

CYCLIC CREEP BEHAVIOR OF THE SINGLE CRYSTAL SUPERALLOY SRR99 AT 980 °C

P.D. Portella, J. Kinder, H. Frenz, A. Bertram

Federal Institute for Materials Research and Testing (BAM)
Unter den Eichen 87, D-12205 Berlin, FRG, Tel. (030)8104-0, Fax (030)8112029

ABSTRACT

Single crystalline Ni-base [001]-oriented SRR 99-specimens were subjected to different stress controlled cyclic creep at 980 °C. At this relatively high homologous temperature the γ/γ' -microstructure was altered during the experiments after less than 50 h. The initial cuboidal γ' -morphology was transformed quite rapidly into a more or less interconnected platelike one, being at least partly oriented in a distinct manner to the loading axis.

KEYWORDS

single crystal superalloy, monotonous creep, cyclic creep, deformation behaviour, γ/γ' -morphology, microstructural evolution, quantitative image analysis, modelling

INTRODUCTION

Creep-fatigue interaction is a commonly used term for the description of a fairly understood interaction of a number of different deformation and damage mechanism. Because both, creep and fatigue phenomena depend not only on testing conditions but also on the relevant microstructure of the material tested. It is claimed that the materials reaction on a superimposed mode of load history might be observable in a more distinctive way, and thereby better understood in single crystalline material.

MATERIAL, EXPERIMENTAL SETUP

Single crystalline Nickel base superalloy SRR 99 is used for aeronautical turbine blades. Specimen with the [001]-orientation were subjected to different stress controlled static and cyclic loading conditions. Static creep tests were performed in a 40 kN static load machine, equipped with a three zone furnace, guaranteeing a temperature gradient along the 50 mm measurement length of less than 3 °K. The elongation was measured using an extensometer with an initial gauge length of 50 mm. A series of interrupted test at 980 °C with an initial stress of 200 MPa was run. Cyclic creep tests were performed using an Instron 100 kN test system. A three zone furnace guaranteed a temperature gradient below 3 K along the specimen axis. Elongation was measured with a MTS-Extensometer with an initial gauge length of 21 mm. Table 1 shows the parameters used:

Table 1: Test parameters for cyclic creep tests
Temperature = const. $\hat{=}$ 980 °C

high stress level [MPa]	200	200	200
low stress level [MPa]	20	20 - 60	-200
High: hold time [s]	300	300	300/30
low: hold time [s]	10 ... 300	300	300/30

MICROSTRUCTURAL OBSERVATIONS

After the experiments the specimens were cut in order to produce cross sections oriented nearly to the (001)-plane, and longitudinal sections near to (100). The dendritic structure and the γ/γ' -microstructure were revealed by preferentially etching the γ' -phase. The microstructure of all chosen specimens was analyzed by a SEM (CamScan S2 equipped with EDX analyzer) mainly by detecting secondary electrons from the specimens sections.

The initial microstructure of SRR99 after standard heat treatment is shown in fig. 1 in the upper left corner. The microstructure is composed of quite well aligned γ' -cuboids being monomodally dispersed in the γ -matrix. The γ' -content was determined by image analysis to be about 60 vol%. In the course of straining during monotonous tensile creep tests this microstructure changed quite rapidly to a lamellar one with γ/γ' -platelets being oriented perpendicular to the stress axis (parallel to the (001)-plane, respectively). This microstructural evolution is also shown in fig. 1 which visualizes the results of several interrupted creep experiments at 980 °C under initial tensile loadings of 200 MPa.

