ON THE ROLE OF TERTIARY γ' PRECIPITATES IN THE CREEP BEHAVIOUR AT 700°C OF A PM DISK SUPERALLOY

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Abstract

The creep behaviour of the PM NR3 nickel-based superalloy has been studied at 700°C in a wide stress range as a function of its microstructure. It was first confirmed that at this critical temperature a coarse-grained microstructure was preferable to a fine-grained one to obtain the highest creep resistance. The role of the tertiary γ' phase precipitates in the creep behaviour of coarsegrained NR3 was investigated at two stress levels typical of both low stress and high stress creep regimes. Careful identification of dislocation mechanisms by transmission electron microscopy was performed in creep strained specimens in order to correlate the macroscopic behaviour with the creep controlling mechanisms. Prior elimination of the tertiary γ' precipitates using an adequate overageing heat treatment promotes easy glide of a/2<110> perfect matrix dislocations between the secondary γ' precipitates and therefore confers to the alloy a poor creep resistance. On the contrary the presence of fine tertiary γ ' particles impedes propagation of the perfect matrix dislocations which are forced to cut the γ' precipitates through two different modes of dissociation depending on the local precipitate distribution. Reduction of the γ phase channel width promotes the decorrelation in the matrix of the two a/6 < 112 partial dislocations constituting an a/2 < 110perfect dislocation. This mechanism which leads to stacking fault configurations extending through both γ and γ' phases evidences the strong resistance of the microstructure against creep deformation. Coupled analysis of the creep behaviour and of the dislocation structures in the low stress creep regime indicates a growing involvement of the grain boundary areas in the macroscopic creep deformation.

Introduction

Nickel-based superalloys processed by the powder metallurgy (PM) route are mainly used for high pressure compressor or turbine disk applications in gas turbine engines. The research objectives explored to improve disk superalloys are driven by the need for larger disks or higher temperature use. At present, most of the turbine disks are submitted to temperatures less than or equal to 650°C. In these conditions, tensile and fatigue strengths are the main criteria retained for the alloy design. But an increase of 50°C of the disk temperature would give a growing importance to the creep strength capability.

The creep behaviour of PM superalloys is greatly dependent on their microstructure which in turn is controlled by the thermomechanical parameters. Most of the studies in this field were devoted to the role of secondary γ' precipitates in the tensile or creep behaviours of these alloys. The effect of the γ' solution temperature [1-4] or the influence of the cooling rate [1-3] were thus frequently investigated. Several studies [1, 5] have shown that very fine secondary γ' precipitates are necessary for low deformation rate in disk superalloys. However, in the case of industrial heat treatments on full scale disks, the use of high cooling rates which ensures the precipitation of fine secondary γ' could be hazardous. After supersolvus solution treatment, rapid quenching can indeed lead to the ruin of the disk by cracking especially for high γ' volume fraction superalloys [6]. So, this type of superalloy (including NR3) must be quenched at a moderate cooling rate. This drawback could be partly compensated through use of an appropriate ageing treatment that essentially affects the tertiary γ' precipitates [4].

However, only few studies focused on the role of the fine tertiary γ' precipitates in the mechanical properties, and mainly on tensile [3] or compressive [5] behaviour. The present work was therefore performed to determine the optimal microstructure for creep resistance at 700°C and, more particularly, to analyse the role of tertiary γ' precipitates in the creep behaviour at 700°C of a high-temperature PM disk superalloy in a wide range of applied stress. A particular attention was paid to the creep deformation mechanisms identified by transmission electron microscopy.

Material

NR3 alloy is a superalloy developed at ONERA for high temperature disk applications (ONERA-SNECMA patent) [7]. Its chemistry (Table I) was defined in order to avoid the precipitation of harmful intergranular and intragranular topologically close-packed (TCP) phase particles which can occur during long-term exposures at high temperatures. The NR3 superalloy was thus selected for this study because of its excellent thermal stability (no TCP phase precipitation after 10,000 hours at 750°C) combined with a high creep resistance. The total volume fraction of strengthening γ' phase was estimated to be within the 0.50-0.55 range. The γ' solvus temperature and the density of NR3 were measured to be respectively 1205°C and 8.05 g.cm⁻³.

