FORMATION OF SERRATED GRAIN BOUNDARIES

AND THEIR EFFECT ON THE MECHANICAL PROPERTIES

IN A P/M NICKEL BASE SUPERALLOY

H. Loyer Danflou, M. Marty and A. Walder

Office National d'Etudes et de Recherches Aérospatiales (ONERA)
BP 72 - 92322 Châtillon Cedex - France

Abstract

To improve the crack propagation resistance of disk alloys, a study of the influence of the different heat treament parameters, which control the grain boundary geometry, was performed on Astroloy fabricated by a powder metallurgy process. Grain boundary serrations, associated with the precipitation of adjacent coarse γ' precipitates, were obtained by heat treating above the solvus for the alloy followed by slow cooling. The size of these coarse γ' precipitates can be controlled by the heat treatment parameters which include: homogenizing temperature, cooling rate and cooling range.The presence of grain boundary precipitates creates serrations by discontinuous precipitation of the γ' phase in the grain boundary region. The precipitates responsible for serrations were uniformly distributed on both sides of the boundaries. The amplitude of the serrations increase with the misorientation between grains. Tensile tests at 750°C showed no real difference between the microstructures, with and without serrations; however the creep fatigue crack growth rate in the alloy with serrated boundaries was found to be significantly slower than that observed in the unserrated material.

Superalloys 1992
Edited by S.D. Antolovich, R.W. Stusrud, R.A. MacKay,
D.L. Anton, T. Khan, R.D. Kissinger, D.L. Klarstrom
The Minerals, Metals & Materials Society, 1992

Introduction

Presently, the temperature capability of most turbine and compressor disc alloys having a fine grain structure is limited to about 650°C. However, in the quest for higher service temperatures where time dependent phenomena start playing a predominant role, it is necessary to increase the grain size of the material. In addition, the increase in temperature causes a severe interaction with the environment, leading to intergranular crack propagation in creep-fatigue conditions. The presence of grain boundary serrations in nickel base superalloys can lead to a lower rate of creep strain and crack development (1,2).

Grain boundary serrations have been studied not only in nickel base superalloys but also in steels and cobalt base alloys (2,3,4). Several investigations were performed to improve, through this particular grain boundary geometry, the crack propagation resistance of disc alloys, especially in INCO718 alloy (5). However, the major emphasis of these programs was on improving properties not understanding microstructure. The improvement in properties were generally small.

The mechanisms of the formation and growth of serrations are not clearly understood. The models to date are qualitative and discribe the phenomena by an interaction between the grain boundary and precipitates, in order to explain the modification in the grain-boundary geometry (3). The precipitates that are responsible for the serrations include the γ' phase in nickel base superalloys or $M_{23}C_6$ type carbides in austenitic stainless steels (6). From a more general point of view, it was observed that a heat treatment above the solvus temperature (supersolvus treatment), followed by a slow cooling at a carefully controlled rate, was necessary to generate the serrated grain boundary structure (4).

The present investigation was therefore undertaken to study the different parameters which might control the formation of serrated grain boundaries with the objective of obtaining the largest possible serrations. An attempt was made through detailed microstructural examinations to identify the mechanisms responsible for the formation and growth of serrations. Finally, mechanical properties of the most different microstructures were evaluated at 750°C by tensile tests and crack propagation tests in creep-fatigue conditions.

Experimental Techniques and Procedures

The alloy used in this study is Astroloy with the composition Ni-16.3Co-14.5Cr-4.35Al-3.65Ti-4.95Mo-0.018C-0.011B-0.047Zr (wt.%). It was prepared by a prealloyed powder metallurgy process using the following steps : melting of the alloy in vacuum (VIM process), atomizing by a rotating electrode process (mean size of powder particles was about 140 microns) and consolidating the powder by extrusion using an 8 : 1 extrusion ratio. The consolidated materials were then heat-treated above the γ' solvus at 1150°C (referred to as T_1) for 1.5 hours, then cooled, either at a mean rate of 100°C/min in order to obtain the usual grain-boundary morphology (smooth boundary), or at rates from less than 1°C/min to a few degrees /min between 1100°C and 1050°C (referred to as T_2) for generating serrated boundaries. It is important to note that the choice of T_2 temperature partly determines the amplitude of the serrations. The grain size observed after the supersolvus heat treatment is about 60-90 µm. A double ageing heat treatment (700°C/24h+800°C/4h) was finally applied to all the specimens. The microstructures were examined, both by optical and scanning electron microscopy on polished and etched samples and by transmission electron microscopy on thin foils.

