

CREEP BEHAVIOUR OF A CAST RENE 41 SUPERALLOY

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ABSTRACT

The creep behaviour of a cast alloy Rene 41, produced by vacuum melting and investment casting has been studied. Round specimens of 6,25 mm diameter were machined from 13 mm diameter rods. Creep tests were carried in air in the temperature range between 855 and 900 °C, using four different stress levels. After failure the fracture surfaces of each specimen were examined by means of scanning electron and optical microscopy. As expected, creep lives diminished with a rise in temperature and/or stress. Results were compared with the various parameters proposed to incorporate temperature, stress and creep lives into a single expression, that allow to predict the time to failure under different temperatures or stress levels. Best fitting of the experimental data was obtained using the parameter proposed by Larson and Miller. Scanning electron microscopy of the fracture surfaces confirmed that failure was produced along the grain boundaries, associated with the segregation of alloy elements and impurities to this zone.

INTRODUCTION

Since the 1930's, when the gas turbine was invented its efficiency has been improved by increasing the gas entry temperature. This increase in temperature has been made possible by a combination of engineering and material developments. The conditions present in modern jet engines require components to withstand high stresses at temperatures above 2000 °F (1093 °C) in some cases [1]. The blades of the gas turbines are subjected to highly complex mechanical, thermal and corrosive stresses in service and are designed to operate for a period of 1000 to 5000 hours. During engine service, microstructural changes occur in the hotter regions of turbine blades aerofoils. If the blades operate at normal design temperatures, structural changes occur over a long period of time without drastically affecting the mechanical properties. On the other hand, if turbine entry temperatures exceed the design limits, the blade aerofoils get over heated and undesirable structural changes and degradation set in affecting the engine performance and blade life [2].

In order that they are able to withstand the extreme conditions of temperature, stress and corrosion found in modern aeroengines, alloy chemistry and microstructure have been developed to produce optimum performance, particularly in terms of elevated temperature creep strength and oxidation resistance [3]. Nickel base superalloys have been successfully used in gas turbines for components exposed to long periods of service at high temperatures. In general these alloys offer good mechanical properties and improved resistance to corrosion and oxidation due to a high chromium content and the precipitation on the matrix of carbides and gamma prime particles [4].

The necessity of diminishing the costs and the time of production has promoted the use of cast components in which the obtained form is near net shape. This supposes a lower material consumption that the necessary to obtain the same component by means of forging and a reduction in machining costs. However, the

possibility of the appearance of cast defects has precluded the use of cast components in critical applications where the failure can have serious consequences, as in the gas turbine of aeronautic engines. It is necessary to assure that these cast components are able to offer a satisfactory work before they are accepted to work in these critical applications.

The nickel base alloys can be strengthened by the following mechanisms: solid-solution strengthening, precipitation hardening and oxide-dispersion strengthening. The second group of materials, is the most widely used in the construction of components that work at high temperatures. The mechanical resistance of these superalloys can be increased with ageing heat treatment, owing to the change in the number, size, shape and distribution of the second phase particles. The alloying elements can be classified as solid-solution strengtheners, elements that form carbides or intermetallic compounds in grain boundaries or within grains and elements that form a coherent gamma- prime phase. Residual elements also are important because they may have a deleterious effect on creep behaviour [5].

Rene 41 alloy constitute a nickel base superalloy whose good properties at high temperatures are due to the strengthening conferred by the presence of γ' ($\text{Ni}_3(\text{Al,Ti})$) particles. This strengthening is maximum when the γ' phase forms little particles, finely dispersed in the matrix. During the permanence in service at high temperatures a growth and coalescence (few particles of larger size) of these particles can take place and, in consequence, a diminution of the mechanical resistance and worse creep behaviour. This alloy is able to work satisfactorily under stress at temperatures very close to its melting temperature (of the order of 85 % of the melting temperature in Kelvin degree). This factor constitutes a very favourable point for the use of this material, since the possibility of working at a higher temperature is translated to an increase in the efficiency of the gas turbine.

