Aging Effect on the Microstructure of the Superalloy Inconel 939

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Abstract

In this work were evaluated the microstructural characteristics by Optical Microscopy (OM) and Scanning Electron Microscopy (SEM) of the intermetallic γ’ (liquation, coarsening and decomposition) in the Inconel 939 alloy after 40000 hours and 850-900°C aging operation conditions. The alloy was vacuum conventional cast. The results show that the liquation phenomena take place in eutectics γ’-γ which are present mainly in the core of the dendritic arms and in the coarse films of carbides along the grain boundaries (GB), the γ’ particles change their original morphology of ordered cuboids of 320nm to disordered and coarse cuboids of 1.2μm, carbides show a morphology change from the original dispersed particles into a coarse continuous films and particles Chinese script type, this affects adversely the mechanical properties such as creep. The results of this evaluation allow to determine the main microstructural damage mechanisms which experiment some components such as blades at high temperatures in industrial conditions.

Keywords: Microstructure, diffusion, second phases, optical metallography, scanning electron microscopy (SEM).

Introduction

Actually, the design of materials considering the specific applications has lead to the incursion in the development of new superalloys of higher properties. Talking of specific applications, we can consider the different operation conditions between the aero engines and the land and marine turbines. The land and marine gas turbines use a lower grade fuel, with high sulfur contents and operate for longer periods (up to 100,000 hours), and relative lower stress and temperatures than those of the aero engines [1-2]. Such long service periods can be reached if the alloy has good microstructural stability. The base Nickel superalloys, are widely used in the fabrication of each land-base and marine gas power generation turbine blades, their high mechanical properties at elevated temperatures are attributed directly to the precipitation of some phases in the solid state during the fabrication, the ordered intermetallic γ’-Ni3(Al,Ti) consider as a first reaction phase and in minor quantity but not les important MC and M23C6 carbides which precipitate in the grain limits [3]. During service the blades of the gas turbines, suffer severe conditions of high temperature and stress, this can lead to some microstructural changes and consequently to the decrease of the mechanical properties such as tensile, creep and fatigue resistance [1-7]. Studies of this superalloys grades has showed that long exposure times to the conditions mentioned above, induce the microstructure to aging (coarsening and coalescence of γ’, MC carbides coarsening and decomposition in M23C6 along the GB, as well as the γ-γ’ eutectics), some of this phenomena can lead to partial liquation regions if the temperature is increased until the temperature were this phenomena can take place [1-3]. The superalloys Ni-base with high contents of chromium and high volume fraction (Fv) of γ’ (50% -
70%), show better corrosion properties and high mechanical resistance at elevated temperatures, this is why these alloys are often used in the fabrication of the Ruston turbine blades, commonly these turbines receive full maintenance every 30,000 to 40,000 hours of service at temperatures between 850°C. Some critical process such as rejuvenation heat treatments and hot isostatic pressing (HIP) are applied to restore the microstructure severely aged, despite of this it is necessary to carry out some microstructural evaluation and thus determine the degradation level and also identify the changes present in the alloy and in this way establish if it is necessary to reduce the time between each maintenance period and avoid the possibility of a catastrophic failure of the turbine.

**Experimental procedure**

The blades used in this study were those of the first stage from the hot section of a Ruston TB 5000 Turbine, which were conventional cast (equiaxed grains) with a Ni-base superalloy Inconel 939, the nominal composition is given in Table I. The burn gas temperature in this stage during a period of service of 40,000 hours at some relative high stress was between 850°C to 950°C. Figure 1, illustrate the zones used for the microstructural evaluation, rectangular samples were cut from both, the root and the airfoil blades, the samples from the root blade were used as a reference of the original microstructure condition, because the temperature in this zone is lower than the temperature reached in the center of the airfoil, and because of that no evident changes in the microstructure can be observed. For this purpose, the samples were electrolytic etched with 10%H₃PO₄, 50% H₂SO₄ and 40% HNO₃, the microstructural comparison (γ'-volume fraction, shape and morphology, MC carbides decomposition and liquation) between these two zones by means of techniques such as Optical Microscopy (OM) and Scanning Electron Microscope (SEM), it was possible to determine the level and type of degradation related with the operational conditions.

