due to γ′ shearing for high applied stresses, and due to dislocation bypassing for lower applied stresses [20,21]. Intermediate temperature regimes (~850–1000 °C) are characterized by a continuous increasing creep rate and plastic deformation occurring by γ′ bypassing [22]. Moreover, a progressive directional coarsening of the γ′ phase (i.e., γ′ rafting) appears (see Fig. 1). At high temperatures (~1000–1150 °C), the directional coarsening is faster and the $a/2\langle 1\,1\,0\rangle$ dislocations glide in the γ matrix and percolate at the γ/γ′ interfaces to relax the γ/γ′ coherency stresses [23,24].

The orientation of the γ′-rafts, as well as the distribution of internal stresses within the initial non-deformed material, depends on the sign and amplitude of the lattice mismatch between both phases. This so-called natural mismatch is defined as:

$$\delta = 2\frac{a_{\gamma'} - a_{\gamma}}{a_{\gamma'} + a_{\gamma}} \tag{1}$$

where $a_{\gamma'}$ and a_{γ} are the free lattice parameters of the γ′ and the γ phase, respectively. In the γ′-rafted material, the

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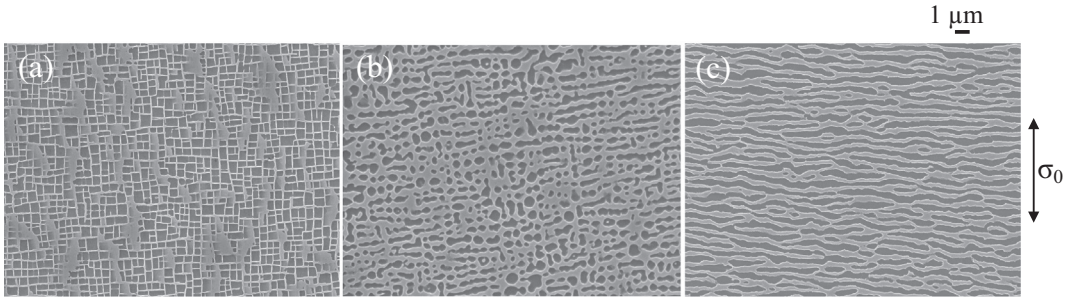

Fig. 1. γ′ rafting during a creep test of a⟨001⟩ oriented single-crystal superalloy at high temperature/low stress: γ/γ′ microstructure at the beginning of the experiment (a), during primary creep stage (b) and during secondary creep stage (c). Note that the γ′ phase appears in darkness.

internal stresses are related to the difference between the natural mismatch and the constrained mismatch in the interface plane defined as:

$$\delta_{\perp} = 2\frac{a_{\gamma'200} - a_{\gamma200}}{a_{\gamma'200} + a_{\gamma200}} \quad (2)$$

The γ/γ′ interface dislocation network, consisting of a/2⟨110⟩ and a⟨100⟩ dislocations [25], contributes in many ways to the mechanical behavior during isothermal creep tests. It first reduces the coherency stresses in the γ corridors perpendicular to the tensile axis [26–28] (for a negative natural mismatch) and promotes the directional coalescence of the γ′ precipitates [29–31], i.e. γ′ rafting. The internal stress level in the rafted microstructure, taken as a stack of alternate γ and γ′ layers, depends on how much the mismatch stresses are relaxed by the average stress field of interface dislocations: it is then related to the difference between δ and $\delta_{\perp}$. Second, the local stress field of these dislocations protects the γ′ precipitates from deformation by a⟨100⟩ superdislocations [22,32,33]. Such superdislocations are believed to be responsible for the acceleration of the creep deformation by relaxing coherency stresses and osmotic forces as they allow the removal of a/2⟨110⟩ dislocations present at the γ/γ′ interfaces [28,34–39]. To a lesser extent, γ/γ′ interface dislocations play also a role in the local chemical composition of the γ phase during creep tests leading to accelerated microstructural evolutions [40,41], known to be damaging [42,43]. However, all these investigations were conducted for isothermal creep conditions and little is known on the role of the γ/γ′ interfacial dislocation network under non-isothermal conditions.

Recent studies have shown that the creep rate under thermal cycling conditions is higher than under isothermal ones and that the lifetime at high temperature is shorter during non-isothermal creep [44,45]. The same last observation was also done by Cormier et al. in the case of a single overheating performed after different creep durations under high-temperature/low-stress conditions [46]. Nevertheless, these authors have also noticed that a longer overheating leads to a longer creep life, i.e. the origins of the plastic deformation during non-isothermal creep tests are complex and cannot be easily forseen. Indeed, le Graverend et al. have also observed that a creep test with a single overheating can exhibit a greater lifetime than the one obtained for an isothermal creep test performed under the same temperature/stress condition [47]. To the best knowledge of the authors, the origins of creep deformation during thermal changes have never been studied before in single-crystal superalloys and represent a challenge to be faced to improve the existing models able to predict the high-temperature non-isothermal deformations.

The aim of the present work is then to get a deeper understanding of the creep-controlling parameters during temperature changes, i.e. the γ/γ′ interface dislocation network stability, the dislocation density in the γ and γ′ phases and the volume fraction of the strengthening γ′ phase, in order to explain the subsequent macroscopic strain rate and amplitude. To do so, non-isothermal experimental conditions ("positive" and "negative" thermal cycling) leading to the entry of dislocations in the γ′ phase are especially performed and followed by in situ X-ray diffraction (XRD) under synchrotron radiation avoiding then any uncertainties in post mortem measurements due to unloading (stress relaxation) or cooling (changes in the volume fraction of both phases) processes [48,49]. This technique allows a better understanding of the physical mechanisms of plastic strain in superalloys and more specifically how each phase is deformed during non-isothermal creep loading by giving access to the internal stresses. This leads then to determine the rate-limiting mechanism of the γ′-raft plasticity.

2. Experimental procedure

2.1. Materials

Experiments were performed using the first-generation AM1 Ni-based single-crystal superalloy. Its nominal chemical composition is given in Table 1. Experiments were performed using samples which had received the AM1 standard heat treatment (3 h/1300 °C/air quench (AQ) + 5 h/1100 °C/AQ + 16 h/870 °C/AQ), leading to a 0.45 μm average γ′ precipitate size and to a ~70% precipitate volume fraction (a typical as-received microstructure is shown in Fig. 1a). Five cylindrical specimens (3.5 mm in diameter and 29 mm of gauge length) were machined out from [001] rods. The samples' stress axis deviation from the perfect [001] crystallographic orientation was less than 3°. A careful gauge length surface polishing was performed to avoid any premature failure during thermomechanical creep tests. Three specimens (subsequently called experiments A, B and C) were crept at 1050 °C/150 MPa

Table 1. Chemical composition of AM1 superalloy (wt.%).

Cr	Co	Al	W	Ta	Ti	Mo	Ni
7.8	6.5	5.2	5.7	7.9	1.1	2.0	Bal.