As it is known, creep is the slow deformation of material under a stress that results in a permanent change in shape. These changes generally are undesirable and can be the limiting factor in the life of a part. During creep of most metals and alloys at temperatures of around $0.4T_m$ (T_m = melting temperature in Kelvin degrees) and above, after the initial strain on loading, the creep rate decreases gradually during the "primary" stage until an apparently constant or steady-state rate is reached during the "secondary" stage. In most cases, the creep rate increases once again during the "tertiary" stage which terminates in fracture. Under these conditions, the total creep strains can be large and the rupture life is usually found to decrease rapidly with increasing stress and temperature. The steady-state creep rate is normally chosen as the parameter most representative of the material behaviour under specified creep conditions [6].

Using of time-temperature parameters (TTP) for presenting and extrapolating high-temperature creep-rupture data has been practiced for many years. A TTP is basically a function correlating the creep-rupture and time, which normally are recorded. When properly developed, these correlations can be used (a) to represent creep-rupture data in a compact form, allowing for analytical representation and interpolation of data not experimentally determined;(b) to provide a simple means of comparing the behavior of materials and of rating them in a relative manner; (c) to extrapolate experimental data to time ranges ordinarily difficult to evaluate directly because of test limitations. By far , one of the most common is the Larson-Miller parameter, which equation is:

$$f(\sigma) = T (\log t_R + C)$$

where T is temperature in °F and t_R is time to rupture, in hours and C is a constant that normally has a value of around 20 [7].

This study describes the creep behaviour of a cast Rene 41 superalloy in the range of temperatures of 855 and 900 °C, using four different stress levels.

EXPERIMENTAL PROCEDURE

The chemical composition of the alloy used in this investigation is presented in the table below:

TABLE 1
CHEMICAL COMPOSITION OF THE CAST RENE 41 SUPERALLOY.

	Cr	Mo	Co	Ti	Al	Fe	C	B	Ni
René 41	19,1	10,2	11,1	3,2	1,6	2,1	0,07	0,005	Bal.

The samples were fabricated by vacuum melting and investment. In investment casting, a ceramic is applied around a disposable pattern, usually wax, and allowed to harden to form a disposable casting mold. The term disposable means that the pattern is destroyed during its removal from the mold and that the mold is destroyed to recover the casting. The basic steps in this process are:

- a) Machining of metal molds of the desired shape. Almost always are constituted of two halves to facilitate in the shake out operation.
- b) Preparation of the called "lost models" by injecting the pattern material (wax) into the metal molds fabricated in the previous stage. This injection is made at low temperatures and pressures.
- c) Manufacture of ceramic shell molds is made by applying a series of ceramic coatings to the pattern clusters. Each coating consist of a fine ceramic layer with coarse ceramic particles embedded in its outer surface. A cluster is first dipped into a ceramic slurry bath. Then is withdrawn from the slurry and manipulated to drain off excess slurry and to produce a uniform layer. The wet layer is immediately stuccoed with relatively coarse ceramic particles either by immersing it into a fluidized bed of particles or by sprinkling the particles on it from above. The fine ceramic layer forms the inner face of the mold and reproduces every detail of the pattern, including its smooth surface. The operations of coating, stuccoing and hardening are repeated a number of times until the required mold thickness is achieved.
- d) Pattern removal by means of autoclave dewaxing.
- e) Ceramic shell molds are fired to remove moisture, to burn off residual pattern material and any organics used in the shell slurry, to sinter the ceramic and to preheat the mold to the temperature required for the casting.
- f) Casting of the metal. Vacuum casting provides cleaner metal and often superior properties. And finally
- g) Postcasting operations: knocked off with a vibrating pneumatic hammer of the shell material that remains on the casting, heat treatment, abrasive cleaning, HIP, machining, chemical finishing treatments...[8].