Tensile and crack propagation tests in creep-fatigue conditions were conducted at 750°C in air on material with and without serrations. The crack propagation tests were made on samples 60x16x5 mm^3 with an edge notch machined by electrodischarge. A precrack was introduced at 650°C at a frequency of 20 Hz. The crack propagation test was conducted with a 10s-300s-

10s cycle with a 300 sec. dwell time at the maximum stress, for a loading ratio R=0.05. Crack length was determined by a D.C. potential drop technique.

Results

Microstructures

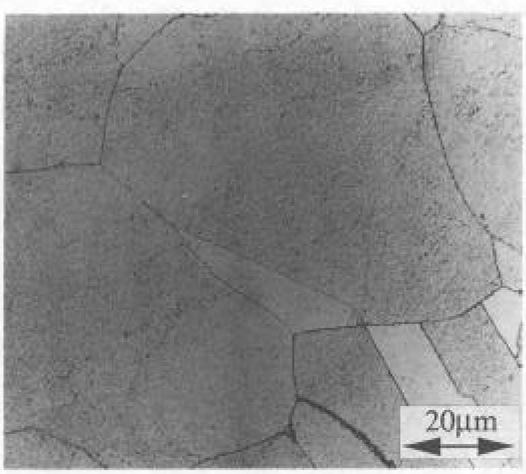
General description (a) Unserrated materials. The reference samples with "smooth" grain boundaries at an optical microscopy scale (Figure 1a) were obtained through the heat treatment conducted under conditions discribed above (cooling rate ≈ 100°C/min). However, at an electron microscopy scale, fine serrations were frequently observed and the size of these serrations was proportional to the size of the γ' precipitates located in contact with the grain boundary (several hundred nanometers). In the grain interior, these cuboidal γ' particles are slightly smaller ($150<\phi<250$nm) and are distribued throughout the material. Finally, there is also an extremely fine spherical γ' population ($5<\phi<10$nm) formed during quenching, which is distributed in the matrix channels between fine particles.

(b) Serrated materials (Figure 1b). The microstructure of the samples with serrated grain boundaries is characterized by four distinct populations of γ' precipitates, all of which have a cube/cube orientation relationship with the matrix :

- There are intragranular coarse particles ($\phi \approx$ several microns) designated as "butterflywings" because of their morphology (Figures 2a and 2c). Examinations carried out at various angles of observation indicate that these particles generally form a group of eight γ' cubes joining a central cube, but occasionally whithout the central cube (7). These particles result from a heterogeneous precipitation and grow preferentially along <111> direction ; this particular configuration of γ' particles originates from the lattice misfit between the two phases. These butterflywings appear simultaneously with the γ' particles precipitating near the grain boundaries and appear to act as obstacles to further grain boundary migration (Figure 2c).

- There are coarse γ' precipitates observed in contact with the grain boundaries (Figures 2a and 2b). These precipitates (1 to 6 microns in length) are generally associated with serrations. They extend along the grain boundaries, but are always associated with one of the two grains. Their preferential coarsening direction corresponds to the <111> direction on intragranular interface side, while there is no apparent preferential direction on the oposite (grain boundary) side.

(a)



(b)

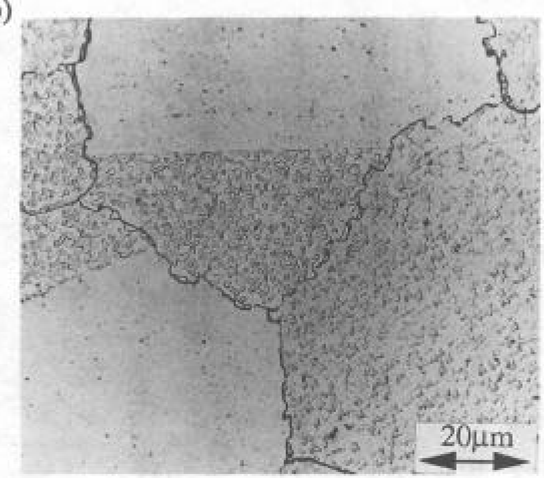


Figure 1 : Optical micrographs of "smooth" and serrated grain boundary samples :
a) "Smooth" grain boundaries and b) Serrated grain boundaries.