Table I. NR3 Alloy Composition (wt. %)

Ni	Со	Cr	Mo	Al	Ti	Hf	В	С	Zr
Bal.	14.65	11.8	3.3	3.65	5.5	0.33	0.013	0.024	0.052

The NR3 alloy was processed by Tecphy and Snecma Moteurs using the following PM route: vacuum induction melting, argon atomisation, powder seeving ($\phi \leq 75 \ \mu m$ (-200 mesh)), hot compaction, hot extrusion and isothermal forging. All the thermomechanical operations were conducted below the γ' solvus temperature.

Three forged pancakes ($\approx 200 \text{ mm}$ in diameter and $\approx 25 \text{ mm}$ in thickness) were supplied by Snecma Moteurs in the following states: i) subsolvus heat treated, ii) supersolvus heat treated, iii) as-forged. The heat treated pancakes were used for the preliminary study of the grain size influence on the creep behaviour of NR3, while the as-forged pancake was used for the detailed analysis of the creep behaviour of the coarse-grained material.

Influence of the Grain Size on the Creep Behaviour

Objective

A preliminary investigation was performed to check if a coarsegrained microstructure was absolutely necessary to achieve the best creep resistance at 700°C in a wide range of applied stress. Actually, it appears clearly that an increase of the grain size brings a substantial improvement of the creep resistance at temperature above 700°C [2, 8]. But at 650°C (under high stress), this effect is less clear or even inverse. So 700°C seems to be a critical temperature when selecting the microstructure suited for an optimal creep resistance. A comparison was thus made between fine-grained and coarse-grained microstructures, respectively obtained using either a subsolvus or a supersolvus heat treatment.

Experimental Procedures

Solution heat treatments were performed by Snecma Moteurs in a vacuum furnace on the whole pancakes. The conditions for the subsolvus and supersolvus heat treatments were respectively 1175°C for 4 hours and 1210°C for 2 hours. The subsequent cooling was performed at a rate of about 80-100°C.min⁻¹. Successive ageing heat treatments for 24 hours at 700°C, then 4 hours at 800°C, both followed by air cooling, were then applied to the pancakes. These ageing treatments are those applied to the N18 superalloy for high temperature applications [8].

Primary γ ' area fraction was determined by image analysis of pictures acquired by optical microscopy (OM). Microstructural observations and measurements of γ ' precipitate sizes were performed by transmission electron microscopy (TEM) using a JEOL 200 CX microscope operating at 200 kV. Thin foils were prepared by electrochemical polishing in a twin-jet polisher using a solution of 45% acetic acid, 45% butylcellosolve and 10% perchloric acid at 263 K and 25 V.

Constant load tensile creep tests were conducted in air at 700°C and 750°C on 3 mm diameter cylindrical specimens.

Experimental Results

<u>Microstructures.</u> The choice of a given heat treatment sequence affects not only the grain size but also the intragranular γ - γ' microstructure. The subsolvus heat treatment retains undissolved primary γ' particles which pin the grain boundaries and therefore limit the grain growth. This treatment results in a trimodal γ' particle size distribution. The supersolvus heat treatment eliminates all the primary γ' phase particles, allowing the grains to grow up to 50 μ m. Only two populations of γ' precipitates remain in this case. Typical distributions of secondary and tertiary γ' precipitates observed in fine-grained and coarse-grained materials are illustrated by the TEM images of Figure 1. The main

microstructural features determined on OM or TEM images are compared in Table II.

Table II. Effects of the Heat Treatments (HT) on the NR3 Microstructure

	Subsolvus HT	Supersolvus HT
Grain size (µm)	≈ 5	30-50
Primary γ' (area %)	20	0
Secondary γ' size (nm)	≈ 200	≈ 500
Tertiary γ' size (nm)	10-40	10-60



Figure 1. TEM dark field images of NR3 alloy: (a) fine-grained microstructure; (b) coarse-grained microstructure.

<u>Creep Behaviour</u>. Tensile creep tests were performed at 700°C in the 350-800 MPa range and at 750°C in the 300-600 MPa range. Due to the long durations of creep tests, especially at the lowest applied stresses, only a few tests were conducted to rupture. We have therefore determined the values of the minimum creep rate in order to compare the creep resistances of the fine-grained and coarse-grained materials. The experimental data reported in Figure 2 show that at 700°C the minimum creep rate is affected by the change of microstructure only for the lowest applied stress (350 MPa). The minimum creep rate of the supersolvus heat treated alloy is significantly lower than that of the subsolvus heat treated material. At 750°C the minimum creep rate of the finegrained alloy is higher than that of the coarse-grained material within all the investigated stress range. This difference in minimum creep rate increases when the applied stress decreases.



