ANISOTHERMAL HIGH-TEMPERATURE CYCLIC BEHAVIOR OF A Ni-BASED SINGLE CRYSTAL SUPERALLOY

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Abstract

Dwell-fatigue tests and variable strain rate tensile tests followed by cycling tests were performed using $\langle 001 \rangle$ -oriented specimens made of a first-generation Ni-based single crystal superalloy. A short thermal jump from the nominal temperature of 1050°C to 1200°C was introduced along the lifetime of dwell-fatigue experiments and at the beginning of tensile tests. Once such thermal loading was applied, a fine γ' precipitation occurred in the γ matrix and induced a large transient strengthening effect on the mechanical properties. Indeed, a transient decrease of the plastic strain rate, corresponding to a reduced magnitude in the hysteresis loops, was measured due to the temperature peak during the dwell-fatigue experiments. In addition, a temperature peak produced a large hardening effect during the tensile tests since a hardening of 160 MPa was created. These transient phenomena are due to a temporary additional strengthening provided by fine γ' precipitates lasting for the time necessary for their dissolution.

Introduction

The development and exploitation of gas-turbine blades cast from nickel-base superalloys in single crystal form has been one of the most successful industrial and commercial ventures relating to advanced structural materials over the last twenty years. These components are used in the hot section of the aero-engine and land-based gas turbines and are therefore subjected to anisothermal high-temperature dwell-fatigue deformations either during in-service operations or severe certification procedures. Such extreme thermo-mechanical environment leads to phase transformations as well as stress and microstructure gradients which dramatically alter mechanical properties. Besides this, during in-service operation of twin engine helicopters, one of the two engines may unexpectedly stop. This implies a brutal temperature rise of up to around 1200°C in the high pressure turbine blades for the engine still operating. Such an event is now included in the certification procedures of turboshaft engines and is known as One Engine Inoperative (OEI) rating [1, 2]. Consequently, creep or dwell-fatigue tests under isothermal conditions become insufficient to tackle the characterization of the mechanical properties in non-isothermal loading conditions. Better predictions of the mechanical behavior under these conditions are therefore required to increase engine efficiency and lower maintenance costs.

The high creep resistance of Ni-base single crystal superalloys is induced by the precipitation of a high volume fraction (close to 70 %) of the long-range ordered L1₂ γ' phase which appears as cubes coherently embedded in a face-centered cubic (fcc) solid solution γ matrix [3] (see Figure 1(a)) and evolves into platelets by directional coarsening during high temperature mechanical testing ($T > 850^\circ\text{C}$) [4]. This volume fraction is modified during thermal

changes above $\sim 850^\circ\text{C}$, which has a tremendous effect on the creep life [5], and depends on the temperature, the hold time and the applied load [6]. It was also recently noticed that an overheating at 1200 °C for 180 seconds performed on $\langle 111 \rangle$ oriented specimens can induce a transient behavior during creep-fatigue experiments by introducing fine γ' precipitates within the γ matrix [7] (see Figure 1(b)). Nevertheless, this transient behavior was never observed during non-isothermal creep loading on $\langle 001 \rangle$ oriented samples [8]. The objective of this work is then to elucidate the role played by the fine γ' precipitates during non-isothermal cyclic loading when the viscoplastic behavior of Ni-base single crystal superalloys is out of equilibrium.

Experimental Procedure

Material

Experiments were performed using the first-generation Ni-based single-crystal superalloy MC2. Its nominal composition is given in Table I. Experiments were carried out on specimens which had received the MC2 standard heat treatment (3h/1300°C/air-quenched (AQ) + 6h/1080°C/AQ + 20h/870°C/AQ) leading to a 0.4-0.5 μm average γ' precipitate size and to a $\approx 70\%$ precipitate volume fraction (a typical as-received microstructure is shown in Figure 3(a)) [9-11]. The testing specimens had a gage length of 14 mm with a prismatic section 6 mm wide and 2 mm thick and were oriented close to a $\langle 001 \rangle$ crystallographic orientation (deviation less than 5°). All samples were machined from MC2 rods from the same heat master and were heat treated simultaneously (solution+aging heat treatments).

