MICROSTRUCTURAL BEHAVIOUR OF A SUPERALLOY UNDER REPEATED OR ALTERNATE L.C.F. AT HIGH TEMPERATURE

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Abstract

Total strain controlled Low Cycle Fatigue tests have been performed at 950°C (1223K) on the AM1 superalloy and its microstructural behaviour has been studied by Transmission Electron Microscopy. The mechanisms of deformation have been studied mainly on repeated fatigue $R_f=0$ but also in alternate one ($R_f=-1$) along the cubic [001] axis. In simple tension localisation of dislocations networks at the matrix/precipitate interface perpendicular to the <001> loading axis occurs. These dislocations are shown to always relax the misfit. In fatigue tests, the larger the total strain imposed is or the greater the number of cycles is, the more the deformation spreads out to the vertical channels. In small total strain, type N coarsening is observed as early as 200 cycles. For $R_f=-1$, compression is shown to deform the vertical channels without formation of networks. Alternate fatigue leads to type N coarsening for very few cycles.

Introduction

Industrial gas turbines blades in use undergo very complicated mixed effects of creep, fatigue and environment. The work presented here concerns mechanical fatigue which occurs more especially in the base of these blades when transitions between either normal runnings or idling of the engine occur. In fact, the fatigue experienced is of thermomechanical type ; but to get basic information, a test temperature of 950°C (1223K) was chosen. As the initiation and growth of cracks in fatigue has been the object of a certain amount of studies in the past, and as the knowledge of the behaviour of nickel based superalloys under fatigue before any cracks appears is rather limited ; the aim of this work was concentrated on the mechanisms of deformation during the damaging stage which is also extremely important since localisation of plastic deformation can condition the further stages. The type of fatigue tests and experimental conditions were chosen as close as possible to real conditions of the material in use : plastic low cycle fatigue, total strain imposed tests, temperature $T=1223K$ (950°C), frequency $f=0.25Hz$, loading along the turbine blades main axis : [001]. Indeed, most studies indexed in literature (cf. References 1 to 12) concern $R_f=-1$ fatigue (R_f is the ratio of the minimum strain to the maximum strain for each cycle). So, although most of our tests have been done at $R_f=0$, some tests with $R_f=-1$ have been performed in order to make comparisons and see the influence of a reversed loading.

Another part of the present work (already published : Décamps, Brien and Morton 1992, Reference 13) compared microstructures after strain-controlled fatigue tests and stress-controlled fatigue tests (same level of initial stress).

Experimental and Method

Mechanical tests have been performed using a tension hydraulic INSTRON machines on samples cut in AM1 superalloy provided by SNECMA (Table I gives the nominal composition of the alloy). Figure 1

presents the metallographic structure of the AM1 alloy. It is pointed out that the two phases composing the alloy are coherent despite a misfit of $\delta = -10^{-3}$ at the temperature of our fatigue tests (Royer 1995, Reference 14). Tests were carried out at 950°C (1223K) at 0.25Hz in air along the crystallographic axis [001]. For $R_f=0$ tests, the range of $\Delta\epsilon^t$ was 0.6-2.2%, the number of cycles varied from 1 to 2.10^4 . Some of the tests were continued to rupture.

Table I : Percentage composition in weight of the AM1 superalloy

Ni	Co	Cr	Al	W	Ta	Mo	Ti
Base	6.5	7.5	5.3	5.5	8	2	1.2

Figure 1 : Metallographic structure of the AM1 alloy

The γ precipitates are cuboids aligned along the cubic crystallographic directions and represent 65% of the total volume at 950°C.

The present work has been mainly concentrated on the $R_f=0$ fatigue and on exploration of the influence of the amplitude $\Delta\epsilon^t$ and of the number of cycles N on the structural mechanisms of deformation. Also, a compression test and two $R_f=-1$ fatigue tests have been carried out. The tables II and III (cf. end of article) index the mechanical details of the different tests carried out in this study. The compression test was performed at the same level of stress as the tension one done at $\Delta\epsilon^t=1.6\%$ in tension.