Figure 2. Minimum creep rate vs stress at 700°C and 750°C for fine-grained and coarse-grained NR3.

Analysis of the creep curves give complementary information about the effect of the microstructure. Thus, although the minimum creep rates were similar for the two microstructures at 700°C and 450 MPa, an earlier acceleration of the creep strain is observed in the case of the fine-grained alloy as compared with the coarse-grained one (Figure 3).



Figure 3. Creep curves at 700°C under 450 MPa as a function of the microstructure (interrupted tests).

Analysis of the whole set of creep curves shows that the difference in creep behaviour between the two materials becomes more pronounced as the temperature increases and as the applied stress decreases. According to these results, the coarse-grained material which exhibits a better creep resistance than the fine-grained one, even at 700°C, was retained for the continuation of the study.

Influence of Tertiary γ' Precipitates on Creep Behaviour at 700°C

Objective

A previous work performed on fine-grained NR3 evidenced the predominant role of the tertiary γ ' precipitates in its creep resistance at 700°C and 700 MPa [4]. An optimised size of tertiary γ ' particles obtained using a single step ageing heat treatment was identified. Similar results concerning the creep strength at 750°C were reported on subsolvus heat-treated wrought UDIMET 720 Li [3]. The main purpose of the present work was to extend the

analysis of the role of the tertiary γ' precipitates in the creep resistance of NR3 to the coarse-grained material and to the domain of low stresses and long creep durations. The initial γ - γ' microstructure was varied in order to check if in these conditions the presence of tertiary γ' particles was still necessary, and if the dependence of the creep behaviour on the size of these precipitates was comparable or not to that shown at high stress in fine-grained NR3. Dislocation structures have been analysed in crept specimens in order to link the macroscopic creep behaviour to the elementary deformation mechanisms. During exposure at 700°C, the size and distribution of the tertiary γ' precipitates is likely to evolve by solutioning/coarsening processes as shown by Wlodek et al. in both N18 and René 88 DT PM superalloys [9, 10]. A further objective of the present study is therefore to provide some keys to analyse the evolution of the creep rate as a function of time and consequently as a function of the characteristics of the tertiary γ' particle population.

Experimental Procedures

The heat treatment sequences were performed on cylindrical rods extracted by spark machining from the as-forged pancake in order to avoid any microstructural variation from one specimen to another one. The coarse-grained microstructure was obtained by applying the supersolvus solution heat treatment previously defined, i.e. 1210° C for 2 hours, followed by controlled cooling at a rate of 100° C.min⁻¹. Three different ageing sequences were then applied to produce various distributions of γ ' phase particles: HT1: 700° C/24h (Air Cooled) + 800° C/4h (AC) HT2: HT1 + 800° C/500h (AC) HT3: 700° C/24h (AC).

The mean secondary and tertiary γ' particle sizes were determined by the following procedure:

- TEM pictures
- Manual outline of each precipitate
- Analysis of edited image.

Data extracted from the image analysis procedure were the area of the secondary γ' precipitates and the diameter of the tertiary ones. Considering cube-shaped secondary γ' particles, their edge length was deduced from the area measurements. The mean distance between the tertiary γ' precipitates in the glide plane of the matrix dislocations was determined on TEM images of stacking faults, as it will be illustrated afterwards.

Tensile creep tests were conducted in air at 700° C on cylindrical specimens with a 3 mm gauge diameter with applied stresses of 500 and 650 MPa. Some tests were interrupted at about 0.2 % creep strain in order to prepare thin foils for TEM observations. These specimens were forced air cooled under stress.

Microstructure and deformation mechanism observations were performed by TEM using a JEOL 2010 microscope operating at 200 kV. Foils were cut normally to the tensile axis and then thinned down by electrochemical polishing in a twin-jet polisher using a STRUERS A3 solution at 263 K and 500 mA.



Figure 4. Effect of the ageing treatment on the γ ' phase precipitates in supersolvus heat treated NR3 (TEM dark field images): (a) HT1; (b) HT2; (c) HT3.

Experimental Results

<u>Microstructures</u>. The grain sizes resulting from the supersolvus heat treatment were within the 30-50 μ m range. The typical γ - γ' microstructures obtained through the different ageing sequences are illustrated by the TEM images of Figure 4. The corresponding microstructural characteristics measured in creep strained specimens are compared in Table III.

