Hindawi Publishing Corporation
Advances in Materials Science and Engineering
Volume 2016, Article ID 1745839, 7 pages
http://dx.doi.org/10.1155/2016/1745839

Research Article

Microstructural Changes during High Temperature Service of a Cobalt-Based Superalloy First Stage Nozzle

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Received 30 October 2015; Revised 7 March 2016; Accepted 8 March 2016

Academic Editor: Amit Bandyopadhyay

Superalloys are a group of alloys based on nickel, iron, or cobalt, which are used to operate at high temperatures ($T > 540^{\circ}$C) and in situations involving very high stresses like in gas turbines, particularly in the manufacture of blades, nozzles, combustors, and discs. Besides keeping its high resistance to temperatures which may approach 85% of their melting temperature, these materials have excellent corrosion resistance and oxidation. However, after long service, these components undergo mechanical and microstructural degradation; the latter is considered a major cause for replacement of the main components of gas turbines. After certain operating time, these components are very expensive to replace, so the microstructural analysis is an important tool to determine the mode of microstructure degradation, residual lifetime estimation, and operating temperature and most important to determine the method of rehabilitation for extending its life. Microstructural analysis can avoid catastrophic failures and optimize the operating mode of the turbine. A case study is presented in this paper.

1. Introduction

Gas turbines blades are manufactured mainly with nickel-based and cobalt-based superalloys. During the commercial operation of gas turbines, which are part of a power station, blades and other components of turbine are subject to natural wear and damage due to various causes which can interrupt continuous operation. The source of damage may be metallurgical or mechanical and is manifested in the equipment operation such as a decrease in the availability, reliability, and performance and an increase in the risk of failure. Also, after a prolonged service, moving blades and nozzles show a decrease in metallurgical characteristics, so the creep strength, fatigue, impact, and corrosion resistance decrease. There are different factors which influence lifetime of the main components of a gas turbine including design and operating conditions, but it is the latter that has an impact on the lifetime of these components. Generally, for most gas turbines, operating conditions are very severe. The following factors have great effect: operation environment (high temperatures, fuel and air contamination, solid particles, etc.), high mechanical stresses (due to centrifugal forces, vibratory, and flexural stresses, etc.), and high thermal stresses (due to thermal gradients).

The phenomena described above do not operate in isolation; typically there are two or more factors being active simultaneously, causing reduction of blades or nozzle lifetime under the following damage mechanism [1, 2], that is, creep, thermal fatigue (low cycle fatigue), thermomechanical fatigue (high cycle fatigue), corrosion and oxidation, erosion, or foreign object damage (FOD).

The type of damage or degradation which occurs in gas turbine blades and nozzles after prolonged service mainly includes external surfaces damage (corrosion, oxidation, cracks, foreign object damage, erosion, and fretting) and internal damage of microstructure, such as γ' phase coarsening, grain growth, micro void growth in grain boundaries, carbide precipitation, and brittle phase formation.

Surface damage produces dimensional deterioration, generating loss of the blade/nozzle original dimension, resulting in increased stress and turbine efficiency reduction. During operation, the material microstructure is affected by high temperature combined with high stresses. However, the extent of deterioration differs due to the following factors: total service time and operation history (number of startups, shutdowns, and trips), gas turbine operation condition (temperature, rotational speed, mode of operation, i.e., base load or cyclic duty), and manufacturing alterations (grain size, porosity, alloy composition, heat treatment).

Then, a brief description of the Ni-base and Co-base base superalloys is given. These alloys are used in the manufacture of critical turbine components (moving blades, nozzles, combustors, and transition ducts) of stationary gas turbines. Fe based or Ni-Fe based superalloys are not mentioned because their use in gas turbine critical components is not common.

Ni-Based Alloys. The nickel-base alloys are the more complex and the most widely used for the hottest components of gas turbines (e.g., gas turbine first stage blades). In the heat treated condition superalloys represent a composite material consisting of several intermetallic phases linked by a metal matrix. The major phases present in these superalloys are [3] as follows: gamma matrix (γ), Ni-based austenitic phase (FCC), usually containing a high percentage of solid solution elements such as Co, Cr, Mo, and W; gamma prime (γ'), which is Ni_3(Al, Ti) based intermetallic phase; Carbides, generally types M_6C and $M_{23}C_6$ which tend to precipitate into grain boundaries; topologically closed packed (TCP) type phases, such as σ, μ, and Laves, which precipitate after prolonged high temperature service.

These alloys can be classified into solid-solution hardened alloys and precipitation hardened alloys or gamma prime (γ') alloys. The former may be forged or cast, contain few elements forming γ' particles, but are hardened by refractory elements such as tungsten and molybdenum and carbide formation, and also contain Cr to impart corrosion resistance (oxidation) and Co to give microstructural stability. Precipitation hardened alloys can also be forged or cast. In addition to γ' particle formation as the main hardening mechanism also incorporates elements such as tungsten (W), molybdenum (Mo), tantalum (Ta), and niobium (Nb).

