THERMAL CYCLING CREEP RESISTANCE OF NI-BASED SINGLE CRYSTAL SUPERALLOYS

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Abstract

This paper reviews the non-isothermal creep resistance of Ni-based single crystal superalloys. For this, the creep resistance under very high temperature thermal cycling conditions of ten different $\langle 001 \rangle$ -oriented Ni-based single crystal superalloys from the first to the fourth generation has been investigated. Thermal cycling creep experiments consist of repeated short overheatings at 1105 °C and 1160 °C applied during a creep test at 1050 °C/120 MPa. Such thermal cycling conditions are representative of the most damaging stages of the certification procedures of small gas turbines in which creep is a life limiting factor for uncooled blades.

It is shown that the creep resistance under such non-isothermal creep conditions is primarily controlled by the γ' volume fraction remaining at high temperature, especially during the temperature spikes at 1160 °C. Indeed, higher γ' -solvus alloys exhibit better creep resistance under such thermal cycling conditions. It is also shown that increasing the rhenium content does not necessarily results in a better thermal cycling creep resistance and that having a good γ' morphological stability (i.e. slow γ' rafting kinetics and γ/γ' topological inversion) results in a better non-isothermal creep life. From the present experimental characterization, some guidelines for the development of non-isothermal creep resistant alloys are presented.

Background and motivations

Ni-based single crystal superalloys are nowadays widely used for the design and manufacturing of blades and vanes of the hottest sections of aero-engines and industrial gas turbines [1]. Several generations of such alloys have been developed to increase the temperature capability in terms of creep resistance, adding rhenium and/or ruthenium as main alloying elements [2-4]. One of the key stages for original engine makers in the development of a new gas turbine for aircraft or helicopter applications is to pass the certification procedures imposed by the airworthiness authorities (e.g., FAA in the USA or EASA in Europe). Such certification procedures are generally composed of various engine tests, including endurance tests and/or accelerated simulated mission engine tests (ASMET). As observed in Fig. 1, a very complex temperature and stress history is encountered during such a certification engine test. They are indeed designed to mimic the different regimes that can be encountered during a common flight (e.g. take off, cruise, landing), but also during emergency events leading to unusual regimes highlighted in Fig. 1 by arrows. Such cycles generally represent One Engine Inoperative (OEI) conditions encountered in the remaining operating engine(s) of aircraft or twin-engine helicopters [5]. These emergency conditions are critical to the blade material resistance since the metal temperature is very close to the γ' -solvus of the alloy in several sections, typically over 1150 °C [5-10]. These extreme

temperature exposures are short (typical OEI regimes are 30 or 150 seconds) but, as illustrated in Fig. 1 or in a previous paper from Mauget et al. [6], they can be repeated several times. Hence, characterizing the creep resistance under thermal cycling (noted as TC in this paper) conditions is a key issue to avoid any failure or catastrophic elongation of uncooled components (such as high pressure blades of low and medium power turboshaft engines for helicopters or thin walled blades of low pressure turbine stages of large turbofan engines) during the certification procedures.

Figure 1. Typical temperature/stress profile during an ASMET (Accelerated Simulated Mission Engine Test) certification procedure. Temperature peaks close to the γ' -solvus of the blade alloy are highlighted by arrows.

In the past, very few studies have been focused on the characterization of the non-isothermal creep resistance of Ni-based superalloys. Pioneering studies were undertaken at NACA (National Advisory Committee for Aeronautics, former name of the NASA) late in the 50's by Rowe and Freeman [11-15] and then in the 70's by Weiss et al. at Technion, Israël [16, 17]. These studies used equiaxed polycrystalline Ni-based alloys with low to moderate volume fractions of strengthening particles (e.g. Udimet 500 in the studies of Weiss et al.) and periodic temperature peaks were introduced in the creep life at a nominal temperature. As a main result of these studies, a faster creep strain rate under thermal cycling conditions was observed compared to that obtained at the nominal isothermal creep conditions. Moreover, it was shown that there was no possibility to calculate the thermal cycling creep life based on isothermal creep databases, since transient changes in the creep strain rate occurred during and just after each overtemperatures. Very first studies on the TC creep resistance of Ni-based single crystal superalloys (MC2 and CMSX-4® alloys) were performed later in France in two different research groups at Cirimat - ENSIACET, Toulouse [18-21] and Institut Pprime, ISAE-ENSMA, Poitiers [22-29], with a continuous support of SAFRAN-Turbomeca. While TC