![Figure 1](image.png)
Table I. Bulk composition of the Alloy IN 939 from the blades used in this study.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Elements wt%</th>
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<tbody>
<tr>
<td></td>
<td>Ni</td>
</tr>
<tr>
<td>IN939</td>
<td>Bal.</td>
</tr>
<tr>
<td></td>
<td>Al</td>
</tr>
<tr>
<td></td>
<td>1.85</td>
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</tbody>
</table>

Results and discussion

Microstructural Characterization of the blade root (reference zone)

The samples obtained from the zone indicated in Figure 1 (root) were mounted, grounded, and finally electro polished with the etchant previously mentioned. The metallographic analysis of this zone was carried out. Previously was mentioned that the blade root will be used as a reference of the original microstructural condition, because during service is not in contact with the burned gas, this means that no microstructural changes occur in this zone. The microstructure consists of large equiaxed grains with an average wide of 156μm (see Figure 2a), also Figure 2b, shows a fine dendritic network with a few interdendritic segregation (black areas). Some dispersed carbides (black spots) are observed. This microstructure is common of the γ’ precipitation Ni-base alloys. Figure 3a shows a close view of the carbides, which are clearly disperse in the matrix as well as in the GB as discreet particles. The morphology of those in the matrix consists of a blocky shape.

Figure 2. Optical micrographs of the microstructure of the blade root (a) Large equiaxed grains, (b) Dendritic network and equiaxed grains.

A close view of the carbides mentioned above, allow to distinguish clearly the particular type of carbide, in this case most of them consist of MC blocky carbides disperse in the matrix and along the GB, also some isolated small discreet particles of M23C6 carbides were observed, these are formed by the decomposition of the primary MC (see Figure 3a). The identification of each type of carbide was done through the morphology and with a microanalysis applying the X Ray Disperse Energy (EDS) technique. Commonly the MC carbides are based on Ti, Ta, Nb and can be found in both the intragranular regions and along the GB, the M23C6 which are based on
Cr with W are found along the GB as discreet particles [2-4]. Figure 3b, shows the microanalysis of the blocky carbides, in accordance with the detected elements (Ti, Ta, Nb) is obvious that these particles correspond with MC carbides, the microanalysis of the small discreet particles, shows the same elements with a small quantity of W which is $M_{23}C_6$ former, some researchers comment $M_{23}C_6$ result from the decomposition of the primary MC carbides [2-3], sometimes it is possible these show a mixed composition, in this case this could be the reason why the small particles showed the same elements of the blocky MC carbides.

**Figure 3.** SEM micrograph of the distribution and morphology of carbides in the blade root (a) Disperse blocky carbides (b) EDS of the MC carbides of the blade root.

A cubical morphology, an ordered distribution, particles size average of 320nm and a 56% $V_F$ (measured in area) of the main intermetallic $\gamma'$ are common values of the original condition of the alloy (solution and age heat treating) in this section of the blade (see Figure 4). There is not evidence of deformation of $\gamma'$ such as elongation or coarsening as the result of exposition to high temperatures and stress, this corroborate the assumption that the blade root it can be considered as a cold zone when the blade is in service because of the reasons discussed earlier.

**Figure 4.** SEM micrograph of the original microstructural condition of the blade.
Microstructural evaluation of the blade hot section (airfoil)

The microstructure of the airfoil zone indicated in Figure 1, consist of elongated grains with an average wide of 205 μm as is shown in Figure 5, compared with the size of the grains observed in the reference zone, there is a considerable grain grow, this can be attributed to the high temperature and high stress service exposition for long periods. Some particles can be observed in the matrix and the GB appears to be a little wider. Figure 6a shows the carbides distribution mainly in the interdendritic regions and along the GB, it is possible to observe two different morphologies, coarse and continuous films and Chinese script type. A close view of these carbides shows clearly the script type morphology MC carbides and the continuous coarse films of M23C6 as is shown in Figure 6b, as mentioned previously this type of carbides are Ti, Ta and Nb base, the microanalysis of these carbides (see figure 7) shows that the main elements present in both correspond with the elements mentioned above and correspond with the MC carbides composition [2-4]. It is well known that MC carbides decompose to M23C6 + γ’ during operation at temperatures between 850°C to 950°C, forming the continuous and coarse films along the GB [2-3]. The same effect of a mixed composition was observed in these zones, this gives as a result, all the different type of carbides show almost the same composition with small quantities of W which is M23C6 former [8].