Investment cast samples was formed by a bundle of ten 13 mm diameter rods. Once finished the casting operations these rods were given a heat treatment consisting in solution treating for 3 hours at 1066 °C, oil quenched and ageing for at 760 °C for 16 hours and air cooled.

The creep tests were performed according to the ASTM E 139 (175) Standard, at different temperatures, between 855 °C and 900 °C and under four stress levels. These tests were carried out in Mayes machines, equipped with three zone furnaces, which guarantee the homogenous heating of the samples. The reason of changing the temperature and the stress in the different tests is to analyse the effect exerted by these two variables, separately or in union, in the creep life of the alloy.

Ten cylindrical specimens of 6,25 mm diameter were machined from 13 mm diameter rods and creep tested were carried in air until fracture. The used stress levels and temperatures were 148 MPa, 156 MPa, 164 MPa, 172 MPa and 855 °C, 870 °C, 885 °C, 900 °C respectively. This test method, in which the applied stress remains constant, modifying the temperature takes the name of "isostress". The drawback of this method is the possible microstructural change of the material that can occur, increasing test temperature. However, in the temperature range considered for this study, no significant microstrucutral changes are expected and the testing procedure is adequate.

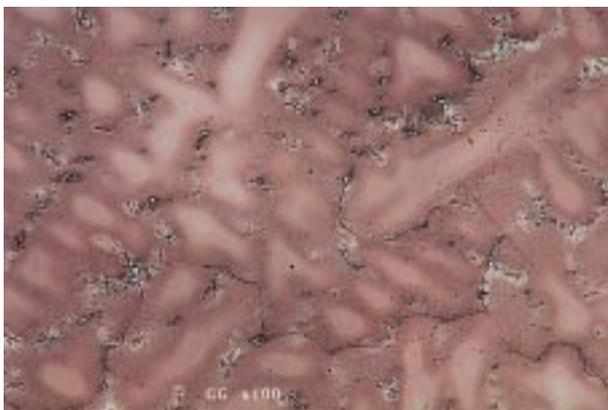
After failure, the fracture surfaces of one half of these specimens were examined in a scanning electron microscope and metallographic samples were prepared from the longitudinal sections of the other halves and observed by means of optical microscopy. A high precision cutter, at very slow speed and with continuous refrigeration - avoids the heating which alter the microstructure - has been used. The polishing of the samples has been made carefully preventing that the various particles present in the microstructure last during this process. This task is slow and costly but necessary to obtain a trustworthy characterization. The observation of the sample only in the optical microscope does not allow the definitive identification of the particles so, it is necessary to use the energy dispersive spectrometry (EDS).

RESULTS AND DISCUSSION

As commented above, the metallographic characterization has been performed by means of the optical microscope and scanning electron microscope, using the energy dispersive spectrometry to identify the nature of the present phases.

The observation of unetched specimens just reveal the presence of creep cavities that can be hidden by the posterior metallographic etching. Even after etching (task quite difficult in this kind of material) is not possible to identify adequately the particles [9]. For this reason the observations carried out in the optical microscope have been confirmed by means of energy dispersive spectrometry in the scanning electron microscope.

The observation at low magnifications reveals a dendritic microstructure, showing a preferential precipitation of the phases in the interdendritic zones, due to the segregation of some elements in these areas, which allow the formation of phases impossible to be with the average composition of the alloy. Higher magnifications make clear the complex character of many of the massive particles. They are composed of a central part with a slender grey shade surrounded by a white compound. Likewise, it is appreciate fine spots, whose resolution is not suitable by optical microscopy. The figure 1, presents the dendritic microstructure at different magnifications of the Rene 41 superalloy.



a)

b)

Figure 1: Microstructure of cast Rene 41 superalloy. a) x100; b) x1000.