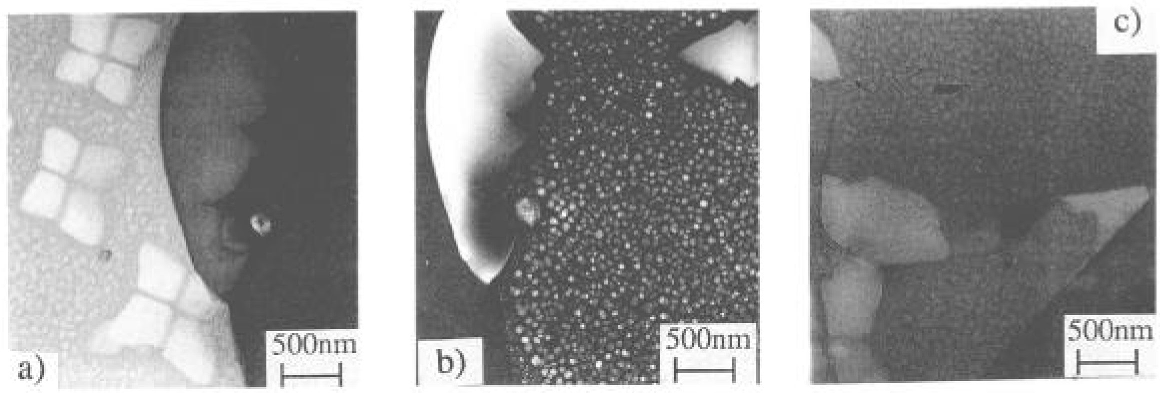


Figure 2 : TEM micrographs of serrated grain boundaries samples :
(a) and (c) dark bright field image and (b) dark field image.

- There are fine cuboidal intragranular γ' particles that are coherent with the matrix and are homogeneously distributed within the grain (Figure 2b), except in the vicinity of the coarse particles; in this case, a γ ' depleted zone is formed. The size of these fine particles ($70<\phi<170$nm) is significantly smaller than that observed in samples with "smooth" grain boundaries ($150<\phi<250$nm).

- Finally, there are ultrafine γ' particles ($5<\phi<20$nm) formed during quenching and are observed throughout the material, including the γ' depleted zone.

Relationship between γ' phase and serrations Each serration was associated with one but sometimes several precipitates of γ' adjacent to the grain boundaries (Figure 2). The average wavelength of serration was found to be directly related to the size of these precipitates located along grain boundaries (Figure 3). However, a correlation between the half-magnitude of serration and the thickness of coarse γ' particle was observed only during the early stages of serration growth. The variation found in the size of γ' particle for a given amplitude of serration could depend on how these irregularly shaped precipitates near the grain boundary are cut during specimen preparation and observed.

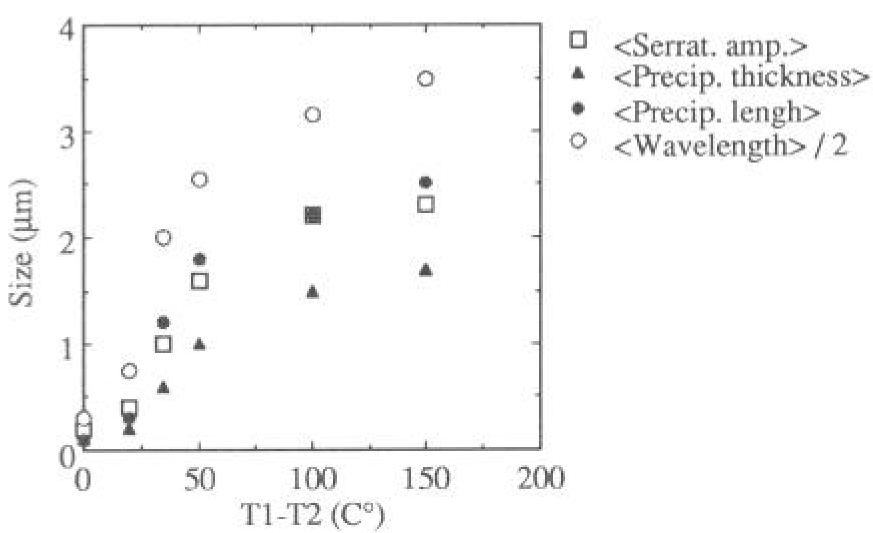


Figure 3 : Evolution of serration size with γ' size during furnace cooling.

