

The Influence of the γ/γ' Microstructure Stability on the Very High Temperature/Low Stress Non-isothermal Creep Behavior of a 4th Generation Single Crystal nickel-based Superalloy

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ABSTRACT

Specific non-isothermal creep conditions were applied to a 4th generation single crystal nickel-based superalloy. It is shown that the residual creep life after overheating depends on the overheating duration and that an optimal pre-creep time exists for which the longest residual life is obtained.

1 INTRODUCTION

Single crystal nickel-base superalloys are widely used as material for high temperature components, especially as aircraft and helicopter turbine blades. Such materials have demonstrated the best capabilities in term of environmental resistance and creep strength. The microstructure of superalloys consists in a high volume fraction of coherently precipitated γ' cubes separated by thin channels of γ matrix. Initially, the γ' phase volume fraction is approximately 70%. Over 1000°C, massive dissolution of the γ' phase occurs and leads to a decrease of mechanical properties in creep. In the latest 4th generation of superalloy developed, Rhenium (Re) and Ruthenium (Ru) were substituted to other refractory alloying elements in order to increase the γ' stability at high temperature. This leads to a great improvement of very high temperature isothermal creep (IC) properties. However, little is known on the behavior of Ni-based superalloys during non-isothermal creep (NIC). Such regime can occur during emergency situations, when one engine of a biturbine helicopter stops. In this case, the remaining engine experiments an abrupt temperature jump to compensate the loss of power. The overheating lasts for few seconds until the cruising regime is recovered. The purpose of this paper is to determine the evolution of the residual creep life and behavior at 1050°C after a jump at very high temperature (1200°C) occurring at different times of the isothermal creep life at 1050°C.

2 EXPERIMENTAL

2.1 Material

The material used for this study is the new 4th generation superalloy MCNG, developed by ONERA. Its weight composition is given in table 1. After standard heat treatments, the volume fraction of the strengthening γ' phase is around 66% and the cube size is about 0.6 μm . The specimens used for creep tests were machined out from single crystal rods with orientation within 5° off [001] along the strain axis.

| Ni | Cr | W | Mo | Re | Al | Ti | Ta | Ru | Hf |
|------|----|---|----|----|----|-----|----|----|-----|
| Bal. | 4 | 5 | 1 | 4 | 6 | 0.5 | 5 | 4 | 0.1 |

Table 1. Chemical composition of MCNG (wt. %)

2.2 Experiments

The IC tests were carried out in air at 1050°C and $\sigma_0 = 140$ MPa using constant-load creep testing machine. The material was tested until rupture in order to get the behavior of MCNG in this creep condition and the isothermal reference creep life t_{iso} . NIC tests consist in performing a short temperature plateau at 1200°C, with heating/cooling rates close to 60°C.s⁻¹, in the course of an IC test at 1050°C/ σ_0 . Therefore, NIC tests consist in 3 steps (Figure 1): (i) the pre-creep step when the material is crept at 1050°C under σ_0 during t_{pre} ; (ii) the overheating step when the material is quickly heated at 1200°C for $t_0 = 30\text{s}$ or 150s and finally (iii) the residual creep, when the material is again crept at 1050°C during t_{res} . A specific test rig, described in [1], is used to achieve the high heating and cooling rates required for the overheating step to be representative of the real conditions.

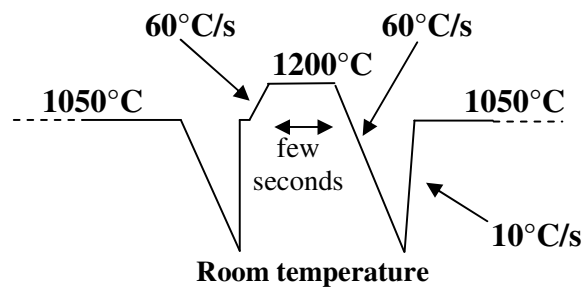


Figure 1: NIC procedure

The evolutions of the residual creep behavior are linked to the two variables of NIC tests t_{pre} and t_0 .

3 RESULTS AND DISCUSSION

3.1 Isothermal creep behavior

The creep curves of MCNG at $1050^{\circ}\text{C}/\sigma_0$ are presented in Figure 2. Figure 2a shows ε vs t and figure 2b shows $\dot{\varepsilon}$ vs t . In this creep condition, there is no visible primary stage. A short incubation period is eventually observed in the first hours of the strain creep curve; during this period there is no measurable strain [2]. In fact, the creep behavior of MCNG at $1050^{\circ}\text{C}/\sigma_0$ can be seen as a short steady-state regime followed by a long tertiary regime. Indeed, the tertiary creep stage starts just before $t/t_{\text{iso}} = 0.30$ and it covers around 70 % of the creep live. This long tertiary finally leads to an important creep strain.