There is considerable formation of eutectics γ-γ’ in both the dendritic cores and in the GB, in the second one, the formation occurs from some carbides located along the GB. The phenomena of eutectic formation, the carbides MC type decomposition and its formation to coarse script type, occur when the alloy is exposed to high temperature for long periods, mainly the diffusion of γ’ and MC formers from the GB and through the matrix. There is a clear difference of the particle size the γ’ between the dendritic cores and the interdendritic regions, being bigger in interdendritic regions because the γ’formers such as Ti and Al tend to diffuse to this zones.

**Figure 5.** Optical Microscope micrograph of large equiaxed grains in the airfoil.
Figure 6. SEM micrographs of the distribution and morphology of carbides in the airfoil, (a) Coarse carbides in the GB and the interdendritic regions, (b) Script MC and continuous coarse films $\text{M}_{23}\text{C}_6$ type carbides, and eutectics $\gamma$-$\gamma'$. 

Figure 8 shows the overage microstructure of the airfoil, because of the exposition to high temperatures and high stress for a long period. The microstructure consist of a duplex $\gamma'$ (coarse ones) and secondary $\gamma'$ particles (small and round particles between the coarse $\gamma'$). A disordered distribution of $\gamma'$, because, morphologies of degenerated cuboids of size between 1.13 $\mu m$, and a 61% $V_F$ are evidence of this overage condition. Some of the $\gamma'$ original cubical morphology changes into elongated ones (rafts) oriented perpendicular to the principal stress direction [5]. The coarsening of $\gamma'$ by an overage condition occurs as follow, the diffusion of $\gamma'$ formers from the matrix to the particles which grow as the result of the diffusion phenomena and by coalescence with other particles of minor size, that’s the reason of the degenerated cubical morphology.

Figure 7. EDS of the MC and $\text{M}_{23}\text{C}_6$ type carbides.
Figure 8. SEM micrograph of the overage microstructure-γ′.

It is clear the blade airfoil zones experimented severe overage phenomena because of the exposition to high temperatures and high stress due to a long operation periods. Figure 9a shows other microstructural degradation phenomena, which was studied previously for different authors [9-10], they identified this phenomenon as incipient melting.

In this particular case, several zones of incipient melting appear along the GB, this could be explained by the next assumption: During the longs period of service and because of the high temperature to which the alloy is exposed, this conditions leads to the enrichment of this zones with Ti, Al, Ta, and Nb γ′ formers by a diffusion phenomena. The formation of eutectics γ-γ′ ahead of this zones is observed, this eutectics can be easily identified by the formation of large nodules of γ′ as is shown in figures 9a and 10. As discussed earlier, the decomposition of the MC carbides along the GB gives as a result the formation of small particles of γ′ besides the coarse film carbides (see in the upper right corner of the Figure 9a), this means that this area is reach in the elements mentioned above as is shown in Figure 9b.
The incipient melting mainly is the result of a γ’ liqation as the result of a zone enriched in γ’ formers, another condition that can produce this incipient melting is the migration of second phase particles, which involves a non-equilibrium melting of particle-matrix interface due to the diffusion of elements resulting in a local concentration [10-11].
CONCLUSIONS

The evaluation of the microstructure in different zones of the blades (root and airfoil) which are Ni-base Iconel 939 cast, showed that after 40,000 hours of operation, the airfoil microstructure compared with the reference zone (blade root) presented severe degradation as a result of overage phenomena, evidence of it is the coarsening of the γ’ and the change of cubical morphology into cubical degenerated one, also coarsening of the original blocky MC carbides and the formation of eutectics γ-γ’ mainly ahead of coarse carbides films. The incipient melting of these regions indicates that the blades experimented some high peaks of temperature during service, this suggest the service temperature reach temperatures above 1000°C.

The degradation of γ’, MC carbides and the incipient melting along the GB, are detrimental to the mechanical properties, it is well known the degradation of γ’mainly reduce the strength and creep resistance of the alloy.

Because of the severe microstructural degradation observed in the blades, it is important to reduce the time period service and in this way avoid possible failures of the turbine.

KNOWLEDGMENTS

The authors would like to acknowledge for the financial support of the CONACyT Mexico and the Paicyt as well as the Universidad Autonoma de Nuevo León.

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