experiments performed at Cirimat consist of periodic “underheatings” in the course of a very high temperature creep test (mostly at 1150 °C), the conditions studied at Institut Pprime consist of periodic overheatings introduced in the high temperature/low stress creep life (mostly at 1050 °C) of the alloys. Specific test benches were developed in both groups to perform such complex experiments. Burner rigs were even developed at Institut Pprime (such as the MAATRE burner) to simulate service conditions and to generate engine tests spectra [6, 9], like the one presented in Fig. 1. As a common result from all these studies, a faster strain rate and higher creep strain at the secondary to tertiary creep stages transition under TC conditions compared to pure isothermal conditions were observed [18, 19, 21, 23, 25, 28], even considering the highest temperature level during thermal cycling for Institut Pprime loading conditions [25]. This faster strain rate under non-isothermal conditions has been correlated to a faster degradation of the γ' structure (i.e. γ' -rafting) compared to pure isothermal conditions [18, 21] and to an easier penetration of dislocations inside γ' particles during cooling [22, 24]. Moreover, coarsened and N-type γ' -rafted microstructure have been shown to be less thermal cycling creep resistant in comparison to the classical γ - γ' cuboidal microstructure [23, 25, 28]. This can be explained both by a faster γ' dissolution [30] and again, by an easier penetration of dislocations inside the γ' -phase. However, no extensive study screening various alloy chemistries, has been undertaken up to now to investigate the key microstructural factor(s) controlling the non-isothermal creep properties. This is the main aim of this paper, whose additional final objective is to propose some guidelines to select/develop creep resistant alloys under such extreme conditions.

Experimental techniques

Materials and creep samples

In this study, the TC resistance of ten different Ni-based single crystal superalloys from the first to the fourth generation has been investigated. They were casted using a standard Bridgman process. Their chemical composition is listed in Table I. The newly developed CMSX-4® Plus alloy [31, 32] has been classified as a third generation alloy according to its chemistry (see Table I). Creep specimens were machined out by spark plasma erosion from bars or plates which were provided by SAFRAN-Snecma, SAFRAN-Turbomeca, GE Global Research and Cannon Muskegon Corporation. Before machining, each bar/plate had received the standard solution and aging heat treatments designed for aero-engine or industrial gas turbine applications (see Table II). These specific heat treatments formed a classical cuboidal γ/γ' microstructure with γ' precipitates having an average 0.4-0.55 μm edge length. Samples were machined close to a $\langle 001 \rangle$ crystallographic orientation, with a primary misorientation less than 5° with respect to the stress axis. Creep samples had a 14 mm gage length and a 4 mm gage diameter [28]. Before creep testing, they were low stress mechanically polished up to a 4000 SiC paper. Final polishing was performed parallel to the stress axis.

Creep testing

Tension creep tests have been performed using a radiant furnace, allowing fast heating/cooling rates. This creep test rig is equipped with a contactless extensometer to avoid any perturbation of the

temperature distribution of the specimens during temperature changes. The TC conditions chosen in this study are shown in Fig. 2. They consist of repeated one minute long overheatings at 1105 °C and 1160 °C introduced alternatively each 15 minutes in the creep life of the alloys at 1050 °C/120 MPa. Whatever the overheating temperature, heating and cooling rates were fixed at 2°C/s and 10°C/s, respectively. Each creep curve has been post-processed to remove variations in thermal and elastic strain resulting from thermal cycling. The interested reader is referred to [28] for additional experimental details. One to five TC creep experiments per alloy were performed. Isothermal creep tests were also performed at 1160 °C/120 MPa up to failure to better analyze the non-isothermal creep resistance of the alloys. To reduce scatter in experimental results, all the experiments (both isothermal and anisothermal ones) were performed using the same creep equipment.

Figure 2. Thermal cycle applied during TC creep experiments at 120 MPa.