Influence of different processing parameters For a better understanding of the mechanisms of formation of serrated boundaries, the influence of homogenizing temperatur, cooling rate through the solvus temperature and temperature range of cooling were examined (8).

- Homogenizing temperature (Table 1a). The homogenizing temperature affects both grain growth and the capability of boundary migration during the formation of serrations. A super-solvus heat treatment, which dissolves $M_{23}C_6$ carbides and γ' particles into solid solution so that the grain boundaries are free to move, appears to be a prerequisite for the development of large serrations. It should be noted that a "subsolvus" heat treatment restricts the growth of serrations to several hundred nanometers.

-Cooling rate (Table 1b). For a given temperature range of cooling (between T_1 and T_2), the cooling rate partly determines the amplitude of the serrations. It should be emphasized that lower cooling rates generally lead to a more uniform distribution of serrated boundaries throughout the material. Slow cooling promotes the development of coarse γ' precipitates. The amplitudes of the serrations observed in the present study varies from 500 nm to several microns for cooling rates corresponding to five and one degrees/min, respectively.

- Temperature range of controlled cooling (Table 1c). The average amplitude of serrations and the distribution of serrated boundaries are influenced by the temperature at the end of furnace cooling , T_2. The amplitude of the serrations can vary with the choice of T_2. Different T_2 temperatures lead to different growth rates of coarse precipitates. It was observed that this temperature should be about one hundred degrees below the solvus temperature in order to obtain the largeest possible amplitude of serrations. There was an increase in the observed coarsening kinetics of the grain boundary γ' at about 35 degrees below the solvus temperature. The grain boundary morphology changes from a fine serration associated with fine cuboidal precipitates to a large serration with coarse γ' particles.

Table 1 : Influence of heat-treatment parameters on the grain boundary morphology.

a) Effect of homogenisation temperature T_1 (cooling rate = 2°C/min; T_2 = 1100°C).

Homo. temp. (°C)	Grain size (µm)	Primary γ' size (µm)	Amplitude of serrations (µm)	Proportion of wavy boundaries
1130	30	0.5-1	0.2	0%
1140	>30	1-2	1-2	<20%
1150	60-90	1-2	<2-3	70%

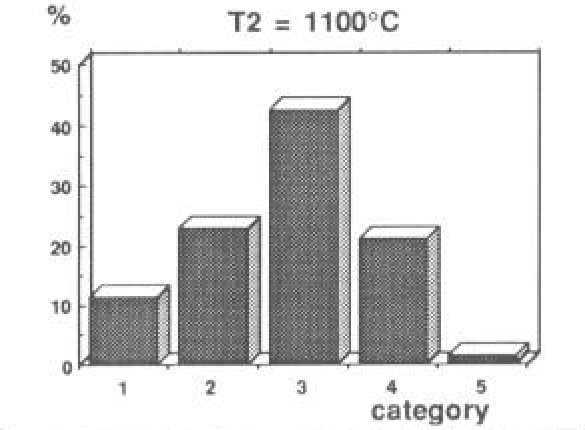
b) Effect of cooling rate (after supersolvus heat-treatment at 1150°C; T_2 = 1100°C).

Cooling rate (°C/min)	Primary γ' size (µm)	Amplitude of serration (µm)	Proportion of wavy bound.
0.5	1-3	2-3	70%
2	1-2	<2-3	70%
5	1-1.5	1-3	<50%
100	<0.2	<0.2	0%

c) Effect of temperature T2 of the end of slow-cooling (cooling rate = 2°C/min).

Temperature range of controlled cooling (°C)	Primary γ' size (µm)	Amplitude of serrations (µm)	Proportion of wavy boundaries
1150 -->1130	<0.5	0.2	0%
1150 -->1120	0.5	1-2	>50%
1150 -->1100	1-2	<2-3	70%
1150 -->1050	1-3	2-3	>80%

Remarks : The grain boundaries are considered as serrated grain boundaries, when their serration amplitudes are larger than 500 nm. The γ' particles examined correspond to the γ' precipitates situated in contact with the boundary.



Category	1	2	3	4	5
Amplitude (µm)	0 - 0.4	0.4 - 1	1 - 2	2 - 3	3 and +

Figure 4 : Example of the size distribution of serration amplitude.