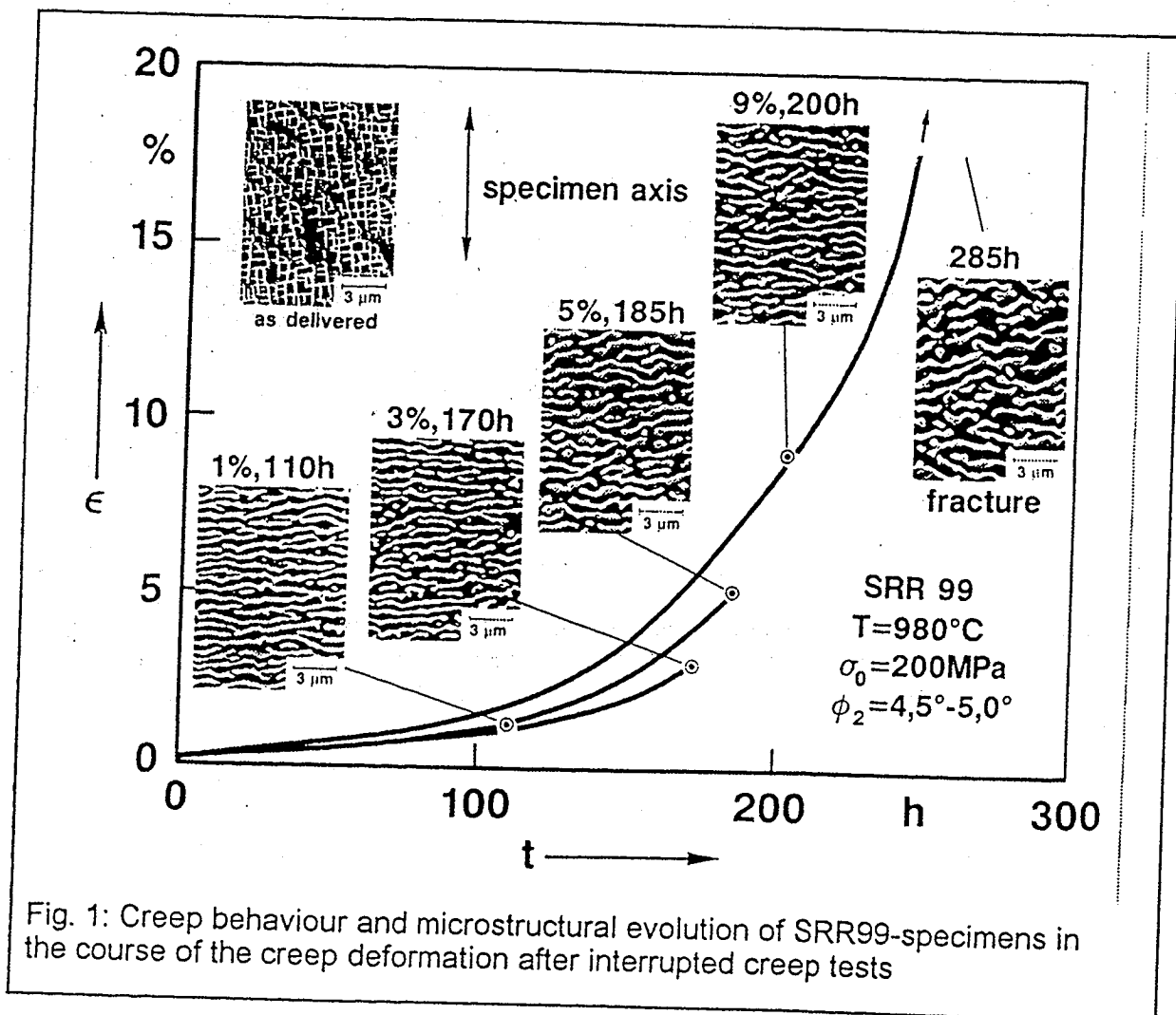


Fig. 1: Creep behaviour and microstructural evolution of SRR99-specimens in the course of the creep deformation after interrupted creep tests

Interestingly, it becomes visible that not only the γ/γ' -morphology changes significantly, but also the γ/γ' -topology. In the initial condition, cuboidal γ' -particles are dispersed in a γ -matrix and already after little straining (1 %) in about 100 h the microstructure becomes inverted, i.e.