All observations of microstructures were made by Transmission Electron Microscopy either on a JEOL 2000 EX, a JEOL 200 CX or a PHILIPS CM20. Thin foils were thinned down first mechanically then by the classic two jets electrochemical method using a 90% acetic acid, 10% perchloric acid solution. Foils were fully orientated in order to follow the projection of the loading axis [001], noted p on the micrographs.

Results

$R_F=0$ fatigue

For $N=1$, $\Delta\epsilon^t=1.25\%$ the microstructure of deformation (that corresponds to a simple tension test) is very anisotropic. The anisotropy is characterised by a preferred localisation of the dislocations in the channels perpendicular to the $[001]$ axis (cf. figure 2) : γ/γ' $[001]$ interfaces are covered by polygonal networks of dislocations.

Figure 2 : Zone A microstructure. $N=1$, $\Delta\epsilon^t=1.25\%$. One can notice the preferential activation of the channels perpendicular to $[001]$ in comparison to the two other channels. Foil cut perpendicular to $[111]$. a/ Zone axis = $[001]$. b/ Zone axis between $[110]$ and $[010]$.

A careful study has been achieved in order to get the localisation of the extra plane associated to the either edge or 60° mixed dislocation localised at the γ/γ' interfaces.

This study requires the full orientation of the dislocations, the sign of the Burgers vector and the relative positions of the two phases. To get the first information, Bright field images with $g \cdot b = 2$ for each dislocation have been taken in order to get the asymmetrical contrast (Hirsh, Howie, Nicholson, Pashley and Whelan 1965 - Reference 15). FS/RH convention has been used (Bilby, Bullough and Smith 1955 - Reference 16) and Burgers vectors are determined by the classic extinction criterion (Howie and Whelan 1962 - Reference 17). The second information is obtained using stereomicroscopy. This study shows that all dislocations localised at the interfaces present an extra plane in the precipitates. So, they are on the right side of the interface to relax the existing misfit between γ and γ' . Dislocations at the interface form polygonal networks generally with 6 sides : they are classic honeycombs found in fcc materials ; interfacial networks of this type are encountered after creep of the same type of superalloys (Fredholm 1986 - Reference 18). Figure 3 presents an example of such a relaxing network.

3b

3c

Figure 3 : Network relaxing the interfacial misfit. $\Delta\epsilon^t=1.5\%$, $N=25$. Arrows indicate the double asymmetrical contrast. Foil parallel to $[111]$. a/ Zone axis = $[101]$, b/ Zone axis = $[111]$, c/ Schema showing the relative position of the two phases : cut seen from the left of photos.

When the number of cycles increases, an expansion of the deformation to the vertical channels is noted. However, the resulting microstructure still presents a strong partition of the plastic deformation regarding the horizontal and vertical channels with an obvious preference for horizontal ones (cf. figure 4). This homogeneity is noted as well if is very big, the bigger is, the sooner the homogenisation is observed (smaller N).

On the contrary, if the number of cycles N increases again, anisotropy is no longer observed : the microstructure of deformation is then completely homogeneous (cf. figure 5).

All the dislocations, either from one interface or the other have been shown to relax the misfit, except for large number of cycles, where some of the dislocations presented the other sign.

So, the deformation microstructures in many foils from each fatigue test from this map have been observed and two major domains were found (cf. map in figure 6). The first called H is a domain where the deformation microstructure is homogeneous, the second is a

domain, called A, gives rise to anisotropic deformation microstructure. This anisotropy is either micromechanical, or comes from the morphology of the precipitates (coarsening). Indeed, for smaller $\Delta\epsilon^t$ (like 0.6%) the dislocations never extend to the secondary channels and the microstructure always stays anisotropic. Moreover, for large number of cycles, $N > 200$, this micromechanical anisotropy results in oriented coarsening of type N (classical notation to describe coarsening perpendicular to the stress axis), i.e. exactly like in tension creep (cf. figure 7).