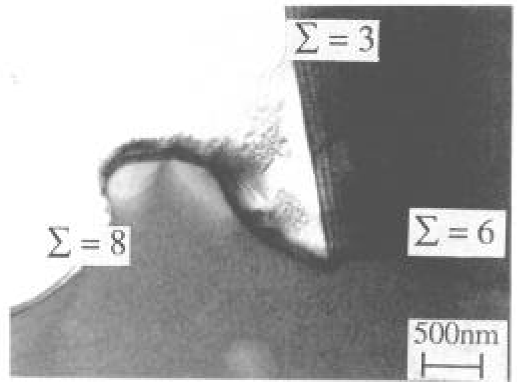


Figure 5 : Example of different boundaries behaviours in function of their coincidence.

Influence of the misorientation between two grains The study of γ ' growth kinetics in contact with the of grain boundary reveals a variation in the size of the amplitude of the serrations from one grain boundary to another (Figure 4). The amplitude of serrations remains uniform on a given grain boundary. Amplitude and wavelength of the serration become more uniform when the specimen is held at the temperature T_2.

In general, grain boundaries can be classified as: low angle boundaries(θ <15°), coincidence boundaries (Σ_i < 75) and random boundaries. To determine if there exists a correlation between the amplitude of serration and misorientation in Astroloy, a systematic examination of misorientation for different boundary morphologies was conducted using electron diffraction. A comparison of different grain boundaries was made by calculating the density of coincidence sites of the CLS (coincidence lattice site) theory (9). The coincidence parameter corresponds to the ratio of the volume of the coincidence lattice to the volume of the primitive cell of the lattice. The experimental results showed that serration of small amplitude were associated with high coinsidence boundaries ($\Sigma_i \leq 6$), while serrations of large amplitude with low coincidence boundaries or random boundaries (Figure 5). It is worth noting that, in most of the cases examined, twin boundaries which generally have a high lattice coincidence were free form serrations.

Mechanical properties

Table 2 shows the results of the tensile tests performed on the two materials (serrated and unserrated). These results indicate that the introduction of the serrated morphology does not modify the properties exept for a slight decrease in the elongation (from 26% to 21%) for the serrated material in spite of the presence of incoherent coarse γ' precipitates.

Figure 6 shows the da/dN versus ΔK behaviour for the two microstructures. The crack propagation rate observed in the serrated material is 3 to 5 times slower than that of the unserrated samples.

Table 2 : Results of the tensile tests at 750°C.

Samples	Grain size (µm)	Elongation (%)	0.2% yield strengh (Mpa)	Ultimate T.S. (Mpa)
Linear G.B.	65-90	26	903	1040
Serrated G.B.	65-90	21	895	1010

Examination of the crack propagation surface (Figure 6) showed a clear difference in the fracture surface morphology. In the unserrated material, cracks propagate intergranularly. Although the propagation mode remains essentially intergranular, some transgranular areas appear in the serrated material, with fatigue striations present on some of them. The crack propagation behaviour of the serrated material could not be unambigiously identified as yet but a proposal will be presented in a later section.

