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Effect of fine γ' precipitation on non-isothermal creep and creep-fatigue behaviour of nickel base superalloy MC2

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ABSTRACT

A novel technique, combining on the one hand creep-fatigue tests with an overheating and creep tests with thermal cycling in the other hand, performed on γ/γ' nickel base single crystal superalloy MC2 have led to an increased understanding of fine γ' precipitation and its strengthening effect.

Both creep and creep-fatigue tests were conducted at 1050 °C with 1200 °C overheating for creep-fatigue experiments and with repeated overheatings at 1100 °C and 1150 °C for creep.

The resulting microstructures of these experiments were examined using both scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It appears, both on creep or creep-fatigue, once an overheating is experienced a fine γ' precipitation occurs in γ matrix. These precipitates seem to have a transient strengthening effect on the mechanical properties. For the creep-fatigue experiments a decrease of the plastic strain rate was measured at once after the temperature peak. In the case of the creep tests under thermal cycling, no extra deformation induced by the overheating at 1100 °C was recorded. However, overheatings at 1150 °C lead to a plastic strain jump which progressively decreases upon thermal cycling, due to the formation of fine γ' precipitates. Furthermore, the γ' fine particles seem to have a hardening effect that vanishes once they dissolve.

1. Introduction

Nickel base single crystal superalloys are widely used in the hottest parts of aircraft turboengines [1]. Blades made of these alloys operate for thousands of hours and withstand a nominal temperature as high as 1050 °C. These alloys are chosen for their high mechanical performances at high temperatures range in particular, their high temperature creep resistance. They take these interesting properties from the precipitation of high volume fraction (close to 70%) of long range $L1_2$ ordered γ' phase which appears as cubes coherently embedded in fcc solid solution γ matrix.

Several studies reported in the literature have been conducted on the isothermal creep behaviour under various temperature and loading conditions [2–18]. Two main domains of microstructure evolution can be distinguished depending on creep test conditions. Indeed, at low temperature ($T < 900$ °C) and high stress, no significant evolution of the initial microstructure is observed; actually the γ' precipitates morphology remains almost unchanged with a possible growing of precipitate edge length due to Ostwald ripening processes. However at high temperature ($T > 1000$ °C) and low stress conditions, a significant modification of the microstructure is noticed consisting of directional coarsening of the γ' precipitates perpendicular to the applied stress axis for the majority of commercial single crystal superalloys with a negative misfit ($\delta = 2(a_{\gamma'} - a_\gamma)/(a_{\gamma'} + a_\gamma) < 0$ where $a_{\gamma'}$ and a_γ are the lattice parameters of γ' and γ phases, respectively). This morphological evolution, or also "rafting", usually takes place at the end of primary creep stage where the γ' phase has entirely coalesced and the γ channels become wider along the applied stress axis [8,14,15].

Besides this, during in-service operation of twin engine helicopters, one of the two engines may unexpectedly stop. This implies a brutal temperature rise up to around 1200 °C for the remaining engine operating. Such kind of event is now included in the certification procedures of turboshaft engines for helicopters and is known as One Engine Inoperative (OEI) rating. Consequently, creep or creep-fatigue tests under isothermal conditions become insufficient to tackle the characterization of the mechanical properties properly in the non-isothermal loading conditions described above. So far only few studies dealing with mechanical testing under non-isothermal conditions have been conducted [19–21]. It was established from these studies that the γ' precipitation evolution is a significant creep controlling parameter. Besides, it is well

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Table 1
Composition of MC2 single crystal superalloy (in wt.%).

Ni	Cr	Co	Mo	W	Al	Ti	Ta
Balance	8	5	2	8	5	1.5	6

admitted in the literature, that the major loss of the mechanical properties at very high temperature ($T > 1100\,^{\circ}$C) is directly linked to microstructure evolution due to the strengthening γ' phase dissolution [22,23].

According to the above-mentioned experiments, the temperature peak seems to be a crucial parameter in controlling mechanical tests at very high temperature conditions. Even more, the one engine inoperative conditions require a special handling of the temperature control, particularly temperature levels and heating/cooling rates. This paper tackles these issues in extreme conditions such as the effect of an overheating or thermal cycling on creep and creep-fatigue tests at very high temperature. The material considered for this study is MC2 nickel base single crystal superalloy free of rhenium and ruthenium made by ONERA. The influence of the overheating on mechanical behaviour and on the microstructure evolution is determined and clarified. A special attention will be paid to γ' fine precipitation effect.

2. Experimental procedure

2.1. Material

The material considered here is γ/γ' MC2 single crystal, classified as a first generation nickel base superalloy. Its microstructure consists of a large amount of refractory elements in addition to cobalt, chromium and aluminium: Table 1 gives its composition in weight percent. The as-received material was homogenized 3 h at 1300 °C then air quenched to dissolve all the γ' particles formed either by eutectic reaction during solidification or by precipitation during cooling. The material was then aged at 1100 °C for 5 h and air quenched, then at 870 °C for 16 h followed by air quench. After the heat treatment, the strengthening γ' phase is cubic with an average precipitate edge length of 0.4 μm and a volume fraction, $f_{v\gamma'}$ of 70% [24–27], as illustrated in Fig. 1.