So, the anisotropy of the deformation microstructure is enhanced by small values of $\Delta\epsilon^t$: whereas large values of N increase the tendency for homogeneous microstructures (except for small $\Delta\epsilon^t$). These two rules and the localisation of the two domains are summarised in the previous map. The type N coarsening domain is noted as well, and the greater intensity of shadowing of the domain reflects the stronger tendency for coarsening.

Very rarely, shearing (except for big $\Delta\epsilon^t$ like 2.2% i.e. for big stresses) has been noted in microstructures, and all interfacial dislocations generally relax the γ/γ' misfit.

Figure 4 : Zone A microstructure. $\Delta\epsilon^t=1.5\%$, $N=25$. Foil parallel to $[111]$, Zone axis= $[101]$, deformation is localised essentially in channels perpendicular to the loading axis, forming networks at interfaces.

Figure 5 : Zone H microstructure : the 3 γ channels exhibit an equivalent density of dislocations. $N=1300$, $\Delta\epsilon^t=1.28\%$. Foil cut perpendicular to $[111]$. Zone axis = $[111]$.

Figure 6 : Map of microstructures of deformation in $R_f=0$ fatigue versus $\Delta\epsilon^t$ and N . This map is found to be divided in two main zones : zone A where the microstructures of deformation are anisotropic and zone H where the microstructures of deformation are homogeneous.

Figure 7 : Zone A microstructure. $N=200$, $\Delta\epsilon^t=0.63\%$. Type N oriented coarsening of the AM1 alloy under $R_f=0$ LCF for a great number of cycles at small $\Delta\epsilon^t$. Foil cut perpendicular to $[111]$. Zone axis = $[112]$.

$R_f=0$ fatigue discussion

To explain the present observation we need to consider the presence, evolution and distribution of internal stresses within the fatigued specimen.

The first one comes from the misfit between the γ phase and the γ' phase. This misfit $\delta = (a - a')/2a$ is equal to -10^{-3} at 950°C , and gives rise to compression (tension) stresses in the γ (γ') phase. a (a') is the γ (γ') phase parameter. These compression stresses are at the origin of the partition of the plastic deformation since the resulting local stresses in horizontal channels and in vertical ones are different. Tritley, the resulting stresses in the vertical channels are weaker than in horizontal ones since the applied and the misfit stresses are of opposite signs. This results in the localisation of the deformation in the horizontal channels.

This preferential localisation of deformation noted also by Benyoucef (1994 - Reference 19) after tension can be explained by the existence of the misfit. This argument had already been put forward by this writer.

After a while, since deformation occurs only in these channels, and since they have such a Burgers vector that they reduce the misfit, misfit stresses will be very low in horizontal channels. That phenomena destroying the source of the partition, added to the natural hardening due to the presence of dislocations themselves induces the extension of deformation to the vertical channels. This expansion is assisted at high stresses by shearing of γ precipitates.

A calculation of the number of pure edge dislocations needed to fully reduce the misfit at one interface can be done via the Brooks formula.

$$\frac{\sqrt{2} d_Y \cdot \delta}{|b|} = \frac{\sqrt{2} (400 \text{ to } 500) \cdot 10^{-3}}{\frac{\sqrt{2} \cdot 3,58 \cdot 10^{-1}}{2}} = 2 \text{ to } 3 \text{ dislocations}$$

where

d = length of the γ precipitates = 400 to 500 nm

δ = absolute value of the misfit between the two phases = 10^{-3}

b = Burgers vector of the considered dislocation : $a/2 [110]$

a = cubic lattice parameter of γ phase

So it means that in $R_E=0$ fatigue after a small number of cycles, the misfit stresses are relaxed, only in the channels perpendicular to $[001]$, since only $\{001\}$ interfaces are concerned by the location of dislocations.