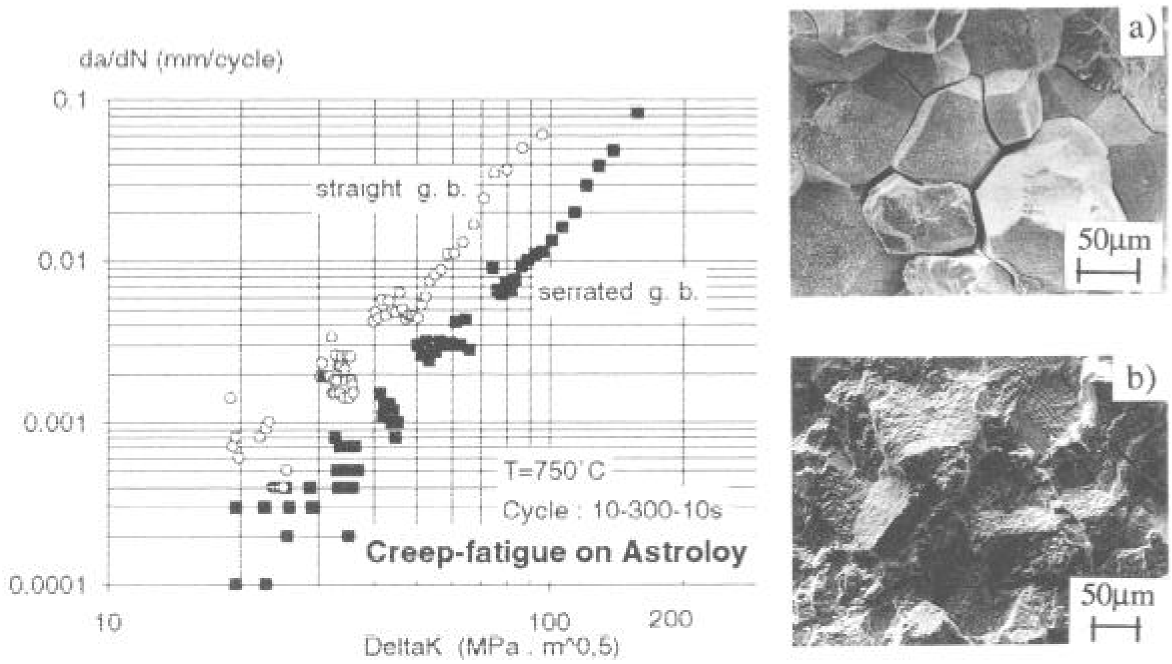


Figure 6 : Results of the creep-fatigue crack growth tests at 750°C in Astroloy; (a) fractography of "smooth" material, (b) fractography of serrated material.

Discussions

Formation of the serrated grain boundaries

Over the temperature range (T_1-T_2) for Astroloy, heterogeneous precipitation takes place in regions like grain boundaries through a classical nucleation and growth process associated with flow of solute both along grain boundaries and within the bulk. However, our observations suggest that some γ' particles dissolve either partially (cf. Figure 2c) or completely in contact with "moving" grain boundaries and such a dissolution contributes to the growth of the coarser, more stable γ' particles in the opposite grain. According to the litterature (10), the complete dissolution of γ' at the moving grain boundary, followed by reprecipitation behind it, can occur at high-angle grain boundaries during recristallization. Generally, the grain boundary is a short-circuit path permitting for the solute to reach more quickly an equilibrium state. Thus, the heterogeneous precipitation of the γ' particles accompanying the formation of the serrations suggests a discontinuous precipitation reaction where the moving grain boundary plays a role in establishing a more stable equilibrium state (11). In the present stage of our investigation, however, it is not totally clear if the formation of serrated grain boundaries is directly associated with a discontinuous reaction.

As regards the grain boundary migration necessary to form the serrations, let us first mention the work of Randle and Ralph (12) who studied the interactions of grain boundaries with γ' precipitates during grain growth. They observed an increase of grain boundary surface (wavy grain boundary morphology) in the situation where a very strong pinning of the grain boundary by γ' particles occurs during a by-pass attempt during recristallization. In this case, the relaxation of lattice coherency strain of the γ' precipitates in contact with grain boundaries leads to their coarsening.

A discussion of another possible migration scheme may be found in the work of Koul and Gessinger(3) who observed serrations in nickel base superalloys during slow coolings. They suggested that γ' nucleates on one side of the grain boundary and due to the presence of the "open" grain boundary in contact with the γ' particle, the coherency strains on the boundary side of the γ' particle are easily accomodated. And a net strain energy differencial between matrix side interface and grain boundary side interface provides a driving force to move the γ' particle in the direction normal to the grain boundary. This model seems to us unsatisfactory in that it does not take into account the evolution of the volume of precipitates. The economy achieved in coherency strain energy seems to be insufficient to provide a driving force for the migration of the γ' particle in view of the increase in grain boundary surface. It is however worth mentioning that the migration of γ' precipitates seems to occur in Astroloy (cf. Figure 2c).

According to Bauer who recently reviewed phenomena related to the grain boundary migration (13), random high-angle boundaries are characterized by a more "open" structure, thus facilitating transfer of atoms from one grain to another by short-circuit diffusion mechanism. Migration of low-angle boundaries is characterized by an activation energy nearly identical to that for volume diffusion, whereas migration of random high angle boundaries is controlled by direct atom transfer across the boundary. Moreover, high-angle boundaries migrate more easily than low-angle boundaries. This is consistent with the correlation established in the present study between serration amplitude and misorientation (or lattice coincidence). Indeed, in our case, the formation of serrations requires migration of the grain boundary and the variation observed in the amplitude of the serration from one grain boundary to another may correspond to different rates of grain boundary migration.