Co-Based Alloys. Cobalt-based superalloys (e.g., X 40, X 45, and FSX-414) are primarily used in the manufacture of all first stage nozzles and in some turbines are used in the last stage due to their good weldability and hot corrosion resistance. These alloys have higher strength at high temperatures than Ni-based alloys and also have excellent resistance to thermal fatigue, oxidation, and corrosion [4]. These alloys have cobalt as the principal alloying element, with significant amounts of nickel and chromium and smaller amounts of tungsten and molybdenum, niobium, tantalum, and sometimes iron. They are mainly hardened by carbide precipitation. Alloys hardening by carbide precipitation contain between 0.4 and 0.85% carbon. Such superalloys consist of an austenitic matrix (fcc) and a variety of precipitated phases such as primary carbides (M_3C_2, M_7C_3, and MC) and coarse carbides ($M_{23}C_6$) and GCP types phases (geometrically compact phases) such as γ'' and η (Ni_3Al) and TCP (topologically close packed) type phases (σ, μ, R, or L) $(Cr, Mo)_x(Ni, Co)_y$ [5].

2. Superalloys Microstructural Degradation during High Temperature Service

There are several microstructural degradation mechanisms occurring in superalloys used in the manufacture of hot section components of gas turbines (nozzles, moving blades, and combustion chambers). The most common degradation mechanisms are aging and coarsening of the γ'-phase, transgranular precipitation growth of carbides in grain boundaries, brittle phases precipitation, and growth of cavities due to creep.

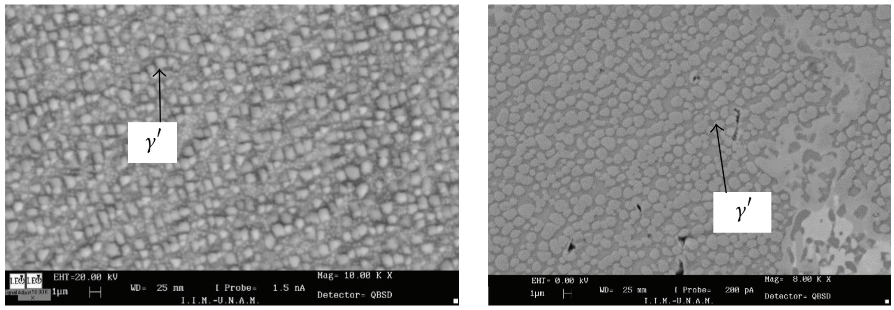
Coarsening and Aging of γ' Phase. The size and shape of the γ' phase in nickel-based superalloys are not stable after long periods of operation at high temperatures. However, after a heat treatment, this phase is very near to equilibrium with the γ matrix and therefore there is little additional precipitation or growth of this phase from the supersaturated matrix. Nevertheless some particles may grow by a diffusion mechanism [6]; that is, the average particle radius increases with aging time, t. This is represented by the following equation:

$$\bar{r}^3(t) - \bar{r}^3(0) = Kt, \tag{1}$$

where $\bar{r}(0)$ is the average radius of the particle at $t = 0$, $\bar{r}(t)$ is the average radius of the particle in time t, and K is the kinetic constant which depends on temperature. Various studies [7, 8] on γ' growth phase in Ni-base superalloys and Fe-Ni-Al alloys have confirmed that growth obeys the law described in (1).

Changes in morphology of the γ' phase modify the mechanical properties of the material's component, since phase γ' is intended to act as a barrier to dislocation movement slowing creep; consequently resistance to this failure mechanism is greatly diminished [9]. In commercial superalloys, the γ' phase changes from spherical to cuboidal shape, although most of the particles have an intermediate form. Aging is revealed as an increase in average particle size. The γ' phase can be identified in the microstructure as particles whose shape is irregular and larger [10, 11]. The shape of this phase in a nondegraded and degraded condition can be observed in Figures 1(a) and 1(b).

Morphology and Degeneration of MC, $M_{23}C_6$, and M_6C Carbides. The role of carbides in superalloys is complex; carbides seem to prefer the grain boundaries as a site location in Ni-base superalloys, while in Co-base and Fe-base superalloys appear to precipitate intragranularly [3]. The most common carbides in all Ni, Fe-Ni, and Co-base superalloys are basic MC, $M_{23}C_6$, and M_6C and seldom M_7C_3 [12]. The most stable carbide found in Ni-base and Co-base superalloys is the MC type, where M represents the element Ti. A fraction



(a) (b)

Figure 1: Blade root micrograph of (a) gamma prime (γ') without degradation and (b) gamma prime (γ') with moderate degradation after 24000 h in service, IN738LC superalloy.