One has to take into account a second type of stresses : a stress coming from the strongly composite structure of such a superalloy. We make use of the plasticity composite model of Mughrabi (1979 - Reference 20) used for inhomogeneous distribution of plastic deformation adopted by monophase metals after fatigue loadings. This idea has also been exploited for nickel base superalloys by different authors (Kuhn 1991-Mughrabi 1992-Véron 1995 - References 21 to 23).

The model has been considered here in the vertical direction (the horizontal effect has been neglected). One can show (to be published by Brien et al.) that these stresses called compatibility stresses find their existence in the presence of plastic deformation in vertical channels. Our calculation gives estimate of these stresses : they are found to equal 80% of $(\sigma_a - \sigma_0)$ where σ_0 is the elasticity limit of γ phase inside the superalloy (including Orowan stresses, stress coming from misfit and hardening stress coming from the solid solution) and σ_a is the applied stress. So, this type of stress doesn't exist in the beginning of the fatigue, but as soon as deformation occurs in vertical channels, compatibility stresses arise in vertical channels and delay them to deform. These second stresses assist the anisotropic partition of the plastic deformation.

Explanations of the type of coarsening observed can be explained by a recent coarsening model developed by Véron (Reference 23) for superalloys deformed under creep conditions. This model is based on the existence of gradients of elastic energy between the interfaces provoked by the anisotropic relaxation of the misfit stresses by dislocations.

These elastic energy gradients are of the same type as those observed by Véron after tension creep : $\{001\}$ interfaces are relaxed as $\{010\}$ and $\{100\}$ ones are not. Moreover the fact that at levels of stresses where the anisotropic micromechanical feature disappears rather early, no oriented coarsening is proves that the micromechanical anisotropy is really the needed condition for the type N oriented coarsening in repeated fatigue.

It is also worth noting, that some of $R_E=0$ tests exhibit bands of deformation, but that phenomena will be discussed separately.

$R_E=-1$ fatigue

The superalloy presents a peculiar microstructure after the $R_E=-1$ fatigue.

After one cycle (one tension followed by one compression), as well as deformation localized in interfaces perpendicular to $[001]$ due to one simple tension, the deformation localizes in channels parallel to $[001]$ (cf. figure 8). The dislocations observed in these vertical channels belong to only one slip system : here $1/2 \langle -10 \rangle \{1-11\}$, as a consequence can not form networks, and they are also found to be of the correct sign to relax the misfit between γ and γ' . A compression test has been

order to confirm that the new observation comes directly from the compression (and not from mixed effects of a compression following a tension). This compression test presents a microstructure where only the channels parallel to $[001]$ are affected by deformation : the dislocation found in each channel belong to only one slip system.

After cycling, here 113 cycles, the precipitates are coarsened in plates perpendicular to the loading axis. So, $R_E=-1$ fatigue produces very fast type N coarsening (cf. figure 9).

Figure 8 : $R_E=-1$ fatigue microstructure after 1 cycle. $N=1$, $\Delta\epsilon^p=2 \times 1.25\%$. Localisation of deformation in the channels containing the $[001]$ axis as well as in the perpendicular one is observed. Foil cut perpendicular to $[111]$. Zone axis = $[101]$.

Figure 9 : $R_E=-1$ fatigue microstructure after cycling 113 cycles. $N=113$, $\Delta\epsilon^p=2 \times 1.25\%$. All foils exhibit type N coarsening. Foil cut perpendicular to $[001]$. Zone axis = $[001]$.

$R_E=-1$ fatigue discussion

The localisation of the deformation in vertical channels can be explained on the base of exactly the same arguments put forward for $R_E=0$ fatigue behaviour. Indeed the internal misfit stresses in vertical channels added to the applied stress result in a much higher local stress in vertical channels than in horizontal ones.