A preferential growth along <111> direction is observed for coarse γ' particles. According to Ricks et al. (14), for the case of high positive misfit, the anisotropy in elastic modulus involves the existence of regions with a minimum lattice compression favorable for growth. In the case of γ' particle in contact with the grain boundary, this preferential growth were observed principally on the matrix (intragranular interface) side of the precipitate. On the grain boundary side, however, such a preferential growth was sometimes observed only for high coincidence boundary. In the other cases, these precipitates are well rounded especially for random boundary, where lattice coherency strain can relax; this suggests that the particles can adopt the morphology which corresponds to the maximum volume for the minimum interface (rounded shape) on the grain boundary side.

Serrated grain boundaries and mechanical properties

As already presented in the Introduction, the elevated-temperature fatigue crack growth behaviour in Astroloy, when subjected to a low frequency, is considered to be a time dependent phenomenon affected by the envirronment. According to Chang (15), oxygen-embrittled materials show a damage zone ahead of the crack. The embrittlement is thermally activated, and the degree of the damage is a function of both the distance from the crack tip and the time for embrittlement. The crack can propagate at a much faster rate in the damage zone. Moreover, it has been demonstrated by Ghonem et al. (16) that oxygen diffusivity in grain boundaries increases with both the stress intensity factor range ΔK and the reduction in the frequency parameter. When frequency increases (frequencies >1 Hz), crack propagation mode becomes transgranular. This can explain why the change of grain boundary morphology does not influence the propagation resistance at high frequency (17). Therefore, the effect of serrated grain boundaries may be very important at a low frequency when creep phenomena begin to dominate. This is just what the results of the crack propagation experiments undertaken in this study on the serrated materials confirms : crack propagation rate 3 to 5 times slower than that of the unserrated materials.

The fractography indicated that some transgranular propagation zones appear in the serrated Astroloy, although the propagation mode remains substantially intergranular. Transgranular propagation is possible in serrated materials, since the grain boundary serration is known to impede grain boundary sliding and thus promote intragranular deformation (1,2,15). The observed intergranular facets reveal a more pertubated crack path, suggesting that this rough surface correspond to the serration whose size is approximately equal to that of coarse γ ' particles. It means that the crack path follows the serrated grain boundaries. However, such a crack path may retard the crack growth, compared to the case of the smooth grain boundaries; this makes us presume that in certain cases some deviation of the crack path from the serrated grain boundaries takes place. In any case, further effort is necessary in order to clarify the crack propagation scheme in the serrated Astroloy.

The serrated structure for Astroloy was not uniform from one boundary to another. The most serrated boundaries are random boundaries with small coincidence. Lim and Raj (18) and recently Watanabe (19) reported that formation of cavities and oxygen embrittlement on random boundaries were considerently more important than that for low coincidence boundaries. If the crack resistance depends on the proportion of high energy random boundaries perpendicular to the stress axis, it is likely that introduction of serrations increases crack growth resistance in creep fatigue conditions by reducing the boundary surface perpendicular to the stress axis for a given boundary. It appears that the most reinforced boundaries by serrations (random boundaries) were the weakest boundaries before serration formation. This can provide a "homogenization" of the strength of various grain boundaries from the crack propagation resistance point of view; the crack growth rate can thus be reduced.

Conclusion

The formation of serrated grain boundaries and their influence on the mechanical properties have been studied in Astroloy prepared by powder metallurgy. The formation of serrated grain boundaries requires precise control of specific heat treatment parameters.The most pertinent parameters are :

- the homogenization temperature ($T_1 > T_s$)
- the cooling rate (< 5°C/mn)
- the cooling range ($T_2 < T_s$ - 50°C)

The formation of serrated grain boundaries which requires grain boundary migration may be classified as a type of discontinuous reaction. Among the various populations of γ ' particles, those in contact with grain boundaries are the most important ones for the formation and growth of serrations. Special attention was payed to the influence of coincidence of the boundary on the development of serrations; random grain boundaries are the most favorable sites for such a development because of their high migration rate.

Although the results of the tensile tests conducted at 750°C on the two grain boundary morphologies showed only a slight difference in the elongation, an important improvement in crack propagation resistance was observed with the introduction of the serrated grain boundaries. This improvement is about a factor of 3 to 5 in crack propagation rate. Such an improvment is associed with a change in crack propagation scheme which becomes more pertubated, while the crack propagation mode seems to remain intergranular.

