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Double minimum creep of single crystal Ni-base superalloys



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ABSTRACT

Low temperature (750 °C) and high stress (800 MPa) creep curves of single crystal superalloy ERBO/1 tensile specimens loaded in the (001) direction show two creep rate minima. Strain rates decrease towards a first sharp local creep rate minimum at 0.1% strain (reached after 30 min). Then deformation rates increase and reach an intermediate maximum at 1% (reached after 1.5 h). Subsequently, strain rates decrease towards a global minimum at 5% (260 h), before tertiary creep (not considered in the present work) leads to final rupture. We combine high resolution miniature creep testing with diffraction contrast transmission electron microscopy and identify elementary processes which govern this double-minimum type of creep behavior. We provide new quantitative information on the evolution of microstructure during low temperature and high stress creep, focusing on γ -channel dislocation activity and stacking fault shear of the γ' -phase. We discuss our results in the light of previous work published in the literature and highlight areas in need of further work.

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1. Introduction

Single crystal Ni-base superalloys (SXs) are used for blades in advanced gas turbines for power plants and jet engines [1–5]. They operate in the creep range, where they have to withstand mechanical loads at temperatures above half of the melting point in Kelvin. During creep, strains gradually increase with time. Creep research focuses on explaining the shape of individual creep curves, which often show stages of decreasing, constant and increasing creep rates referred to as primary, secondary and tertiary creep [6–12]. However, under conditions of low temperature and high stress creep (LTHS-creep, here: 750 °C, 800 MPa), SXs can show creep curves with peculiar shapes [13–22]. SXs operate at temperatures up to 1100 °C. Therefore, in the present work, we refer to 750 °C as a low temperature. Fig. 1 provides an example for LTHS-creep. It shows data from five interrupted creep tests from the SX ERBO1/C [23,24]. We discuss the details of Fig. 1 later. Here it is important to highlight that when plotted as logarithmic of strain rate vs. strain or vs. logarithm of strain, the creep curves show two minima, separated by an intermediate maximum, Fig. 1b and c. This behavior which we refer to as double minimum creep (DM-creep) was first reported more than two decades ago [13–16]. While the

early decrease in creep rate towards the first minimum did not receive much attention, the subsequent increase of creep rate up to the intermediate creep rate maximum was reported in several studies [17–22]. Why creep rates evolve in this way is an open question. The scientific objective of the present work is to explain the elementary processes which govern DM-creep.

2. Material and experiments

2.1. Material and scanning electron microscopy

The present work is part of the research program of the collaborative research center SFB/TR 103 [23]. We investigate a SX of type CMSX-4, referred to as ERBO/1C. All details concerning this designation, the alloy composition and the heat treatment of the material are given elsewhere [24]. Fig. 2 shows micrographs obtained using scanning electron microscopy (SEM) with secondary electron (SE) contrast. Fig. 2 shows the γ/γ' -microstructure of the material before and after creep. SEM was performed using a FEI system of type Quanta 650 FEG, all details regarding the SEM procedure have been published elsewhere [25]. Fig. 2a shows a {100} cross section prior to creep. The γ' -cubes (ordered L1₂ phase) have average cube edge lengths of 0.4 μm (present work and [24]). Their volume fraction is between 70 and 80%. The average γ -channel (fcc crystal structure) width is 65 nm (present work and [24]). Fig. 2b shows a SEM micrograph taken from a {111} cross

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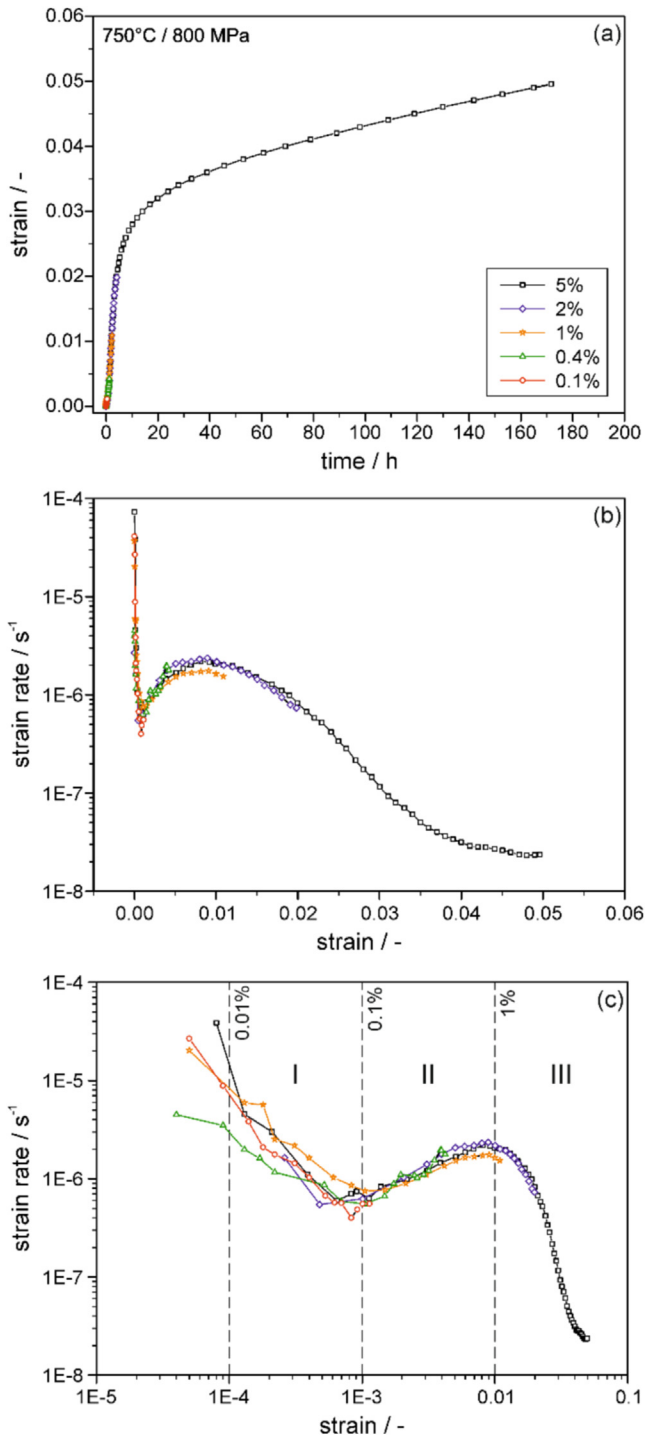