An excitation energy of 10 kV has been used to avoid that radiation of the matrix interfere in the identification of the little phases. The figure 2a presents a scanning electron microscope micrograph of some of the particles present in the Rene 41. The analyse of the central zone of these particles, marked with an A in the figure, shows that it is constituted by titanium, principally, and a little quantity of molibdenum, elements that can form an intermetallic compound or a carbide. To achieve an exact identification of the composition of the particles wave length Spectrometry was used. This analysis reveals a clear peak of carbon, pointing that the particles correspond to carbides of the type MC, where M is basically titanium with a little part of molibdenum. The external part of the particles (marked B) is constituted by molibdenum, chromium and carbon forming carbides of the type M₆C. The particles marked with a C possess a simple morphology constituted by molibdenum, chromium and carbon and can be identified as a M₂₃C₆ carbide.

The figure 2b presents the morphology of the spots detected in the metallographic examination in the optical microscope. This phase has been identified as γ' , owing to in the analysis by energy dispersive spectrometry appears peaks of nickel, aluminium, titanium and other ones smaller of chromium, molibdenum and cobalt. It is necessary to use dark field transmission electron microscopy to detect the real shape and to measure the size of these particles, which can change during high temperature exposure [10].

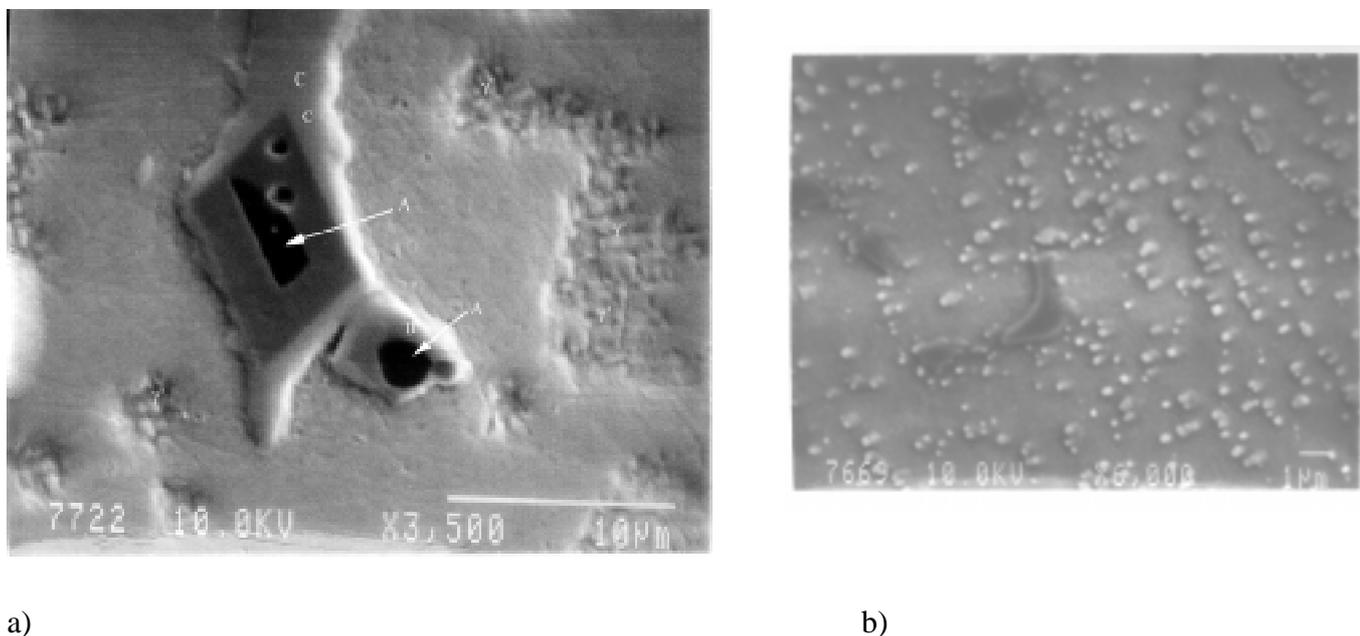


Figure 2: Cast Rene 41. a) SEM micrograph of some particles; b) γ' phase