Achnowledgments

This work was sponsored by the Direction des Recherches, Etudes et Techniques (DRET). The authors wish to thank Dr. T. Khan, Dr. S. Naka at ONERA and Prof. T. H. Sanders 's Jr. on sabbatical leave from Georgia Tech (Atlanta, Ga) at ONERA for many fruitful discussions.

References

1. J.M. Larson and S. Floreen, "Metallurgical Factors Affecting the Crack Growth Resistance of a Superalloy", Metall. Trans. A, 8A (1977), 51-55.
2. M. Zhiping, Y. Ruizeng and G. Lian, "Effect of Zigzag Grain Boundary on Creep and Fracture Behaviours of Wrought γ' Strengthened Superalloy", M.S.T., vol. 4, 540-547.
3. A.K. Koul and G.H. Gessinger, "On the Mechanism of Serrated Grain Boundary Formation in Ni-Base Superalloys", Acta. Metall., 31(7) (1983), 1061-1069.
4. A.K. Koul and R. Thambouraj, "Serrated Grain Boundary Fotmation potential of Ni-Based Superalloys and its Implications", Metall. Trans. A, 16A (1985), 17-26.
5. A.K. Koul et al., "Development of a Damage Tolerant Microstructure for Inconel 718 Turbine Disc Material", Superalloys 1988, ed. by S. Reichman et al. (Warrendale, PA : The Metallurgical Society, Inc., 1988), 3-12.
6. O. Myagawa and al., "Zig-zag Grain Boundary and Strength of Heat Resisting Alloys", Proc. Third Int. Symp. on the Metallurgy and Manufacture of S.A., 1976 Seven Springs (Pennsylvania), 245-254.
7. M. Doi and T. Miyazaki, "The Effect of Elastic Interaction Energy on the Shape of γ' Precipitates in Ni-Base Alloys", Superalloys 1984, ed. M. Gell et al. (Warrendale, PA : The Metallurgical Society, 1984), 543-552.
8. H. Loyer Danflou, M. Marty and A. Walder, "Superalliages à base de nickel pour disques de turbomachines destinés à être mis en oeuvre par metallurgie des poudres", Rapport Technique 42/1931M, 1991.
9. H.F. Fischmeister, " Structure and Properties of High-angle Grain Boundaries", J. Physique , 46(85), Suppl. C4, 3-23.
10. A. Porter and B. Ralph, "The Recristallization of Nickel-base Superalloys", Journal of Materials Science, 16(1981), 707-713.
11. I. Kaur and W. Gust, "Diffusion along Migrating Boundaries", Fundamentals of grain and interphase boundary diffusion(2nd edition), (Editor Kaur and Gust, Ziegler Press, Stuttgart (1989).
12. V. Randle and B. Ralph, "Interactions of Grain Boundaries with Coherent Precipitates during Grain Growth", Acta. Metall., 34(5) (1985), 891-898.
13. C.L. Bauer,"Mechanisms for Grain Boundary Migration", Defect and Diffusion Forum, (Editor F.J. Kedves and D.L. Beke), 66-69 (1989), 749-764.
14. R.A. Ricks, A.J. Porter and R.C. Ecob, "The Growth of γ' Precipitates in Nickel-Base Superalloys", Acta. Metall., 31(1983), 43-53.
15. K.M. Chang, M.F. Henry and M.G. Benz, "Metallurgical Control of Fatigue Crack Propagation in Superalloys", J.O.M., Dec. 1990, 29-35.
16. H. Gonem and D. Zheng, "Depth of Intergranular Oxygen Diffusion during Environment-Dependent Fatigue Crack Growth in alloy 718", Materials Science and Eng., A150 (1992), 151-160.
17. H.F. Merrick and S. Floreen, "The Effects of Microstructures on Elevated Temperature Crack Growth in Nickel-Base Alloys", Metall. Trans. A, 9A (1978), 231- 236.
18. L. C. Lim and R. Raj, "Effect of Boundary Structure on Slip-induced Cavitation in Polycristalline Nickel", Acta. Metall., 32(8) (1984), 1183-1190.
19. T. Watanabe, "Grain Boundary Design for Control of Intergranular Fracture", Materials Science Forum, (Editor G.S.Was and S.M. Bruemmer), 46 (1989), 25-48.