Fig. 1. Creep curves from interrupted tests of ERBO/1C (750 °C, 800 MPa). (a) Strain ϵ as a function of time t . (b) Logarithm of creep rate as a function of strain. (c) Logarithm of creep rate as a function of logarithmic strain.

section prior to creep. Due to the spatial arrangement of the γ' -cubes these can have triangular (small arrow pointing up) and hexagonal (small arrow pointing down) γ' -shapes on the metallographic cross section (see schematic sketches in Refs. [26,27]). Fig. 2c and d shows {100} and {111} cross sections after 5% (260 h) creep at 750 °C and 800 MPa. From Fig. 2c and d it is clear that there is no significant rafting. Previous transmission electron microscopy (TEM) studies have shown that the initial material prior to creep

contains only a low dislocation density in the γ -channels and no planar faults in the γ' -phase [25,28].

2.2. Miniature creep testing

Miniature creep specimens have two advantages as compared to standard size specimens. First, a larger number of creep specimens can be obtained from a limited amount of material. Second, it is easy to obtain precisely oriented specimens. $\langle 001 \rangle$ miniature tensile creep specimens were prepared combining the Laue method with electro discharge machining. Miniature creep testing was performed using a creep machine from Denison Mayes, equipped with a vertically movable three zone furnace. The heating zones were monitored by three thermocouples and controlled by Eurotherm controllers. The miniature creep specimens (gauge length: 9 mm) were positioned in the temperature constant zone of the furnace (at 750 °C: > 100 mm). In addition to the three thermocouples which control the three heating zones, two measurement thermocouples were fixed to the upper and lower ends of the gauge lengths. The precision of the temperature measurement at 750 °C is ± 1.5 °C. The miniature specimens are mounted in special grips consisting of an ODS alloy PM 3030, reinforced by ceramic Al_2O_3 insets. Displacements were measured using ceramic rod in tube extensometry and strain sensors positioned outside of the furnace. In the present work, the specimens were heated to the test temperature of 750 °C under a preload close to 20 MPa in 2 h. The small preload is required to keep the load line aligned. The load, corresponding to a stress of 800 MPa, was then applied within only a few seconds. The immediate elastic reaction of the specimen/grip assembly was not considered as creep strain. With respect to the scientific objectives of the present work it is important to highlight that the specimen was heated up to test temperature as fast as possible, that the preload was much smaller than the test load and that the full test load was applied within only a few seconds. Further details of miniature tensile creep testing have been published elsewhere [29–34].

Miniature creep tests were interrupted after strain intervals ranging from 0.1 to 5%. Fig. 1a presents corresponding creep data in a strain ϵ vs. time t plot. Fig. 1b shows how the creep rate evolves with strain in a log-linear plot of strain rate vs. strain. Fig. 1a and b prove that our material shows a reproducible creep behavior. This also holds for very small strains, as can be seen from the logarithm of strain rate vs. logarithm of strain plot in Fig. 1c. The first dashed vertical line in Fig. 1c corresponds to a plastic strain of 0.01%. For our specimen geometry (gauge length: 9 mm) this corresponds to a displacement of about 1 μm . This type of displacement can be resolved by electronic/mechanic data collection systems. It is, however, close to the resolution limit of creep testing. The scatter of the five creep curves shown in Fig. 1c between 0.01 and 0.1% strain is small and decreases with increasing strain. The results presented in Fig. 1c clearly show that there is a decrease of creep rate between 0.01 and 0.1%, where the first creep rate minimum is observed (marked by the second vertical dashed line in Fig. 1c). It takes about 30 min to reach this first creep rate minimum. The creep rate then increases towards an intermediate maximum at about 1% (third vertical dashed line in Fig. 1c), before it decreases towards a global minimum close to 5% strain. In Fig. 1c we identify three regimes referred to as I, II and III. In regime I creep rates sharply decrease towards a first minimum. In regime II, the creep rate increases from the first sharp minimum towards an intermediate maximum. Then, in regime III, creep rates decrease from the intermediate maximum towards the second broad global minimum. A full 750 °C and 800 MPa $\langle 100 \rangle$ tensile creep curve for the material of the present study which shows the evolution of creep rate after the second minimum towards final rupture is shown in Ref. [34]. Tertiary creep

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