Table I. Chemical composition of MC2 nickel base single crystal superalloy (in wt.%).

Ni	Cr	Co	Mo	W	Al	Ti	Ta
balance	8.0	5.0	2.0	7.8	5.0	1.5	6.1

Microstructural Characterization

Microstructural analyses were carried out using Zeiss 982 Gemini scanning electron microscope operating at 25 kV. The samples were cut parallel to the loading axis and mechanically polished up to a mirror finish. The γ' precipitates were revealed by selective dissolution of the γ' phase using a triacid etching solution of 66% HCl, 17% HNO₃ and 17% CH₃COOH 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 [12].

Figure 1. (a) As received microstructure of MC2 alloy highlighting a cuboidal γ' morphology. (b) Microstructure after an overheating of 180s at 1200°C without an applied stress on a as-received microstructure.

Mechanical Testing

The thermo-mechanical tests were performed using an hydraulic machine equipped with an induction-heating system. The temperature was controlled using K-thermocouples spot-welded in the specimen heads in order to not induce any sources of damage in the gauge length. In addition, a pyrometer recorded the temperature in the middle of the gauge length. A temperature accuracy of $\pm 2^\circ\text{C}$ was achieved with such a procedure [13]. Deformation was recorded and controlled using a 12-mm-gage-length axial extensometer with ceramic rods.

Dwell-fatigue experiments at 1050°C were performed using an asymmetric cycle, as shown in Figure 2. A thermal jump with no applied stress was introduced after a certain number of cycle set-up using the experimental results obtained from creep experiments [14]. The complex tensile test at 1050°C consists of starting with a variable strain rate at 10^{-3} s^{-1} up to 1% of deformation, 10^{-5} s^{-1} from 1 to 1.5%, 10^{-3} s^{-1} from 1.5 to 2% (see Figure 3), then followed by

Figure 2. Mechanical cycle used to perform the creep-fatigue experiments at 1050°C.

fifty mechanical cycles at 10^{-3} s^{-1} between 2 and 1% (see Figure 4). Contrary to the dwell-fatigue experiments, two thermal jump durations were examined, viz. 30 s and 150 s, in order to determine whether the influence of the fine γ' precipitates is dependent on a kinetics of dissolution.

Results

Complex Tensile Tests: Variable Strain Rate - Cycling

Variable strain rate tensile tests were performed at 1050°C. The mechanical behavior following an initial thermal jump of 30s and 150s is compared with the isothermal mechanical behavior (see Figure 3). It clearly reveals that the duration of the overheating does not matter since the two durations give the same results. However, a thermal jump before starting the tensile test strengthens the alloy by creating a large hardening of nearly 160 MPa followed by perfectly plastic behavior. It also seems that the effect of the thermal jump does not impact the viscosity since the decrease in the strain rate from 10^{-3} to 10^{-5} s^{-1} leads to the same magnitude drop, namely about 150 MPa. Finally, the last loading at 10^{-3} s^{-1} shows the disappearance of the hardening induced by a thermal jump since all conditions give the same mechanical response. However, the first cycle of the cyclic loading performed at 10^{-3} s^{-1} between 1 and 2% shows a difference between the isothermal and the thermal jump conditions (see Figure 4(a)). Indeed, the isothermal cycle reaches -183 MPa as a minimum stress whereas the cycles for 30s and 150s reach -217 and -209 MPa, respectively. This strengthened behavior vanishes by the fiftieth cycle, as shown in Figure 4(b). So, the strengthening effect brought about by an overheating is still effective after 535s (end of the first cycle) but disappears after 1,515s (end of the fiftieth cycle).