Microstructure and physical properties characterization

The γ/γ' microstructure after TC creep experiments has been observed by scanning electron microscopy (SEM). After failure, longitudinal sections of the samples and subsequent mechanical polishing up to a mirror finish (1 μm diamond spray) were performed. The γ' microstructure was revealed by selective dissolution of the γ' phase by immersion in a solution made of 33% HNO₃ and 67% HCl (vol. pct), at 4 °C for 8 seconds. Observations were performed using the secondary electron imaging mode of a JEOL JSM® 7000F field emission gun SEM operating at 25 kV. Stereological characterizations were performed using the Visilog software and specific algorithms developed at Institut Pprime [33]. Both γ' area fraction, γ' particles average dimensions (length, thickness) and γ/γ' connectivity number [34] have been determined after testing up to failure in each specimen, using 5 pictures at least.

The γ' -solvus temperatures of each alloys presented in Table I were either extracted from the literature for Mar-M200 + Hf and CMSX®-10K [35, 36], from ONERA results for AM1, AM3 and MCNG [37, 38], from our own experiments for MC2 and CMSX-4® [25, 30, 39] and from DSC experiments performed by the Cannon Muskegon Corporation for CMSX-4® Plus alloy [32]. An example of such a determination using a DSC experiment is presented in Fig. 2.

Table I. Chemical Compositions of Alloys (Wt. %)

Alloy	Provider	Solvus (°C)	Density (g/cm ³)	Gen. ^o	Composition (wt %)												
					Ni	Co	Cr	Mo	W	Ta	Al	Ti	Re	Ru	Hf	Nb	Others
Mar-M200 + Hf	Snecma	1205 +/- 10	8.61	1	Bal.	9.5	8.6	/	11.8	/	4.9	1.9	/	/	1.58	0.86	0.13 C & 0.02 Fe
René N4	GE Global Research	1230 +/- 10	8.56			7.5	9.8	1.5	6.0	4.8	4.2	3.5	/	/	0.13	0.50	0.05 Si
AM1	Snecma	1268 +/- 5	8.59			6.5	7.8	2.0	5.7	7.9	5.2	1.1	/	/	/	/	/
AM3	Turbomeca	1264 +/- 2	8.25			5.5	8.0	2.2	5.0	3.5	6.0	2.0	/	/	/	/	/
MC2	Turbomeca	1265 +/- 5	8.63			5.0	8.0	2.0	8.0	6.0	5.0	1.5	/	/	/	/	/
René N5	GE Global Research	1270 +/- 5	8.63	2	Bal.	7.5	7.0	1.5	5.0	6.5	6.2	/	3.0	/	0.15	/	0.05 C & 0.01 Y
CMSX-4	Cannon Muskegon	1293 +/- 7	8.70			9.7	6.4	0.6	6.4	6.5	5.6	1.1	2.9	/	0.10	/	/
CMSX-4 Plus	Cannon Muskegon	1334 +/- 5	8.93	3	Bal.	10.0	3.5	0.6	6.0	8.0	5.7	0.9	4.8	/	0.10	/	/
CMSX-10K	Cannon Muskegon	1353 +/- 12	9.05			3.3	2.2	0.4	5.5	8.2	5.7	0.2	6.3	/	0.03	0.10	0.01 Si
MCNG	Turbomeca	1293 +/- 2	8.75	4	Bal.	/	4.0	1.0	5.0	5.0	6.0	0.5	4.0	4.0	0.10	/	0.1 Si

Table II. Heat Treatment Conditions of Alloys

Alloy	Solution Heat Treatment	Aging Treatment
Mar-M200+Hf	1210 °C/0.5 h + 1225 °C/0.5 h + 1240 °C/4 h/GFC	1100 °C/5 h/AQ + 870 °C/16 h/AQ
René N4	Fully Solutioned – Solution Heat Treatment Confidential to GE Global Research	1079 °C/4 h/AQ + 899 °C/4 h/AQ
AM1	1300 °C/3 h/AQ	1100 °C/5 h/AQ + 870 °C/16 h/AQ
AM3, MC2	1300 °C/3 h/AQ	1080 °C/6 h/AQ + 870 °C/16 h/AQ
René N5	Fully Solutioned – Solution Heat Treatment Confidential to GE Global Research	1120 °C/4 h/AQ + 1080 °C/6 h/AQ + 870 °C/16 h/AQ
CMSX-4®	1277 °C/2 h + 1288 °C/2 h + 1296 °C/3 h + 1304 °C/3 h + 1313 °C/2 h + 1316 °C/2 h + 1318 °C/2 h + 1321 °C/2 h/GFC	1140 °C/6 h/AQ + 871 °C/20 h/AQ
CMSX-4® Plus	1313 °C/2 h + 1318 °C/2 h + 1324 °C/6 h + 1335 °C/6 h/GFC	1163 °C/6 h/AQ + 871 °C/20 h/AQ
CMSX®-10K	1315 °C/1 h + 1329 °C/2 h + 1335 °C/2 h + 1340 °C/2 h + 1346 °C/2 h + 1352 °C/3 h + 1357 °C/3 h + 1360 °C/5 h + 1363 °C/10 h + 1365 °C/15 h/GFC	1152 °C/6 h/AQ + 871 °C/24 h/AQ
MCNG	1310 °C/3 h/heating at 3 °C/h + 1340 °C/4 h/GFC	1100 °C/4 h/AQ + 870 °C/16 h/AQ