Visual examination of fracture surfaces is enough to detect their very marked faceted topography. As can be seen in the figure 3a, the examination in the optical microscope of the metallographic samples prepared from the longitudinal sections reveals the presence of cavities in zones close to the fracture. The figure 3b shows a cavity present in one of the creep samples. As can be seen, a lot of phases are situated close to it. As commented, in the matrix dispersed fine particles correspond to the γ' phase, the lips of the cavity are constituted of MC carbides pointing towards a effect of this constituent in the failure process. The internal part of these particles (darker shade) has a different composition, constituting the compact hexagonal η phase (Ni_3Ti), undesirable phase that used to form in alloys where titanium content is much higher than the aluminium content. In the present case aluminium and titanium contents can be considered as correct and probably the presence of this phase is due to local segregation and has been promoted by the long exposure.

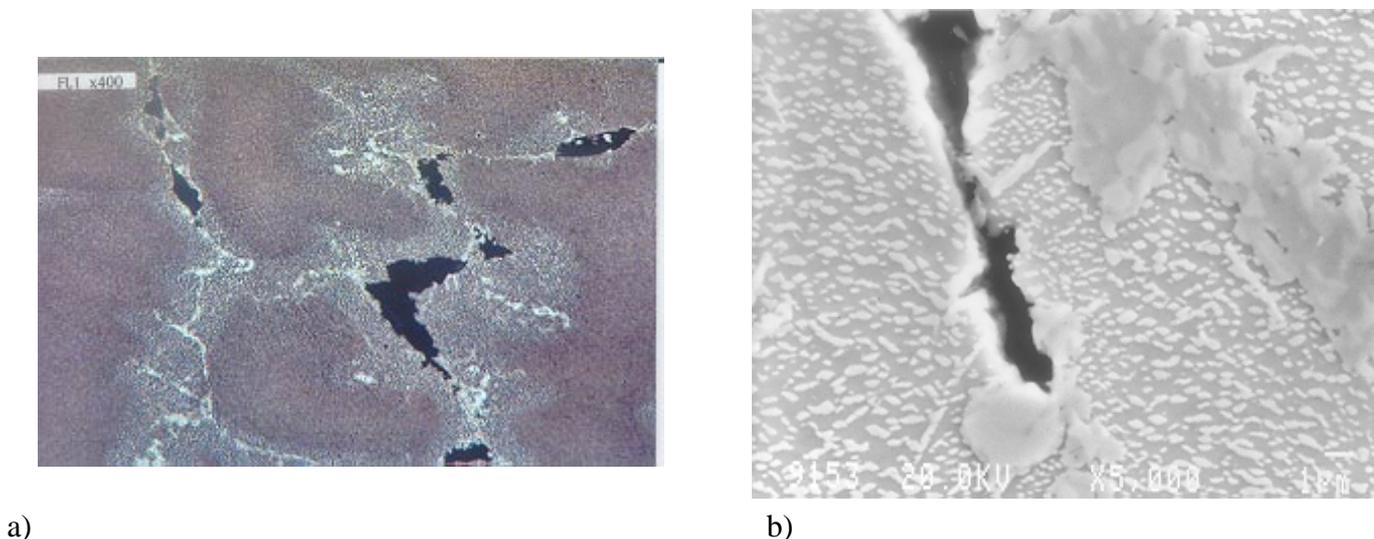


Figure 3: a) Cavities present in zones close to the fracture. b) Particles surrounding a cavity.

The fracture surfaces appear totally covered with oxidation. The analysis by EDS reveals the presence of chromium as predominant element, indicating the influence of environment on high temperature service. Nevertheless, corrosive attack did not penetrate in the bulk material, pointing towards a good protection by the presence of chromium in the alloy. The contribution of aluminium to this protective action has been also claimed but in the present case no significant amount of this element was detected in the oxide film. The more plausible reason for this behaviour is the small amount of aluminium in solid solution (most of it is combined forming γ' particles) that could participate in this process.