Table III. Microstructural Characteristics of Supersolvus Heat Treated NR3 after Creep at 700°C ($\sigma = 650$ MPa, $\epsilon = 0.2\%$)

Haat	Mean secondary	Mean tertiary	Mean γ	
treatment	γ' precipitate	γ' precipitate	channel	
	size (nm)	size (nm)	width (nm)	
HT1	313 ± 16	35 ± 4	52 ± 2	
HT2	405 ± 20	Absent	140 ± 14	
HT3	287 ± 15	27 ± 3	33 ± 1	

Addition of a long-term ageing for 500 hours at 800°C (HT2) to the standard heat treatment procedure (HT1) led to the total dissolution of the tertiary γ' precipitates and to a slight coarsening of the secondary ones. Application of a single ageing treatment (HT3) produced a smaller mean size of tertiary γ' precipitates as compared to HT1.

<u>Creep Properties.</u> As shown in Figure 2 for coarse-grained NR3, the stress-dependence at 700°C of the creep rate $\dot{\boldsymbol{\varepsilon}}$ can be described by the power law:

$$\dot{\varepsilon} = A\sigma^n \tag{1}$$

where σ is the applied stress, n the power coefficient, and A a constant [11, 12]. A high stress-dependence regime with a stress exponent n close to 13 was identified in the range 565-750 MPa. In the low-stress regime (350-500 MPa), the stress exponent was determined to be close to 6. The transition between these two stress-regimes occurs around 550 MPa. According to these results, tensile creep tests were conducted at 700°C in the two stress-dependence regimes: 650 MPa for the high-stress regime and 500 MPa for the low-stress one. Some tests were interrupted at 0.2 % creep strain to prepare thin foils for TEM observations.

At 650 MPa, the highest creep resistance was obtained with HT3, whereas HT2 conferred to the NR3 alloy the worst creep strength (Figure 5a). The 0.2 % creep strain was reached after 350 hours for HT3, after 140 hours for HT1 when only 7 hours were necessary for HT2.

At 500 MPa, the lowest creep strength was also obtained with the HT2 heat treatment (Figure 5b). HT1 and HT3 heat treatments led to quite similar creep behaviours, unlike that observed under 650 MPa. In spite of the technical difficulties of measurement inherent in the very low creep rates ($< 10^{-9}s^{-1}$), HT3 seems to lead to a slightly higher creep resistance than that obtained with HT1.



Figure 5. Interrupted creep test curves at 700°C of coarse-grained NR3 as a function of the ageing heat treatment procedure: (a) $\sigma = 650$ MPa; (b) $\sigma = 500$ MPa.

Deformation Mechanisms. In order to identify precisely the role of the tertiary γ' precipitates in the creep behaviour of NR3, *post mortem* dislocation analysis was performed by TEM on crept specimens. Whatever the initial microstructure, no significant differences in dislocation substructures were observed after creep at 650 MPa or 500 MPa.

In NR3 containing only secondary γ' precipitates (HT2), the deformation is homogeneously distributed within the grains: it results essentially from bypassing of the secondary γ' precipitates by a/2<110> matrix dislocations through combined glide and climb processes (Figure 6). Typical features resulting from this mechanism are dislocation loops around precipitates in superimposed {111} slip planes.

In the specimens containing a bimodal γ' distribution (HT1 and HT3), the deformation is rather heterogeneously distributed in a given grain (Figure 7). Many a/2<110> dislocations are concentrated near the grain boundaries (Figure 8). In these areas the dislocations generally circumvent the γ' particles but they sometimes cut the secondary and tertiary γ' precipitates as pairs connected by an antiphase boundary (APB) [13]. Within the bulk of the grain, <112>{111} slip operates in several glide planes leaving stacking faults in extended deformation bands (Figure 7).



Figure 6. Bypassing of the secondary γ' precipitates in HT2 NR3 after 0.2 % creep strain at 700°C and 500 MPa.



Figure 7. Dislocation structure in a grain of HT3 heat treated NR3 after 0.2% creep strain at 700°C and 650 MPa.



Figure 8. Perfect dislocations near a grain boundary in a grain of HT1 heat treated NR3 after 0.2% creep strain at 700°C and 650 MPa.