*AQ = Air Quench, GFC = Gas Furnace Cooling

Figure 3. Derivative of the heat flow during a DSC experiment used to determine the γ' -solvus temperature of CMSX-4® Plus alloy on heating.

Experimental results

Isothermal creep behavior at very high temperature

The isothermal creep curves at 1160 °C/120 MPa for all alloys, except MCNG, are shown in Fig. 4. This creep testing condition has been chosen since temperature spikes at 1160 °C are introduced periodically in the creep life of the alloys at 1050 °C/120 MPa (see Fig. 2) and since it is the hottest conditions of the thermal cycle. It will hence generate most of the creep elongation and damage during TC creep experiments. As observed in Fig. 3, the creep curves of AM3, AM1, MC2, René N5, CMSX-4®, CMSX-4® Plus and CMSX®-10K are typical of

the γ' rafting regime as defined by Reed et al [4], with a very limited primary creep stage corresponding to the γ' rafting process, a well-defined secondary creep stage and a steep tertiary creep stage corresponding to creep strain localization [40]. Under these extreme creep conditions, it is also observed that Mar-M200 + Hf and René N4 exhibit very poor creep resistance, with creep lives of respectively 3 and 46 minutes and no clearly defined steady-state creep stage. Under this creep condition, the creep life of the stronger alloy (CMSX®-10K) is ~ 1500 times longer than the weakest one (Mar-M200 + Hf).

Thermal cycling creep behavior

Figure 5 compares the TC creep resistance of the alloys investigated in this study. For the sake of clarity, only one creep curve for each alloy has been kept to lighten this figure. For most of the alloys, except CMSX-4® Plus and CMSX®-10K, the creep curves are characterized by “steps” corresponding to each creep strain jump induced by one single overheating. These plastic strain jumps are especially observed for temperature peaks at 1160 °C and almost no strain jump is observed for overheatings at 1105 °C, except at the very end of the tests in the tertiary creep stages. This strain increment during temperature dwell at 1160 °C is spectacular for Mar-M200 + Hf alloy (see Fig. 5b, black curve) due to its very low creep resistance at 1160 °C/120 MPa (see Fig. 4b). All these observations are in good agreement with past studies [25, 28]. Contrary to all other alloys, almost no creep strain jumps are observed for CMSX-4® Plus and CMSX®-10K during most of their TC creep life (see Fig. 5), except at the very end of the test. This is clearly related to their very high creep strength at 1160 °C/120 MPa (Fig. 4) compared to the other first and second generation alloys.

Figure 4. Isothermal creep curves at 1160 °C/120 MPa in full time scale (a) and magnification on the first five hours of creep (b).

For this specific temperature history, a huge variation in creep life, by a factor of nearly 160 between Mar-M200 + Hf and CMSX®-10K was observed. This reduced difference in time to failure (or number of thermal cycles to failure) results from the fact that temperature overheatings at 1160°C only account for 3% of the thermal cycle (see Fig. 2) and creep damage can also develop during the lower temperature states of the tests. It has also to be noted that failure always occurred during an overheating at 1160 °C/120 MPa, whatever the material and sample. Hence, γ/γ' microstructure observations that will be presented in the next section will be comparable.