The geometry of the testing specimens is given as follows: for the creep-fatigue tests, the samples were 14 mm in gauge length with a prismatic section of 6 mm wide and 2 mm thick and were oriented ⟨0 0 1⟩ and ⟨1 1 1⟩. For creep tests, the samples were 14 mm in gauge length and 4 mm in diameter and had only ⟨0 0 1⟩ orientation. The influence of the sample orientation will be discussed only for creep-fatigue tests and enriched by the results obtained from the non-isothermal creep tests.

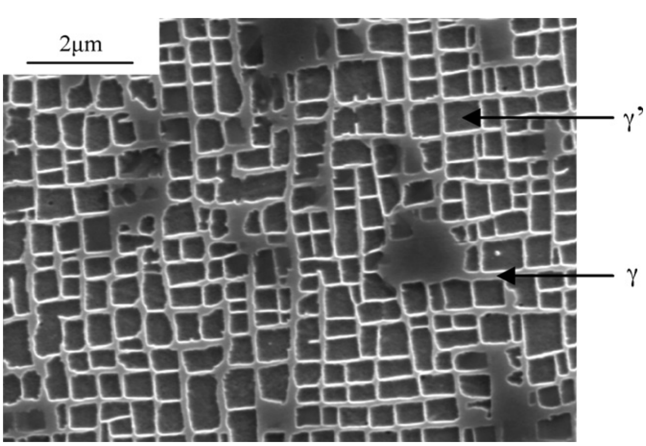

Fig. 1. As-received microstructure of the MC2 single crystal superalloy after standard heat treatment.

2.2. Microstructure

The microstructure analyses were carried out using Zeiss 962 and Zeiss 982 Gemini scanning electron microscope operating at 25 kV. The samples were cut parallel and/or perpendicular to the loading axis and previously polished mechanically up to a mirror finish. The different scales of γ' precipitation were revealed by selective dissolution of the γ' phase using a triacid etching solution of 66% HCl, 17% HNO_3 and 17% CH_3COOH at room temperature. Image processing was performed using Visilog® software which enhances the image features allowing better determination of different precipitates sizes and their volume fractions [26].

Further fine microstructure analyses were conducted using a Philips CM20 transmission electron microscope operating at 200 kV. Discs of 3 mm diameter were cut from the gauge length of the tested specimens near the fracture surface by means of electric discharge machine. The thickness of the samples was reduced to below 70 μm by mechanical grinding. The electron transparent foils for TEM were then prepared by twinjet polisher using a solution made by 5% perchloric acid, 35% glycerol in methanol. The polishing conditions were −20 °C and 30 V.

2.3. Mechanical testing

The principal challenge facing us consists of reproducing in the laboratory the brutal temperature rise that occurs when only one aircraft engine is operating. This means that the very high temperature as well as the heating and cooling rates during mechanical tests have to be well controlled to make sure that the experiments fulfil the one engine inoperative conditions [19,20]. To reach this goal, induction heating was chosen and a Labview® program was developed to control the currents generated by the inductor. The input signal waveform was chosen to allow monitoring the temperature accurately during overheating. An additional proportional integral derivative loop was introduced to avoid any temperature overshoot during the simulation of the temperature jump which could induce dramatic effects on the mechanical response of the material [28].

To keep an access to the tested specimens during mechanical testing, an open inductor was used. This allows measuring the strain by using a contact extensometer. In parallel, to ensure a homogeneous temperature field in the specimen gauge, the penetration depth of the magnetic field produced by the induction, was taken into account. Assuming that the resistivity of MC2 alloy is the same as that of CMSX-4 one determined previously as a function of temperature by Roebuck et al. [24], we found a resistivity of $\rho_{1473\,K} = 1630\,\mathrm{n\Omega{\cdot}m}$ at 1200 °C. This means that the penetration depth relative to our test conditions (frequency is 100 Hz) is equal to 2 mm. This value corresponds to the specimen thickness. So, one may conclude that the compensation of induced currents may happen and disturb the heating. However, using Eq. (1), we find that for a thermal diffusivity α equal to $4.6 \times 10^{-6}\,\mathrm{m^2\,s^{-1}}$ and a thickness equal to 2 mm, the time needed to homogenize the temperature by thermal conduction is 0.2 s. This value is lower than the temperature rise up from 1050 °C to 1200 °C during the overheating which lasts for 5 s.

$$t_{\text{homogenization}} = \frac{(e/2)}{\alpha} \qquad (1)$$

An example of the evolution of the temperature measured using a spot welded S thermocouple in the centre of the gauge length is provided in Fig. 2 for an overheating lasting 180 s. It can be seen that the heating rate is in agreement with the one engine inoperative conditions and the cooling rate is 17 °C/s from 1200 °C to 800 °C,