Careful analysis of the stacking fault configurations showed that two different shearing mechanisms involving Shockley partial dislocations are operative in HT1 and HT3 specimens. In the areas where the distances between the tertiary γ' precipitates are the largest, stacking faults are noticed only in the γ' particles (Figure 9). On the other hand when the interparticle distance locally decreases, stacking faults are observed extending within both γ and γ' phases (Figure 10).



Figure 9. Shearing configurations involving stacking faults in the (a) secondary or (b) secondary and tertiary γ' precipitates.

The first stacking fault configuration results from the dissociation of perfect a/2<110> matrix dislocations when they enter into the γ' precipitates. This dissociation mechanism was previously identified by Condat and Décamps in the AM1 single crystal superalloy after tensile creep deformation at 760°C [14]. It produces an a/3<112> superpartial which glides through the γ' phase creating a superlattice intrinsic stacking fault (SISF) and an a/6<112> partial which remains behind at the γ - γ' interface according to the following reaction:

$$\frac{a}{2}[110] \rightarrow \frac{a}{6}[\overline{1}1\overline{2}] + SISF + \frac{a}{3}[211]$$
(2)

When the a/3<112> dislocation has totally gone through the γ' particle, it recombines with the a/6<112> partial to form again a

a/2 < 110> dislocation, but leaving an a/6 < 112> dislocation loop around the precipitate.



Figure 10. Shearing configurations involving stacking faults extending through γ matrix and (a) secondary or (b) secondary and tertiary γ' precipitates.

The mechanism producing stacking faults in both γ and γ' phases is more complex and has been recently identified by Décamps et al. [15] (Figure 11). The process starts with the decorrelation of the two a/6<112> Shockley partials of an individual a/2<110> dissociated matrix dislocation (Figure 11a). The leading partial bows between the γ' precipitates, leaving in the γ matrix an intrinsic stacking fault in its {111} slip plane. When this partial enters a γ' precipitate, it creates a complex fault (CF) combining an APB and a superlattice stacking fault [16] (Figure 11b). This complex planar defect has a greater energy than an APB and above all than a superlattice stacking fault. A new a/6<112> partial therefore nucleates from the entering one forming a superlattice extrinsic stacking fault and an a/3<112> superpartial (Figure 11b) according to the following reaction:

$$\frac{a}{6}[112] \rightarrow \frac{a}{6}[\overline{1}\ \overline{1}\ \overline{2}] + SESF + \frac{a}{3}[112]$$
(3)

The γ' precipitate is thus sheared by a a/3<112> dislocation with creation of a SESF leaving an a/6<112> dislocation loop around it (Figure 11c).

The two mechanisms were observed to operate simultaneously in a same grain. However their respective activity depends on the applied ageing heat treatment. The stacking fault configurations involving both γ and γ ' phases were thus observed more frequently within the HT3 heat treated material than in the HT1 one.



Figure 11. Model of γ ' precipitate shearing by a decorrelated Shockley partial of a single dissociated matrix dislocation [15].

Discussion

Analysis of the creep behaviour of coarse-grained NR3 at 700°C coupled with the identification of the elementary deformation mechanisms shows unambiguously the significant role of the population of tertiary γ' precipitates. Analysis of the dislocations in the crept specimens clearly demonstrates that the mobility of the a/2<110> matrix dislocations is highly dependent on the characteristics of these fine strengthening particles, that in turn affects the macroscopic creep strain rate.

The mean distance between the γ ' particles is the key factor which determines the mode of matrix dislocation propagation in the γ - γ ' microstructure. It influences in particular the value of the Orowan stress required to bow and therefore to propagate the dislocations between the γ ' precipitates. The Orowan stress τ_{or} can be simply defined by the following relation:

$$\tau_{\rm or} = 2T/bL \tag{4}$$

where b and T are respectively the Burgers vector and the line tension of the bowing dislocation and L is the channel width between the precipitates. Typical values of the Orowan stress calculated for an a/2 < 110> and a decorrelated a/6 < 112> dislocations are reported respectively in Tables IV and V. These calculations show that propagation of dislocations is easier for edge segments than for screw ones. Moreover, bowing of partial dislocations is favoured as compared with perfect ones.