Microstructure observations

Microstructure observations were performed after failure in each alloy. Figure 6 shows selected SEM observations, both at low magnification to show the general features of the non-isothermal creep failure and at higher magnification to show the typical γ/γ' microstructure. Three alloys representative of a low (René N4), medium (CMSX-4®) and high TC creep strength (CMSX®-10K) alloy were chosen. As a common observation for Mar-M200 + Hf, AM3, MC2 and René N4, a very pronounced strain localization is observed close to failure. For CMSX-4®, CMSX-4®-Plus, MCNG and CMSX®-10K, the situation is quite different, with a more homogeneous elongation all along the gage length of each specimens (see upper pictures of Fig. 6 for CMSX-4® and CMSX®-10K). The creep failure always occurs by a coupled creep strain accumulation-cavitation process. It is observed qualitatively in Fig. 6 an increased fraction of opened pores close to failure, in the necking area of the samples, especially for René N4 and CMSX-4®.

Figure 5. Thermal cycling creep curves in full time/strain scales (a) and magnification on the first 40 hours/10 % of creep strain (b).

For the strongest alloys like CMSX-4® Plus and CMSX®-10K, smaller pores leading to crack initiation are evenly distributed within the gage volume. They are mainly nucleating close to the remaining eutectics as observed in Figure 7. Even if these alloys present the highest TC creep resistance, reducing the amount of remaining eutectics would probably further increase their creep resistance, by increasing the amount of “efficient” γ' particles and by retarding pore nucleation.

According to Fig. 6, a rafted γ' microstructure is observed in all alloys after failure. This lamellar structure is more regular far from the fracture surface (see third line of pictures in Fig. 6, corresponding to observations 5 mm away from the fracture) in comparison to 1 mm away from failure (see second line of pictures in Fig. 6). This results from the increase in stress triaxiality in the later stages of creep deformation when strain localization occurs [40]. It is also observed qualitatively in Fig. 6 an increase in γ' content from René N4 up to CMSX®-10K, as well as an increased number of connections between γ' rafts. Figures 7 and 8 provide quantitative evaluations of the variation in γ' area fraction and connectivity number in all alloy after failure. These stereological analyzes have been correlated to the number of thermal cycles up to failure in these figures and no clear alloy identification has been done in these plots in an aim to analyze in an objective way the microstructural factors controlling the TC creep resistance. It is here recalled that the connectivity number, which has been characterized using pictures taken at a 2,500X magnification, is defined as [34, 41]:

$$Na(\gamma') = (N_T - N_{TP})/2S \quad (1)$$

Figure 6. Microstructure observations at the end of thermal cycling creep tests for René N4, CMSX-4® and CMSX®-10K. Note that the γ' phase appears in dark in these pictures.

Figure 7. Example of cavitation and early stages of crack development close to a eutectic particle in CMSX®-10K.

In this equation, N_T and N_{TP} are respectively the number of terminations and number of triple points of the γ' skeleton and S is the area of the analyzed field [34].

According to Figure 8, it is clearly observed a better very high temperature TC creep durability with an increase of the γ' content. The average number of thermal cycles to failure is only 2 for a γ'

content of $\sim 15\%$ (case of Mar-M200 + Hf alloy) while it reaches 319 for a γ' content of $\sim 56\%$ (case of CMSX®-10K alloy). The increase in TC creep resistance is especially obvious for a γ' content greater than 45-50%.

Figure 8. Evolution of the number of thermal cycles up to failure as a function of the γ' area fraction measured in the gage section of each sample 1 mm far from the fracture surface.

The evolution of the TC creep resistance follows an opposite trend with the connectivity number. It is indeed observed that alloys having a negative connectivity number (i.e. alloys in which the γ/γ' topological inversion had occurred [34, 41], namely,

CMSX-4® Plus, CMSX®-10K and MCNG here) have a better TC creep resistance than alloys without γ/γ' topological inversion. This is a clear indication that under such non-isothermal conditions, the progressive microstructure degradation in the form of γ' rafting and subsequent γ' platelets interconnections is not the main controlling parameter for TC creep as it is commonly considered under isothermal conditions [34, 42].

Figure 9. Evolution of the number of thermal cycles up to failure as a function of the connectivity number measured in the gage section of each sample 1 mm far from the fracture surface.