Dwell-Fatigue Experiments

Dwell-fatigue experiments were performed at 1050 °C and the mechanical cycle used is presented in Figure 2. A unique thermal jump of 30s at 1200°C without applied stress was introduced after 0.33 hour/12 cycles, 2.4 hours/87 cycles and 4.5 hours/162 cycles. These three conditions will be subsequently named TJ 12, TJ 87, and TJ 162, respectively. The introduction of such a temperature jump, whatever its position in the isothermal dwell-fatigue life of the alloy, always led to a decrease in the time to failure in comparison with the pure isothermal condition (see Figure 5(a)). Indeed, the isothermal lifetime is 15.7 hours/566 cycles whereas it is only 6.4 hours/218 cycles, 8.1 hours/292 cycles, and 14.0

Figure 3. Variable strain rate tensile tests. Two thermal jump durations were studied, namely 30s and 150s.

Figure 4. Strain controlled cyclic loading at 10^{-3} s^{-1} between 1 and 2% deformation performed after the variable strain rate tensile test shown in Figure 3(a) First cycle and (b) fiftieth cycle.

hours/506 cycles for TJ 12, TJ 87, and TJ 162, respectively. Furthermore, thermal jumps create a decrease in the plastic strain rate, as shown in Figure 5(b). As already discussed in the previous

section, this strengthening effect is transient and may last up to 1,100s in the case of a thermal jump introduced after 12 cycles. Thermal jumps also induce a decrease in the accumulated plastic strain since the starting strain after the overheating is lower than the one prior to it. This drop is in the order of 0.05% strain, as highlighted in Figure 5(c).

A fatigue-like representation allows us to extract additional information (see Figure 6). First, mechanical cycling creates a cyclic ratcheting effect already observed in [15], explaining the evolution of the accumulated plastic strain in Figure 6(a). The hysteresis loop opening ($\Delta\epsilon_p$) is decreased after a thermal jump (Figure 6). It is the reason of the reduction in the plastic strain rate: the ratcheting rate is decreased. Most importantly, the shape of the loops highlights a negative plastification in the compressive portion prior to the overheating (see Figure 6(c)). This mechanical response is temporarily modified after a thermal jump since no negative plasticity is recorded for 47, 82, and 88 cycles when an overheating is introduced after 12, 87, and 162 cycles, respectively.

Discussion

Creep vs. dwell-fatigue

Few studies have been done on the stress-controlled interactions between creep and fatigue in Ni-based single crystal superalloys [16-21]. Contrary to EI 698 VD alloy under repeated loading at 650 °C [16] and to AM1 alloy under alternate loading at 950 and 1100 °C [17], the lifetime of isothermal dwell-fatigue experiments is not bounded by creep and fatigue lives in a Wöhler representation (creep life being the lower bound). Indeed, dwell-fatigue is much more damaging than creep, as shown in Figure 7(a) for which the dwell-fatigue life (16h) is almost six times smaller than the creep life (93h). A similar result was obtained for polycrystalline

Figure 5. (a) Cyclic ratcheting during dwell-fatigue Creep experiments using the deformation recorded at maximum stress, i.e. 160 MPa. (b) Strain evolution during and after the thermal jump performed after 12 cycles showing a decrease of (b) the plastic strain rate and of (c) the plastic strain consequently to it.

Figure 6. Stress-strain loops for the dwell-fatigue experiments with a thermal jump after (a) 12 cycles and (b) 87 cycles. (c) and (d) are the same loops that presented in (a) and (b), respectively, but centered on the loops before and after the thermal jump. (e) Stress-strain loops for the creep-fatigue experiments with a thermal jump after 162 cycles.

Figure 7. Comparison of (a) the creep deformations and (b) the strain rate evolutions (b) for the isothermal dwell-fatigue and creep experiments [24] at 1050°C/160 MPa.