Table IV. Orowan stress τ_{or} for a/2<110> matrix dislocations

Heat	γ channel	$\tau_{\rm or}$ (MPa)		
Treatment	width (nm)	Edge character	Screw character	
HT1	52	160	783	
HT2	140	60	291	
HT3	33	253	1234	

Table V. (Drowan	stress	τ_{or} for	decorrel	lated
a/6	<112> r	natrix	disloca	ations	

Heat	γ channel	$\tau_{\rm or}$ (MPa)		
Treatment	width (nm)	Edge character	Screw character	
HT1	52	4.8	270	
HT2	140	1.8	100	
HT3	33	7.5	426	

Measurements of the distance between tertiary γ' precipitates were performed on images similar to those shown in Figures 9b and 10b. This method gives a realistic view about the obstacles encountered by an a/2<110> or a/6<112> dislocation in its {111} glide plane. Corrections were performed on the measured values in order to take into account the foil incline. Depending on the specimen, between 70 and 230 measurements were realised for determination of the secondary γ' size, between 930 and 1020 for the tertiary γ' size and between 170 and 370 for evaluation of the distance between tertiary γ' particles.

The values of the line tension T were calculated for edge and screw segments of dislocations in the framework of anisotropic elasticity using the DISDI program of Douin [17]. The values of the elastic constants used for this calculation were extracted from the data published by Pollock et al. for the γ matrix of the CMSX-3 nickel-based superalloy [18]. They give a good estimation of the elastic constants in NR3 because they do not vary significantly from a nickel-based superalloy to another one. The values thus retained at 700°C were respectively C₁₁ = 210 GPa, C₁₂ = 142 GPa and C₄₄ = 101 GPa. The calculated values of the line tension are respectively $1.05 \cdot 10^{-9}$ J.m⁻¹ and $5.13 \cdot 10^{-9}$ J.m⁻¹ for the edge and screw segments of a/2<110> dislocations, and $1.82 \cdot 10^{-11}$ J.m⁻¹ and $1.03 \cdot 10^{-9}$ J.m⁻¹ for the edge and screw segments of decorrelated a/6<112> dislocations. The moduli of the a/2<110> and a/6<112> dislocations were taken as b = 0.254 nm and 0.146 nm.

To propagate a matrix dislocation between γ ' precipitates, the resolved shear stress acting on it must be higher than the local Orowan stress. By taking the Schmidt Factor (SF) value of 0.5 for the most highly stressed <110>{111} or <112>{111} slip systems, the maximum resolved shear stress on a matrix dislocation is respectively 325 MPa and 250 MPa during the high stress regime and low stress regime creep tests.

In the overaged HT2 specimens where tertiary γ' particles have been completely eliminated, the Orowan stress for both edge and screw segments of a/2<110> dislocations is less than the resolved shear stress whatever the investigated creep regime. Propagation of a/2<110> dislocations between the secondary γ' precipitates is consequently quite easy that explains the corresponding dislocation structures and the poor creep resistance compared to specimens containing tertiary γ' particles.

Propagation of a/2 < 110 > dislocations in the grains becomes much more difficult in the HT1 and HT3 specimens due to the presence of the tertiary γ' precipitates. It is not the case near the grain boundaries due to the high local compatibility stresses existing between the adjacent misoriented grains. In these areas, few cutting events of γ' precipitates by pairs of a/2 < 110 > dislocations were observed whatever the size of the tertiary γ' particles. Within the bulk of the grains in HT1 and HT3 specimens the mobility of the screw parts of the perfect matrix dislocations in the γ channels is strongly limited as the corresponding value of the Orowan stress is much higher than the maximum value of the resolved shear stress, and even if cross-slip can sometimes aid the dislocations to bypass the precipitates. The mobility of the edge segments must be higher but only in the grains favourably oriented in respect to the tensile axis where the resolved shear stress overcomes the local Orowan stress. Anyway the mobility of the entire dislocation lines is strongly limited which does not allow the a/2<110> dislocations to spread in the γ channels over long distances.

In the areas where the γ channels are not too small, the a/2<110> dislocations can however bow on short distances between the precipitates. To further propagate they must however enter into the γ' particles where they dissociate according to the reaction (2) with creation of SISF only in the γ' phase (Figure 9).

In the regions where the γ channel are the narrowest, bowing of a/2<110> dislocations in the matrix becomes practically impossible. Data reported in Table V show that the Orowan stress values calculated for a single decorrelated a/6<112> partial are significantly lower than those estimated for the perfect matrix dislocations, mainly due to their better flexibility but also to their shorter Burgers vector modulus. That explains the decorrelation of the two partials constituting an a/2<110> perfect dislocation, although that creates an extended SISF in the matrix. In this case, the total energy needed for the decorrelation process and the creation of the SISF must indeed be lower than the energy required to move an a/2<110> dislocation. Propagation of the leading decorrelated a/6<112> dislocations in the matrix, then their dissociation in γ' precipitates according to the reaction (3) produce stacking fault bands involving the both phases.