Discussion

Analysis of the thermal cycling creep strength

According to the previous experimental results, it is observed that the TC creep resistance at very high temperatures of Ni-based single crystal superalloys is mainly controlled by the remaining γ' content at high temperatures (Fig. 8). Such an analysis can be performed objectively since all specimens failed during an overheating at 1160 °C. Even if failures did not occur exactly

after the same duration during the last overheating, the difference in γ' dissolution kinetics is negligible since it has been shown by Giraud et al. that the dissolution kinetics is only temperature sensitive once a given amount of high temperature creep deformation, (nearly 0.3% in CMSX-4®), has been achieved [30]. Hence, in that case, a very good correlation between the TC creep resistance and the γ' volume fraction at 1160 °C is observed. This correlation can also be translated in terms of γ' -solvus temperature. Indeed, it is observed in Fig. 10 a very good correlation between the durability in TC creep and the γ' -solvus temperature presented in Table I. It is also noticed that the ranking in alloy's creep resistance during isothermal creep at 1160 °C/120 MPa is almost the same as that under TC conditions, clearly indicating that most of the creep damage during non-isothermal conditions is generated by temperature jumps at 1160°C. All these results are in good agreement with past papers analysing microstructural factors contributing to the very high ($T > 1100^\circ\text{C}$) temperature creep resistance of Ni-based single crystal superalloys and showing that under such conditions, the remaining γ' fraction is the main creep life controlling parameter [4, 37, 43]. Indeed, keeping a high γ' content at high temperature efficiently impedes the dislocation motion at high temperature by increasing both the Orowan stress [23, 44] and the number of γ/γ' interfaces, hence lowering the dislocation climb velocity [45].

The development of a γ/γ' topological inversion during TC creep follows previous findings of Caron et al. under isothermal conditions [34]. They showed that this microstructure evolution can only occur if the γ' content is over 50%, what is observed experimentally in the present study for CMSX-4® Plus, CMSX®-10K (see Figs. 8 and 9). However, the interesting result from the present study is the apparent insensitivity of the thermal cycling creep life to this γ/γ' topological inversion at very high temperatures (Fig. 9). It is indeed observed that stronger alloys in TC creep exhibit this topological inversion while it is commonly considered as a severe microstructure degradation leading to creep strain acceleration at intermediate temperatures [34, 42].

Figure 10. Correlation of the thermal cycling creep strength with the γ' -solvus temperature.

Normalizing the time spent at 1160 °C/120 MPa during TC creep experiments by the isothermal creep resistance at this temperature, an interesting observation occurs. It is indeed observed in Fig. 11 that strong alloys such as CMSX-4®, CMSX®-10K and CMSX-4® Plus can resist a lower number of overheatings at 1160 °C relative to their isothermal creep resistance (between 5 to 8%) at this temperature compared to weak alloys like Mar-M200 + Hf or René N4 (nearly 60% and 40% respectively). This can be interpreted in the following way: the non-isothermal creep resistance of Mar-M200 + Hf under these conditions is mainly controlled by its intrinsic creep resistance at 1160 °C/120 MPa (i.e. its low γ' content) while for alloys such as CMSX-4® Plus and CMSX®-10K, the tests being longer, the microstructure degradation in the form of γ' rafting and γ/γ' topological inversion has enough time to take place (see Fig. 9). This γ/γ' degradation limits the number of thermal cycles the alloy can bear. Indeed, as shown by Steuer & al. and Giraud & al., the coarsening of the γ' particles and N-type γ' rafting are by far less TC creep resistant compared to a γ/γ' cuboidal microstructure [23, 25].

Figure 11. Fraction of time spent at 1160 °C/120 MPa during thermal cycling creep relative to the isothermal creep life plotted as a function of the isothermal creep life at 1160 °C/120MPa.

Guidelines for alloys development

According to the present analysis of the TC creep resistance, it is seen that two main guidelines have to be followed simultaneously to increase the intrinsic TC creep resistance of Ni-based single crystal superalloys:

- Stabilize the γ' phase at high temperature (i.e. maintain a high γ' volume fraction during overheatings).
- Limit the γ' degradation kinetics, such as γ' coarsening, N-type directional coarsening and γ/γ' topological inversion

Figure 12 shows Thermo-Calc simulations of the γ' volume fraction at thermodynamic equilibrium in the 1000 °C-1400 °C temperature range, using the TCNi8 database. Despite slight differences in γ' solvus temperature compared to experimental data presented in Table I for the six selected alloys presented in this figure, it is clearly observed that stronger alloys in TC creep at very high temperature have a higher γ' content over all the temperature domain encountered during the tests (case of CMSX-4® Plus and CMSX®-10K). It is pointed out that these simulations agree quite well with the fact that alloys having undergone γ/γ' topological inversion spend most of their time

during TC creep experiments with a γ' volume fraction over 50%. It is also observed that René N5, AM1 and MC2 have very similar evolutions of γ' content as a function of temperature in Fig. 12, in good agreement with their almost identical TC creep lives (see Fig. 10).