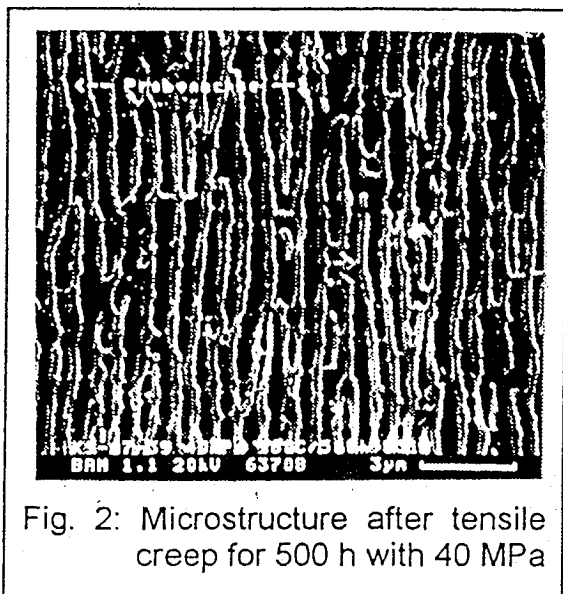


Fig. 2: Microstructure after tensile creep for 500 h with 40 MPa

now platelike γ -particles are dispersed in a γ' -matrix.¹ Even after almost zero straining during a tensile creep experiment with very low applied stress (i.e. 40 MPa) the earlier stages of this microstructural evolution before the microstructural inversion could be observed (fig. 2). Chopping of the elongated γ' -platelets formed at the early experimental stages might be a principal reason for this microstructural inversion in the course of further straining and one can see in fig. 1 that the continuation of creep then leads to a coarsening of the γ -platelets in stress direction but concurrently to a decrease of their lateral dimensions. According to the results of the image analysis on cyclically loaded specimens (see there for comparison) we assume that also under monotonous creep conditions significant reductions of the γ' -contents are occurring.

This evolution of the γ/γ' -microstructure and the softening during the final stages of our creep experiments might be explained perhaps by an increasing dislocation activity on $\{111\}$ -planes which are known to be favoured in Ni-based alloys (1). The successively resulting redistribution of both phases should increase the free length of dislocation paths when this microstructural alteration occurs, thereby increasing the strain rate in the final creep stage.

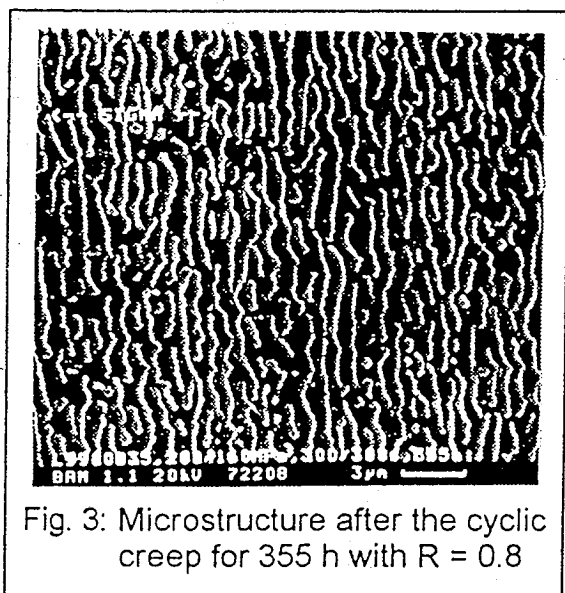


Fig. 3: Microstructure after the cyclic creep for 355 h with $R = 0.8$

Cyclic creep of the specimens leads to the same lamellar γ/γ' -microstructure at least as long as these experiments can be regarded as special tensile creep experiments being "switched" between two different tensile stress levels. A typical microstructure after cyclically straining to 4.8 % within a total experimental time of 355 h is shown in fig. 3. This specimen was cyclically stressed for 300 s at 200 MPa and 160 MPa each ($R = 0.8 = 160 \text{ MPa} : 200 \text{ MPa}$), which means that the cumulated time under high and under low stress was about 180 h each. The content of γ' was reduced to 51 vol % in the course of this experiment. In comparison to fig. 1 one can see that the resulting microstructural evolution is very similar to the creep specimen being strained to 5 % in 185 h.

This means that the mechanisms for such an evolution should be the same. It should be mentioned, that cyclic creep with higher stress relief (i.e. $R = 0.1 = 20 \text{ MPa} : 200 \text{ MPa}$) led to little strains of only about 0.7 % after 524 h total experimental time (i.e. 262 h at high stresses) and again to a similar microstructure to the one which is shown in fig. 1 for 1 % strain after 110 h. From these identical microstructural evolutions in the course of monotonous and cyclic creep experiments we claim, that the causal mechanisms may be probably strain-induced. The γ' -content after this cyclic experiment was determined in the (001) -section to 54 vol% in a dendritic and 55 vol% in an interdendritic region, whereas in

¹It has to be pointed out, that in literature usually the same morphological evolution of the γ/γ' -microstructure is described (2). The principal difference to our results is the interpretation of the microstructural examinations, i.e. according to the literature γ always acts as matrix with dispersed γ' -plates.

a dendritic region in a (111)-section the γ' -content was 53 vol%.