A major difference between $R_E=0$ and $R_E=-1$ fatigue is the much higher efficiency of diffusion effects in alternate fatigue. Coarsening is observed much earlier for $R_E=-1$ fatigue : only 113 cycles ; the type of coarsening is of N type : exactly the same type as in repeated fatigue or in tension creep. So, alternate fatigue has a very strong effect on diffusion. Explanations of the type of fatigue observed can be explained by the coarsening model already mentioned. Two hypothesis coming from experiments observations are made : 1/ Simple tension (compression) localises the deformation only in horizontal (vertical) channels ; 2/ For each cycle, the previous phenomena applies and that the plastic deformation has the same absolute value for tension and compression. So, it means that twice as much volume of γ phase

is affected by deformation in compression than in tension, and after N cycles in alternate fatigue one horizontal channel contains globally twice as much deformation as a vertical channel. Moreover, as all the dislocations studied are found to relax the misfit: it means that the misfit in the horizontal channels will be reduced twice as quickly as that in the vertical channels: the elastic energy gradients then observed are of exactly the same type than in $R_E=0$ fatigue.

So, on the base of rather rough but reasonable hypothesis and reasoning, it is possible to explain the type N coarsening observed in alternate fatigue.

Conclusion

- A map of microstructures of deformation has been established in $R_E=0$ fatigue versus the number of cycles N and the total strain for the fatigue; different zones are pointed out on this map: an homogeneous H domain, an anisotropic A domain as well as a coarsening domain. These domains are accounted for on the base of the influence of internal stresses like misfit stresses and compatibility stresses coming from the strong composite structure of the superalloy. The interpretation of this map leads us to structural mechanisms of deformation which rule $R_E=0$ fatigue deformation.

- The behaviour of the AM1 superalloy is very different in $R_E=0$ fatigue and in $R_E=-1$ fatigue. We point out these differences and try to explain them.

- It is shown that alternate strain-controlled fatigue ($R_E=-1$) at 950°C (1223K) leads very quickly to a rafted structure perpendicular to the loading axis [001] (less than an half an hour) and that the type of coarsening is exactly the same as for creep tension tests. The alternation of applied stress is shown to have a very strong effect on the speed of diffusion.

- This work shows also that the observed type of coarsening in either $R_E=0$ or $R_E=-1$ fatigue can be explained using a recent coarsening model developed for superalloys deformed by creep.

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Table II : Mechanical details of the repeated LCF tests

$\Delta\epsilon$ tot. %	Cycle Total number of cycles	Maximal Stress in $\sigma_{\text{tens}}^{\text{max}}$ MPa	Maximal Stress in compression $\sigma_{\text{comp}}^{\text{max}}$ MPa	Plastic deformation per cycle %	Plastic cumulated deformation % (+/-)
0.63	1 stabilized cycle 200	458 415	9 43	0.01	2 (1)
0.70	1 1/2 lifetime 5630	608 428	- 178	0.04	200
0.79	1 1/2 lifetime 17117	663 438	- 212	0.06	1027
0.79	1 1/2 lifetime 21314	642 395	- 277	0.06	1279
1.09	1 1/2 lifetime 3345	815 447	- 453	0.12	401
1.27	1 1	739	-	0.153	0.153 (0.002)
1.33	1 25	661 637	143 157	0.247	0.47 (0.06)
1.25	1 25	732 632	57 151	0.16	2.10 (0.46)
1.26	1 1300	759 497	47 238	-	≤ 125 (15)
1.28	1 2 1300	785 764 451	50 67 357	-	125 (15)
1.6	1 5 25 200	704 663 611 520	39 76 123 205	0.80	160 (estimation)
1.6	1 5 25	707 670 620	56 81 126	0.80	20 (estimation)
2.2	1 2 3 4 5 200	975 950 930 920 910 610	85 110 126 140 150 470	1.10	100 (estimation) (33)

Table III : Mechanical details of the alternate LCF tests

$\Delta \epsilon_{tot}/2$ %	Cycle Total number of cycles	σ_{tens}^{max} Mpa	σ_{comp}^{max} Mpa	Plastic deformation per cycle %	Total cumulated plastic deformation per cycle %
1.26	1	932	952	(+)0.253 (-)0.449 (+) 0.111	0.81
1.26	1 Stabilized 113	960 874	887 887	0.77	80 to 90