Scanning electron microscopy confirmed that failure was produced along the grain boundaries, associated with the segregation of alloy elements and impurities to this zone as it has been commented previously. The analysis by EDS of the particles found in the cavities reveal a significant peak of iron, together with smaller ones of chromium and titanium. More plausible identification point towards Laves phases, promoted by the presence of iron in the composition of the alloy. It must be remarked that metallographic examination of the virgin material did not reveal the presence of these particles. There are two possible explanations. The first one would attribute their presence to precipitation during high temperature exposure. A second possibility would consist in the presence of these particles in the virgin material but have been undetected during the metallographic examination. Even if in depth research is needed before reaching a definitive conclusion, the thorough examination of the virgin material supports the first hypothesis.

The strong influence of iron in the formation of Laves phases has been shown in some previous works and the maximum amount of this element is limited in those components that operates in the highest range of temperature. Maximum amount allowed in cast Rene 41 is 5 % and in the studied material is even lower (2,1 %) but its effect on the formation of Laves phases is evident as most of the iron is concentrated in these particles. A more strict control of the amount of iron in the alloy is likely to produce an even better creep behaviour and it is recommended.

In the following table the results of the creep tests are presented:

TABLE 2
CREEP TESTS RESULTS

Reference	Temperature (°C)	Stress (MPa)	Time to fracture (h)
FL-1	900	172	116
FL-2	900	164	118
FL-3	900	156	136
FL-4	900	148	176
FL-5	885	172	144
FL-6	870	172	327
FL-7	855	172	606
FL-8	870	156	511
FL-9	855	156	824
FL-10	870	148	819

It can be verify that an increase of the temperature or of the stress lead to a diminishing in the creep life of the alloy. In an attempt to include the effect of both parameters - stress and temperature - and to make a prediction of the residual life, several models have been proposed.

Best fitting of the experimental data was obtained using the parameter proposed by Larson and Miller. Plot of the logarithm of the time to failure versus the inverse of the test temperature, for various stress levels are represented in the next graph.