superalloys, viz. N18 [22] and Udimet 720 [23], and even on a single crystal at 950 °C [19]. The authors attributed this phenomenon to a modification of the deformation mechanisms, namely a progressive transition from shearing (pure fatigue) to by-passing (pure creep) while increasing the hold time. In addition, compression is an aggravating factor concerning damage, as already shown for the polycrystalline materials GH4049 [25] and N18 [22] and for the single crystal AM1 [26]. It is due to a lower yield stress in compression than in tension for most of the single crystal superalloys [27]. It implies a greater shearing of the γ' particles in compression which is considered to be a damage mechanism responsible of the tertiary creep stage, namely an increase in the plastic strain rate leading to failure [28-30]. Finally, cracking is faster in fatigue in a γ' -rafted microstructure perpendicular to the stress axis [31-33]. Indeed, once a crack is formed, it usually propagates along the γ -channels or the γ/γ' -interfaces [34, 35] and avoids cutting the γ' -phase for not forming highly energetic anti-phase boundaries (APB). Therefore, a microstructure formed during a dwell-fatigue test (Figure 8) with a positive mean stress is not appropriate for hindering crack propagation.

Compressive loading will not only bring a greater shearing of the γ' particles by the mobile dislocations, it will also allow pinned dislocations at γ/γ' interfaces to propagate again, as shown by Carroll *et al.* for the CMSX-4® at 950 and 1020°C while switching the load in creep from tension to compression [36]. This depinning phenomenon is highlighted when comparing the duration of the transient decrease of the plastic strain rate after a thermal jump while performing repeated and alternate loadings at the same temperature/resolved shear stress condition. Indeed, the duration of the decrease of the plastic strain rate during repeated fatigue tests on $\langle 111 \rangle$ -oriented specimens was 3,000s [7] whereas it was recorded to be about 1,000s in the present study (alternate loading on $\langle 001 \rangle$ -oriented specimens). For equivalent temperature/resolved shear stress conditions, the shorter the duration of the transient decrease in the plastic strain rate is, the easier the shearing of γ' precipitates is. Therefore, alternate dwell-fatigue loading leads to higher occurrence of γ' shearing and then causes a greater accumulation of the plastic strain compared to repeated loading. The difference in the mechanical behavior between creep and dwell-fatigue is also exposed in Figure 7(b). Indeed, contrary to creep experiencing a secondary creep stage between 0.4 and 1.5% strain, dwell-fatigue does not have such constant plastic strain rate regime but is in an accelerated plastic strain regime.

Effect of ultrafine γ' precipitates on the transient strengthening

The experimental results bring an insight into the effect of a short temperature peak at very high temperature ($T > 1150^\circ\text{C}$) on the cyclic response of $\langle 001 \rangle$ -oriented specimens made of Ni-based single crystal superalloys. In fact, thermal jump reduces the plastic strain rate for dwell-fatigue experiments. The same trend has been already observed along the $\langle 111 \rangle$ orientation [7]. This phenomenon was attributed to the interactions between the fine γ' precipitates within the γ channels and the mobile dislocations: γ' shearing is the main deformation mechanism for high resolved shear stress (RSS) whereas Orowan by-passing is for low RSS [37]. It was shown that a RSS of 62 MPa for a $\langle 111 \rangle$ orientation was necessary to trigger by-passing mechanisms. Therefore, a reasonable assumption was to consider that a dwell-fatigue experiment performed at a maximum of 151 MPa on $\langle 001 \rangle$ -oriented specimens (Schmidt factor of 0.41) would create the proper condition to measure decreased plastic strain rate consequently to a thermal jump. The transient hardening measured in Figure 3 and Figure 6 after a thermal jump is due to a transient increase of the apparent yield stress (see Figure 3) caused by the fine γ' particles precipitating in the γ channels and pinning dislocations for the time required for their dissolution [38, 39] or a size allowing γ' shearing [40]. Moreover, Kakehi also pointed out that the fine γ' precipitates play an important role in strengthening materials under creep at 700 °C/820 MPa by homogenizing strain, avoiding the creation of dislocation ribbons shearing the γ and γ' particles through $\langle 112 \rangle \{111\}$ -type glide, and modifying the dissociation mode of the γ -phase dislocations in Shockley partial [41]. That leads to increased interactions between slip systems and therefore more hardening. So, γ' tertiary precipitation has an important effect on the mechanical behavior just after a thermal jump event, as already shown in isothermal conditions [42-44]. From Figure 3, the hardening can come from either an increase of the isotropic hardening and/or of the kinematic hardening, more specifically from its kinetics since its magnitude is unchanged by referring to the isothermal result, i.e. each experiments reach 440 MPa at 1% deformation. However, Figure 4 proves that there is an effect of the plastic strain history on the kinematic hardening, as shown in [45, 46] for steel, which is not the case for the isotropic hardening.