As the distance between γ' precipitates is on average smaller in HT3 than in HT1 specimens, the frequency of stacking fault configurations involving both γ and γ' phases must be consequently higher, that is actually observed within both low stress and high stress creep regimes. The more frequent occurrence of this deformation mechanism in the HT3 specimens is a strong indication of the difficulty for dislocations to move through the corresponding γ - γ' microstructure in agreement with the resulting highest creep resistance.

Stacking fault configurations involving both γ and γ' phases were previously observed in PM superalloys. Bhowal et al. [1] examined dislocation substructure in PM subsolvus René 95 presenting a small spacing of the secondary γ' precipitates. These specimens were tensile creep tested at 650°C under high stress (850 and 965 MPa). Sinharoy et al. [5] observed stacking faults extending through several precipitates and including the matrix in a PM supersolvus KM4 specimen tensile creep tested at 650°C under 800 MPa. In these studies, the mechanism producing stacking faults in both γ and γ' phases was not precisely identified as Décamps et al. did in [15]. However, Bhowal et al. underline that this type of dislocation motion is restrictive in multiplication and in mean free path of the dislocations. Furthermore, Sinharoy et al. emphasize that the passage of a single partial dislocation leads to a very small deformation although the fault is extended. These comments are consistent with the low creep rate observed in the HT3 NR3 specimens.

In the low stress regime, the creep resistance sensitivity to the tertiary γ' distribution still exists but is significantly reduced as compared to the high stress regime. This feature coupled with the decrease of the power law stress exponent with the applied stress tends to prove that the strengthening effect due to the presence of tertiary γ' precipitates is so high that the main contribution to the macroscopic strain rate comes now from grain boundary area deformation rather than from grain deformation mechanisms. In the low stress regime, when these precipitates are present the resolved shear stress acting on the matrix dislocations is too weak to generate a significant creep deformation in the bulk of the grains whatever the size of these strengthening particles (at least for the γ' size range under consideration in this study). On the other hand grain boundary areas deform by a/2<110>{111} slip whatever the tertiary γ' size. It could be also suggested that grain boundary sliding may contribute to the overall deformation in the low stress range, but no experimental evidence of this mechanism has been shown until now.

Obviously the bulk of the grains still deforms by $a/2<110>\{111\}$ slip in the overaged HT2 material when the applied stress decreases due to the complete absence of tertiary γ' particles. The HT3 microstructure thus still shows the highest creep resistance in the low regime stress as compared to the two other microstructures.

Finally it can be pointed out that the sensitivity of the creep deformation mechanism to the distribution of the tertiary γ ' particles could be used to estimate the evolution of the population of these particles all the way through a creep test. By measuring the relative proportions of stacking fault configurations (2) and (3), it could give a good assessment of the ageing rate of the microstructure.

Conclusions

The role of the tertiary γ' precipitates in the creep behaviour at 700°C of coarse-grained PM NR3 superalloy was shown to be predominant within a wide stress range. Complete elimination of these precipitates by overageing promotes a high creep deformation rate as soon as the load is applied and therefore a poor creep resistance. TEM observations show that deformation operates by a/2<110>{111} slip with bypassing of the secondary γ' precipitates by the Orowan mechanism. On the other hand presence of fine tertiary γ' precipitates in the γ matrix between the coarser secondary γ ' particles impedes spreading of a/2<110> dislocations through the γ - γ ' microstructure. Depending on the ageing procedure, and therefore on the mean size and spacing of these particles, two types of stacking fault configurations have been observed in the crept specimens containing tertiary γ particles. The frequent occurrence of decorrelation of the two a/6<112> partials constituting an a/2<110> dislocation signals the difficulty for such a dislocation to bow between the precipitates in the specimens where the mean spacing of the tertiary γ' is the smallest. In this case, the creep resistance is the highest whatever the creep stress regime. The influence of the tertiary γ' precipitate distribution on the creep behaviour of coarse-grained NR3 decreases with the applied stress. That indicates a growing proportion of grain boundary area deformation in the creep strain rate of the material compared with the contribution of the deformation mechanisms within the grains.

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