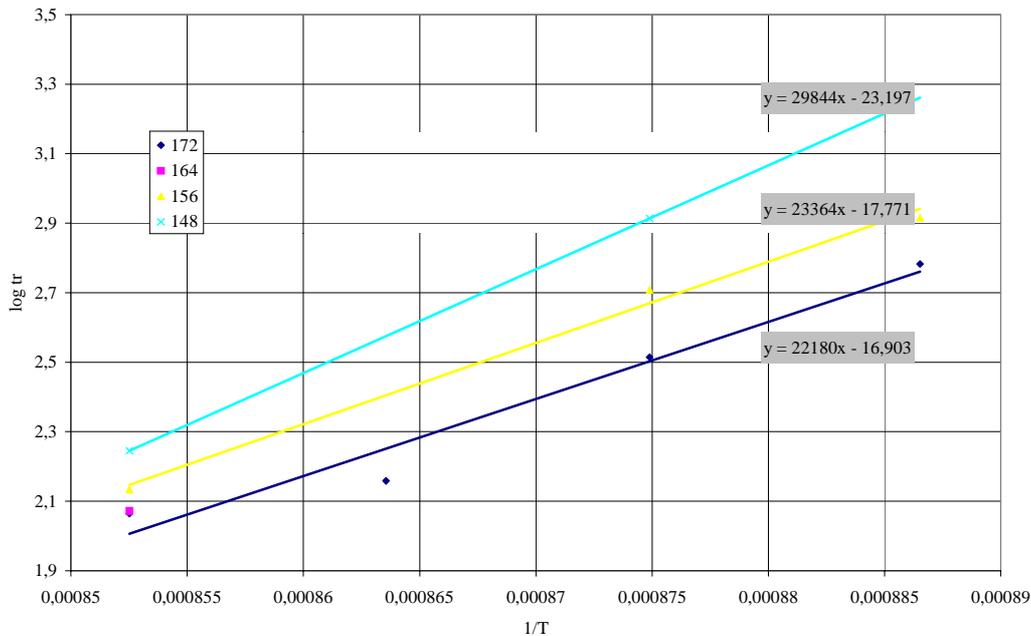


Figure 4: Larson Miller parameter

The results are straight lines that intersect the axis at points between 17 and 23. Moreover, if all these lines are forced to converge to the same point, a value of 18,3 is obtained, in a very good agreement with the initially proposed value of 20, even more if values between 10 and 40 are reported for various metallic materials. Consequently, it is expected that Larson and Miller parameter will give a good prediction of the creep performance of this alloy and could be used to evaluate the future performance of real components.

Comparison of these results with creep lives reported for wrought material reveal a better creep behaviour of the investment cast samples, encouraging to use this material in components that operate at high temperature under constant stress. Fatigue test which were previously carried out, also showed a very good performance of this cast material, pointing towards that also under alternate stresses cast components can operate safely [11].

CONCLUSIONS

The analysis of the results obtained along the fulfilment of this project has allowed to achieve the following conclusions:

- The creep behaviour of the investment cast René 41 alloy can be considered excellent, exceeding even the life of some forged alloys. It can be observed a good correlation between the experimental results and the predictions made based in the utilization of the Larson-Miller parameter, which can be used to evaluate the service life of the components.
- A metallographic characterization have been performed to identify the phases present in the microstructure of the samples by means of the optical and scanning electron microscopes, aided by the EDS. The identification is quite difficult owing to the phases are complex and particles with similar appearance can correspond to phases totally different. However, γ' , various carbides and a small amount of iron rich particles that are mainly concentrated at the fracture surfaces (probably Laves phases formed during the long exposure at high temperature) were identified. A more strict iron content is recommended.

- c. The presence of iron promotes the formation of Laves phases that contribute to the failure of the component. A more strict control of the content in this element would optimise the creep performance of the alloy.
- d. It has been performed the examination of both fracture surfaces and metallographic samples in the scanning electron microscope and in the optical microscope. The creep failure is basically intergranular, forming cavities associated with particles present at the grain boundaries.

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REFERENCES

1. Bowman, R., Antolovich, S.D. (1998). *Metallurgical Transactions A*. Vol 19A, 93.
2. Ramakrishna, S., Rao, Y.K., Mukhopadhyay, A.K. and Krishnadas Nair, C.G. (1990). *The Minerals, Metals and Materials Society*, 411.
3. Vallés, J.L., Arrell, D.J., Bressers, J. (1994). *The Materials Challenge*. Vol. 5, 8.
4. Ferrari, A. Effect of microstructure on the early stages of creep deformation of an experimental nickel base alloy.
5. Metals Handbook, Vol. 3, 207.
6. Evans, R.W. and Wilshire, B. (1985). In: *Creep of Metals and Alloys*. The Institute of Metals, 3.
7. Boyer, E. (1988). In: *Atlas of Creep and Stress-Rupture Curves*. ASM International, 3.6.
8. Casting. Metals Handbook. Ninth Edition, 253.
9. Atxaga, G., Erauzkin, E., Irisarri, A.M. (2000). 1ª Jornada de Metalografía. INASMET. San Sebastian: March 2000.
10. Irisarri, A.M. (1980). Tesis Doctoral: *Cinética del envejecimiento de algunas aleaciones del sistema Ni-Al a temperaturas elevadas*. E.S.I.I. San Sebastian.
11. Atxaga, G., Irisarri, A.M. (1999). *Comportamiento a fatiga a temperatura elevada de una aleación moldeada Rene 41*. Anales de Mecánica de la Fractura., 161.