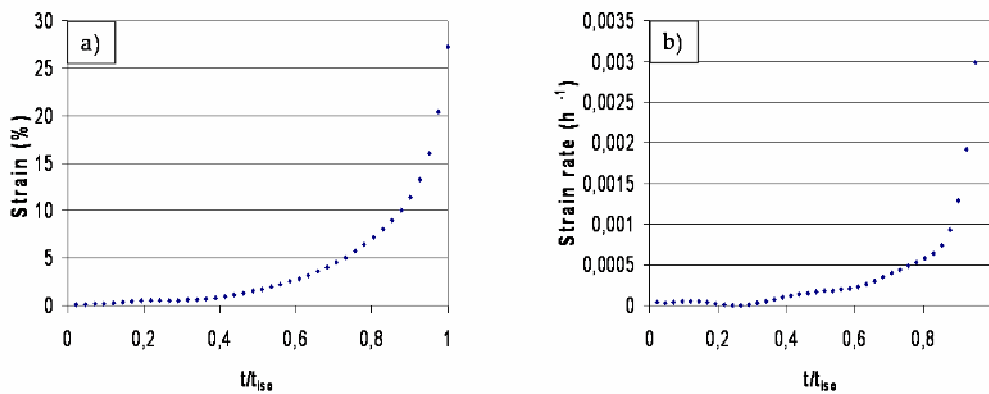


Figure 2: Creep behavior at 1050°C / 140 MPa , (a) $\varepsilon = f(t)$, (b) $\dot{\varepsilon} = f(t)$

Overheating were performed after $t_{\text{pre}}/t_{\text{iso}} = 0.08, 0.16, 0.34$ and 0.68 . Microstructure observations of the different pre-crept specimens were performed by scanning electron microscope (SEM).

3.2 Microstructures for the different t_{pre}

At $t_{\text{pre}}/t_{\text{iso}} = 0.08$, the microstructure is at an intermediate state of rafting. It is almost completed in dendrite cores (DC) while interdendritic regions (IR) still exhibit well defined γ' cubes (Figure 3).

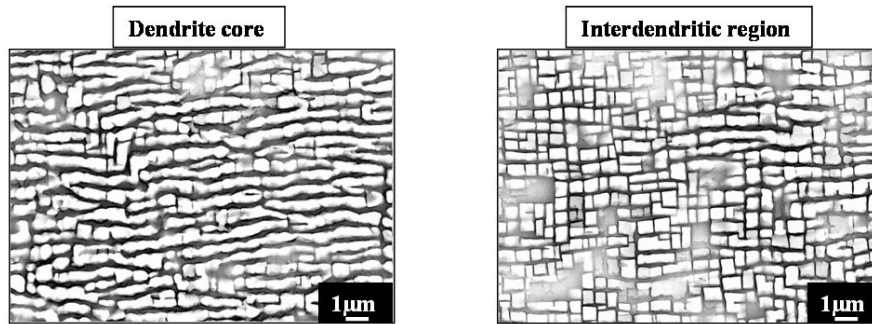


Figure 3: Heterogeneous microstructure after 50 hours of creep

At $t_{pre}/t_{iso} = 0.16$, the microstructure is very homogeneous, i.e. the rafted microstructure is established in the entire specimen (Figure 4). Average channel widths are very close in DC and IR (332 nm in DC, 337 nm in IR). As for observations on the initial cube microstructure, γ' rafts are thicker in IR (566 nm in average) than in DC (510 nm in average).

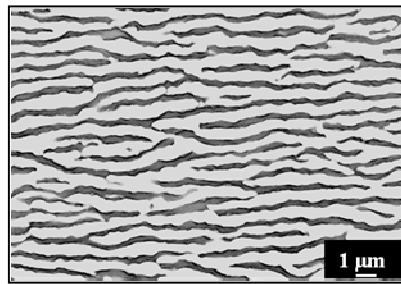


Figure 4: Homogeneous raft microstructure after 100h of creep

At $t_{pre}/t_{iso} = 0.34$, rafts are still well established and regular, but the microstructure has evolved compared to the one obtained at $t_{pre}/t_{iso} = 0.16$. In fact, the γ' rafts have noticeably thickened. The γ channels have widened in both DC and IR, but γ widening is greater in the IR ($\Delta \gamma = +44\%$ in IR, $\Delta \gamma = +12\%$ in DC).