A very different microstructural evolution under cyclic creep occurred, when the loads have been changed between tensile stresses of 200 MPa and compressive stresses of -200 MPa ($R = -1$). Again, the hold times at each stress level were 300 s. Because of the strain reversion in the course of the cycling (instead of the stress relief during the other cyclic experiments) the final deformation after the total experimental time of 328 h was therefore negligible. The maximum values of strains were recorded with $\epsilon = \pm 0.4\%$. In fig. 4 it is shown, that the γ/γ' -microstructure has been developed mainly inclined at about 50° to the stress axis after cycling, thus forming a γ -network parallel to (100) with irregular γ' -particles inside. The angle mentioned coincides quite good with the crystallographic angle of 54° between (001) and (111), therefore it seems obvious that under these circumstances many of the γ/γ' -interfaces are reoriented parallel to (111)-planes, again due to pronounced dislocation activities on these preferred planes (1). This is why we assume, that the microstructural evolution in γ/γ' -containing Ni-base alloys is not only stress- but also to different amounts strain- (i.e. dislocation-) induced. The image analysis for this special microstructure revealed a strong decrease of the γ' -content to 47 vol% in dendritic regions but a very less strong decrease to 57 vol% in interdendritic regions of the specimen. This difference is thought to be caused by small angle boundaries initially present in the interdendritic regions which seem to promote the redistribution of the phases.

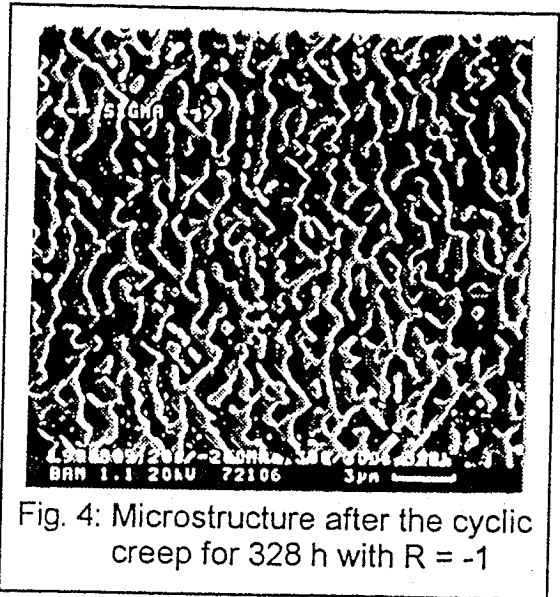


Fig. 4: Microstructure after the cyclic creep for 328 h with $R = -1$

γ/γ' -interfaces are reoriented parallel to (111)-planes, again due to pronounced dislocation activities on these preferred planes (1). This is why we assume, that the microstructural evolution in γ/γ' -containing Ni-base alloys is not only stress- but also to different amounts strain- (i.e. dislocation-) induced. The image analysis for this special microstructure revealed a strong decrease of the γ' -content to 47 vol% in dendritic regions but a very less strong decrease to 57 vol% in interdendritic regions of the specimen. This difference is thought to be caused by small angle boundaries initially present in the interdendritic regions which seem to promote the redistribution of the phases.

MODELLING

For design purposes of turbine blades a constitutive model is needed which describes the behaviour under both monotonous and cyclic loads, at least in their primary and secondary stages. As the main loading direction of such blades of single crystal material or of directionally solidified material is the $\langle 001 \rangle$, one could at a first step limit the consideration to uniaxial stress states in this orientation. For other stress states the material behaviour is dominated by the anisotropy and needs a fully three-dimensional anisotropic model.

In the present paper a material model is shown, which is capable to describe the uniaxial creep behaviour [see 3 - 10] and, furthermore, serves for the three-dimensional generalization [see 4, 7]. The starting point for such a modelling is given by the two differential equations in time which are based on a four-parameter viscoelastic BURGERS-model

$$\dot{\sigma} = (C+K) \dot{\epsilon} - (C/L + C/D + KL) \sigma + C/D \tau \quad (1)$$

$$\dot{\tau} = K(\dot{\epsilon} - \sigma/L) \quad (2)$$

Here σ denotes the stress, ϵ the strain, and τ an internal variable with dimension *stress*. The four material parameters are two elastic moduli C and K with dimension *stress* and two viscosities L and D with dimension *stress/time* which depend on the applied stress. The strong non-linearity of the creep behaviour is described by the stress dependence of the viscosities

$$D = D_0 \exp(-B |\sigma|) \quad (3)$$

$$L = L_0 \exp(-B |\sigma|) \quad (4)$$

with (positive) material constants B , D_0 , and L_0 . As the exponents in Eqs. 3 and 4 are always non-negative, high stress intensities lead to low viscosities and thus accelerate the creep. Under monotonous creep conditions D and L are constant and can be easily determined from experimental data. The instantaneous elastic response is governed by the elasticities in parallel $C + K$. The secondary or steady state creep viscosity is determined by L , whereas D is responsible for the primary creep behaviour.