This first guideline, which is the most important one, can be reached by increasing the γ' -solvus temperature, adding, e.g. rhenium, tantalum and ruthenium. In the author's opinion, adding rhenium is not necessarily the most efficient solution to increase the TC creep resistance since it is observed in Figure 13 that alloys without any rhenium (e.g. AM1, AM3, MC2) can provide comparable non-isothermal creep lives when compared with second generation alloys such as René N5. Moreover, considering rotating components such as high pressure turbine blades, the density corrected properties have to be considered. Rhenium is known to increase the density of Ni-based alloys and, as an example, it has been observed that the intrinsic advantage in TC creep life of CMSX®-10K over CMSX-4® drops from a factor of 5.2 under same applied stress to a factor of 1.9 under same specific stress (not shown here). Finally, rhenium is also known to increase the γ/γ' lattice mismatch magnitude [37, 46], and hence, the γ' -rafting kinetics. Careful adjustments in chemistry by limiting rhenium to a reasonable level (i.e. 2 wt. % at maximum) and by increasing the tantalum content are hence recommended. Adding tantalum will also lead to an increase in the intrinsic γ' strength to dislocation cutting, as shown by Brundidge for Ta-modified René N5 [47].

Figure 12. Thermo-Calc simulations of the γ' volume fraction at thermodynamic equilibrium using the TCNi8 (Ni25) database.

Considering the possibilities to retard the γ' degradation kinetics, one has to adjust chemistry to reduce the lattice misfit magnitude, or to design positive lattice misfit alloys. As shown by Giraud et al. a P-type rafting is efficient in reducing the average creep strain rate under tension TC creep conditions without, however, increasing the creep life [25]. Co-based alloys could have been good candidates to develop such a vertical (P-type) γ' -rafting under high temperature creep loading and to reduce the dislocation climb kinetics [48]. However, it has to be admitted here that Co-based alloys have too low γ' -solvus temperatures to optimize TC creep resistance [49] and more generally, limiting the γ/γ' lattice mismatch magnitude is difficult to reach having in mind the increase in γ' -solvus temperature.

Probably, one of the most effective ways to improve both the stability of the γ' phase at high temperature (i.e. to keep a high γ'

content and to increase the γ' -solvus temperature), and the resistance to γ' coarsening would be additions of platinum. It has indeed been shown recently by Van Sluytman & al. that adding 2.5 at. % of platinum in a second generation alloy with 1.0 at. % of rhenium clearly reduce the coarsening rate of the γ' phase compared to classical Ni-based alloys [50]. Moreover, it has been shown in this interesting paper that platinum mainly partitions to the γ' phase and stabilize the γ' volume fraction at very high temperature compared to Ni-based superalloys. The γ' content remains almost constant up to 1200 °C, at a value of nearly 0.55, a value by far greater than all Ni-based alloys studied in the present paper (see Fig. 12). Hence, adding platinum up to a reasonable content (not to alter density), seems to be a promising way to stabilize the γ' phase at very high temperature and further improve TC creep resistance of Ni-based single crystal superalloys.

Figure 13. Thermal cycling creep resistance plotted as a function of the rhenium content.

Conclusions

The tension creep resistance under thermal cycling at very high temperature of ten different Ni-based single crystal superalloys has been investigated. Alloys from the first, second, third and fourth generations were studied. Thermal cycling creep experiments were composed of repeated short overheatings at 1105 °C and 1160 °C in the creep life at 1050 °C/120 MPa of the alloys. From the present study, the following main conclusions can be established:

- the creep resistance under such non-isothermal conditions is primarily controlled by the remaining γ' volume fraction at high temperature, especially during temperature spikes at 1160 °C.
- even if no direct correlation is observed between the thermal cycling creep durability and the microstructure degradation (i.e. N-type directional coarsening, γ/γ' topological inversion), it was observed that strong alloys during thermal cycling creep can survive less temperature overheatings at 1160 °C relative to their isothermal creep life at this temperature. This results from the longer test duration, allowing γ' -rafting and/or γ/γ' topological inversion to occur.

From the present study, some guidelines are proposed to develop alloys with higher thermal cycling creep resistance.

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