After $t_{pre}/t_{iso} = 0.68$ at 1050°C / 140 MPa, the γ/γ' microstructure is greatly disturbed (Figure 5). The γ' phase is very irregular and has considerably thickened. Topological inversion is clearly established: the γ' phase now surrounded the γ phase. These phenomena are even more developed in the IR so that rafts cannot be distinguished anymore in these regions. Such behavior has already been mentioned for superalloys with a γ' volume fraction higher than 50% in [3]. Concerning the different behavior between DC and IR, it's linked to the effects of the discrepancies in chemical composition between the two regions due to the alloying elements segregation [4, 5].

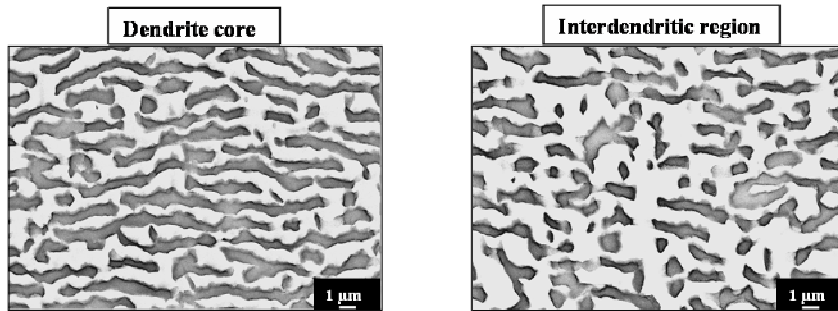


Figure 5: Microstructure after 400 hours of creep

In conclusion, rafting of the γ' phase completes between $t_{pre}/t_{iso} = 0.08$ and 0.16 . In fact, the long tertiary creep stage is promoted by the constant evolution of the γ/γ' rafted microstructure in IC conditions. Soon after rafting, the microstructure of MCNG evolves rapidly towards topological inversion between γ and γ' . Such a phenomenon is known to be coincident with the tertiary stage onset [3, 6].

3.3 Non-isothermal creep results

Figure 6 shows an example of the evolution of creep strain rate against time during NIC of the material. In this case, $t_{pre}/t_{iso} = 0.34$ and $t_0 = 150$ s.

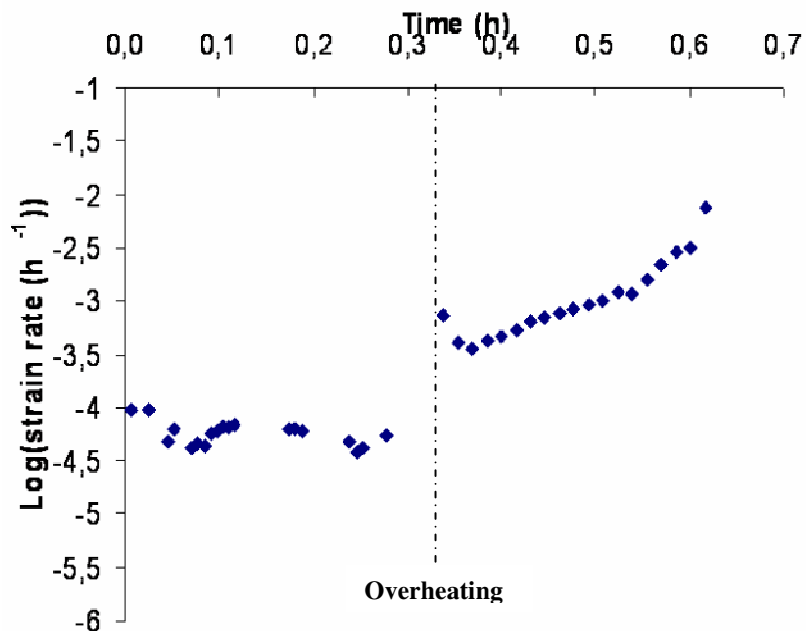


Figure 6: NIC behavior, $t_{pre}/t_{iso} = 0.34$, $t_0 = 150$ s

During an overheating, MCNG cumulates a very small strain that never exceeds 0.3%. During the residual creep step, the material exhibits a primary creep stage which amplitude increases along with the overheating duration t_0 . At the end of the primary stage, the behavior of the material is comparable to the IC with accelerated kinetics (Figure 6).

Microstructure quantifications in terms of γ' fraction ($F_{\gamma'}$) were performed just after overheating and showed that $F_{\gamma'}$ has decreased. This phenomenon is even more marked after a longer overheating duration t_0 (Table 2). In fact, the γ' rafts thin down and the γ channels thicken. Surprisingly, the thickening of the γ channels is greater than the thinning of the γ' rafts; In fact, the γ channels benefit not only from the dissolution of the γ' but they are also strained during the overheating.