High-temperature recovery process

A decrease in the plastic strain observed after an overheating event is attributed to dynamic recovery processes. It is consistent with

Figure 8. Microstructural observations every 2 mm from the rupture surface after (a) a creep-fatigue test at 1050°C/ $\sigma_{\max}$ =160 MPa (t_r =16h) an after (b) a creep tests at 1050°C/160 MPa (t_r =218h)

what was already measured during non-isothermal creep loading under synchrotron radiation [47, 48]. Indeed, high magnitude thermal jumps, viz. $\Delta T > 175$ °C, for temperatures above 950 °C foster dynamic recovery processes leading to a decrease of the perpendicular lattice misfit, i.e. of the internal stresses, when the temperature comes back to its initial value. The following question is therefore raised: why are single thermal jumps not beneficial since all the conditions are aligned to improve the lifetime? Quickly dissolving the γ' phase during temperature peaks from 60% to 30% in volume fraction releases the γ/γ' interfacial dislocations [49, 50] within the γ channels. The subsequent cooling creates a rapid growth of the γ/γ' interfaces which will therefore influence to the penetration of dislocations into the γ' phase [48]. This is commonly considered as a plastic mechanism responsible for the tertiary creep stage by increasing the plastic strain rate [51]. Thus, the damage induced by an overheating is essentially the increase of the plastic strain in the γ' phase ($\epsilon_p^{\gamma'}$).

Plasticity and precipitation

It is also of interest to notice that the longer the pre-cycling is, the shorter the decrease of the plastic strain rate is, despite a difference in the plastic strain: 0.3 % for the TJ 12 experiment and 0.96% for the TJ 162 experiment (Figure 5). However, it is untrue when the stress-strain curves are considered (Figure 6). In fact, the decrease in the magnitude of the plastic strain is dependent on the level of plastic strain reached while inserting the thermal jump. This dependency seems to reach an asymptote, as the one obtained by Giraud *et al.* for the CMSX-4® [6]. They showed that above a 0.33% of creep strain introduced at high temperature ($T > 900$ °C), the γ/γ' interfacial network is stabilized and there was no more effect of the plastic strain on the precipitation. Besides, the effect of dislocation network on diffusion processes was already reported in [4, 52]. Interfacial dislocation networks foster the dissolution of the γ' phase and γ -channel dislocations are preferred sites for precipitation [53]. Thus, the difference between the number of cycles for which a decrease in the magnitude of the plastic strain has been recorded (Figure 6), i.e. 47, 81 and 87 cycles respectively for TJ 12, 87, and 162, is due to the difference in the stabilization of dislocation networks.

Conclusion

Non-isothermal monotonic (complex tensile tests) and cyclic loading (dwell-fatigue) have been investigated in a first-generation Ni-base single crystal superalloy. Dwell-fatigue loading was recorded to be much more damaging than creep, especially because a compressive loading was applied which allowed a greater shearing of γ' precipitates and a depinning of dislocations. Furthermore, ultra-high thermal jumps created both recovery processes, leading to a decrease in the plastic strain, and ultrafine γ' precipitates nucleating upon cooling and strengthening the alloy by pinning the dislocations until the time necessary for their dissolution. From a modeling point of view, it was shown that fine γ' precipitation could modify both isotropic and kinematic hardening.

Fine γ' precipitation plays a key role in the non-isothermal mechanical behavior of Ni-based single crystal superalloys by modifying both monotonic and cyclic behaviors. Therefore, they have to be considered further in order to better predict the mechanical behavior and lifetime of structural components

subjected to such intricate and harsh thermomechanical loading paths.

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