The damage evolution has been implemented into this model by a KACHANOV-damage variable δ between 0 and 1 which reduces the apparent cross section area to the effective one and, accordingly, amplifies the nominal stress σ up to the effective stress

$$\sigma_e = \sigma / (1 - \delta) . \quad (5)$$

The evolution equation for δ is taken in analogy to Eqs. 1 and 2

$$\delta^* = C_1 \sigma + C_2 \dot{\epsilon}^* + C_3 \tau + C_4 \delta . \quad (6)$$

Comparison with experiments show that in this specific case only the first and the last term are needed for satisfactory results. The remaining seven (positive) constants of the entire model C , K , B , D_0 , L_0 , C_1 , and C_4 have been identified by creep test at 980° C for SRR99.

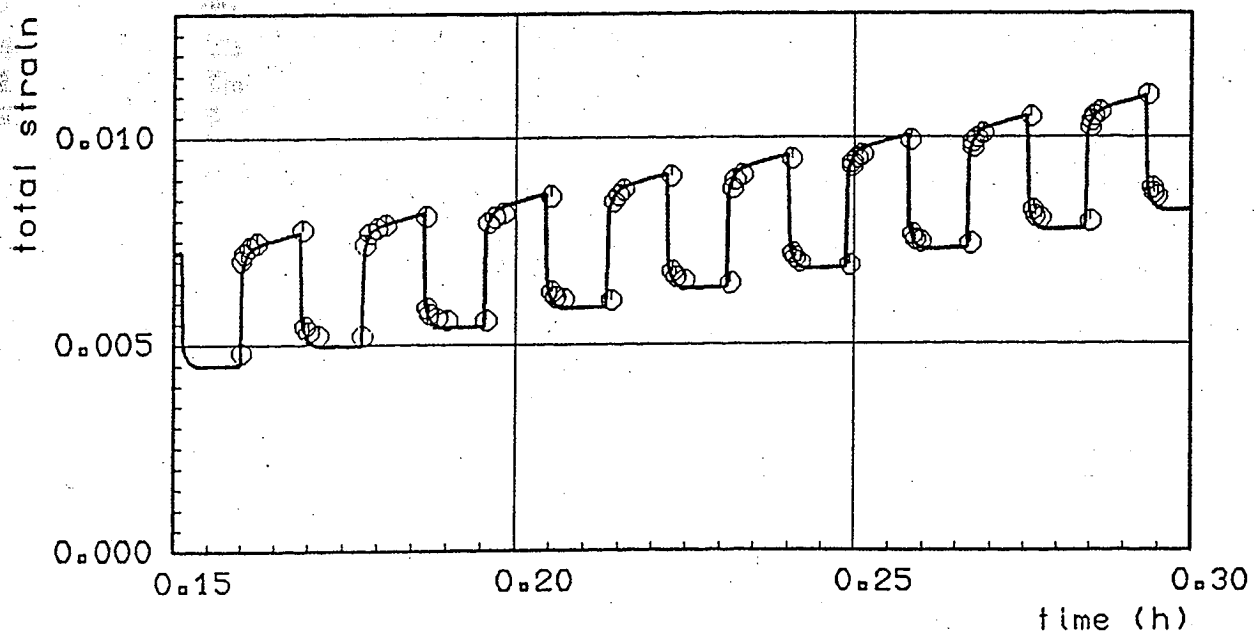


Fig. 5: Cyclic creep test (symbols) and model calculation (curve).

This model has been applied to describe the uniaxial response of SRR99 under both monotonous and cyclic creep conditions with or without hold times as well as under LCF conditions. In Fig. 5 the experimental data (symbols) and the calculations by this model (curves) are shown for a cyclic creep tests with hold times. In the Fig.5 a selection of cycles have been plotted for the upper load $\sigma_u = 200 \text{ MPa}$ with upper hold time $t_u = 30 \text{ s}$ and lower load $\sigma_l = 20$

MPa with lower hold time $t_h = 30$ s. In both upper and lower hold times a short primary creep phase can be observed before a steady state creep is again reached. This primary creep is in load direction for the upper hold time, but against it in the lower case, an experimental finding which is reproduced by the model.

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