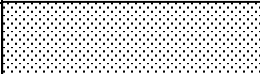

| | $t_0 = 30s$ | $t_0 = 150s$ |
|--|--|---|
| Initial microstructure ($t_{pre}/t_{iso}=0$) |  | $\Delta F_s \gamma' = - 13.4 \%$ |
| Microstructure for $t_{pre}/t_{iso} = 0.16$ | $\Delta F_s \gamma' = - 5 \%$ | $\Delta F_s \gamma' = - 10 \%$ |
| Microstructure for $t_{pre}/t_{iso} = 0.34$ | $\Delta F_s \gamma' = - 5.5 \%$ |  |

Table 2: Variation of γ' phase fraction after overheating

$$\Delta F_{S_{\gamma'}} = \frac{F_{S_{\gamma'}}^{post-overheating} - F_{S_{\gamma'}}^{pre-overheating}}{F_{S_{\gamma'}}^{post-overheating}}$$

It was shown in a previous paper [7] that post-overheating primary stage results from the deviation from the equilibrium γ' fraction and temporally increased dislocation activity in the γ channels. After an overheating, when residual creep at 1050°C proceeds, γ' rafts coarsen again towards the temperature equilibrium γ' fraction; the strain rate slows down along with the resulting narrowing of γ channels, by reducing dislocation motion.

Figure 7 plots t_{res}/t_{iso} against t_{pre}/t_{iso} for the two overheating durations. The isothermal reference, represented by a straight line, is also given on the figure. In most of the cases, an overheating penalizes the total creep life compared to IC life. A longer t_0 leads to a smaller residual creep life (the dotted line is above the continuous one). In fact, whatever t_0 , the residual minimum strain rate is always higher than the pre-creep strain rate. This strain rate increase leads to a faster tertiary stage initiation that finally reduces the creep life. Indeed, the higher the residual creep rate, the shorter the residual creep life.

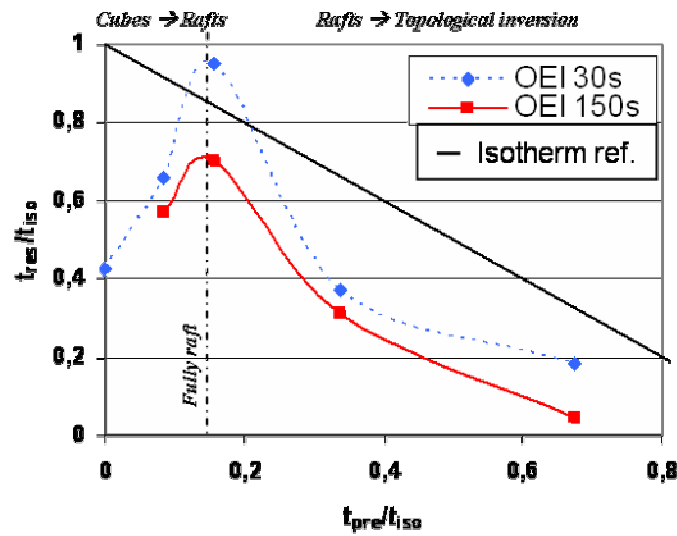


Figure 7: Evolution of residual creep life t_{res}/t_{iso} vs. pre-creep time t_{pre}/t_{iso} for the two overheating durations

Concerning the influence of t_{pre} , the tendency is the same for the two overheating durations: MCNG presents a maximum residual creep life when the overheating occurs for $t_{pre}/t_{iso} = 0.16$. In fact, the residual creep life seems to be linked to the γ/γ' microstructure on which the overheating is simulated: the impact of the overheating on the creep life is the smallest when it is performed on a homogeneous and stable fully rafted microstructure. Therefore, along with the effect of t_0 , it suggests that the overheating promotes a faster evolution of the microstructure (favoring raft destabilization) favoring accelerated creep. Complementary microstructure analyses are in progress in order to check out the evolution kinetics after an OEI.

4 CONCLUSION

The γ/γ' microstructure of MCNG is in constant evolution at $1050^\circ\text{C}/\sigma_0$; this tendency to destabilization is assumed to be responsible for the particular isothermal creep behavior of the material in this condition. The occurrence of an overheating in the course of an IC test reduces the total creep life of the material. Nevertheless, it exists an optimal pre-creep time (t_{pre}) for which the longest residual live is measured after the overheating. This is linked to both the homogeneity of the rafted microstructure and its evolution developed during t_{pre} .

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