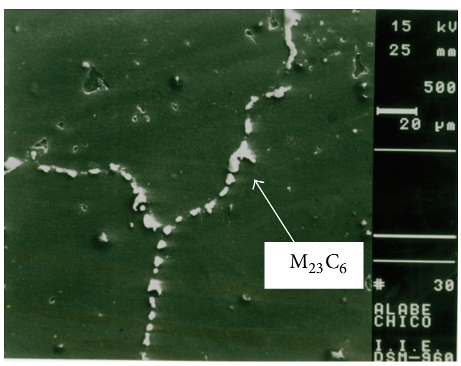


Figure 2: $M_{23}C_6$ carbides precipitated in grain boundaries, IN738LC superalloy, after 24000 h of service.

of Ti can be replaced by Nb, Ta, W, and Cr, depending on the alloy composition. In Co-based superalloys containing W, WC carbide is dominant [13]. This carbide generally has a pseudo cubic or script shaped figure; it precipitates as discrete particles distributed heterogeneously through the alloy in intragranular or transgranular locations. The source of carbon needed in the heat treatment of these alloys is taken from the WC. In the course of a prolonged service, MC primary carbides decompose into secondary carbides rich in chromium ($M_{23}C_6$). MC carbide decomposition occurs by diffusion of carbon into the γ matrix and γ' phase, resulting in the formation of $M_{23}C_6$ carbides near the matrix-interface [14], as shown in Figure 2. The MC decomposition can be stated by the reaction:

$$MC + \frac{\gamma}{\gamma'} \longrightarrow M_{23}C_6 + \gamma' \qquad (2)$$

or

$$(Ti, Mo)\,C + (Ni, Cr, Al, Ti) \longrightarrow Cr_{21}Mo_2C_6 + Ni_3\,(Al, Ti) \qquad (3)$$

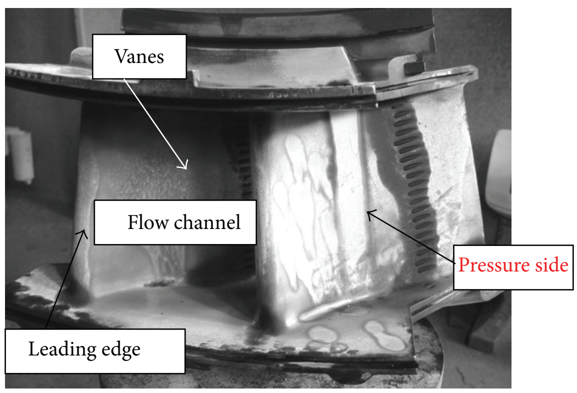
The above reaction occurs at a temperature of approximately 980°C (1800°F) but has also been observed at a temperature of about 760°C (1400°F) [15, 16]. The $M_{23}C_6$ carbide has a significant effect on the superalloys properties. Its critical location (grain boundaries) increases the rupture strength inhibiting grain boundary sliding. However, failure by break may be originated by fracture of these particles.

Phase Topologically Close Packed (TCP). Superalloys have high levels of refractory elements such as Mo, W, Re, Ru, and Ta, in order to increase creep and crack resistance [17, 18]. These elements function as solid-solution enhancers of both the γ matrix and the γ' phase. Re is a strong hardener; it precipitates mainly in the γ matrix and apparently slows degeneration of the γ' phase. High amounts of refractory elements make the superalloy prone to form TCP phases, the σ phase being the most common in Ni-base superalloys [19]. It has been shown that the formation of these phases has a detrimental effect on the creep rupture life of superalloys. These phases increase the strain rate of both conventional and single crystal superalloys [20, 21]. Other detrimental effects on superalloys are a decrease in ductility, impact resistance, and thermal fatigue.

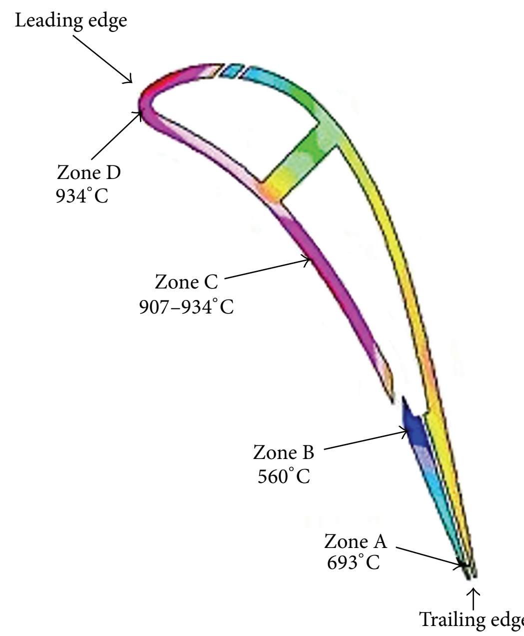
3. Case Study: Degradation in Service of a Gas Turbine First Stage Nozzle Segment

The nozzle segment (the complete wheel comprises 16 segments with two blades per segment) of the first stage of a gas turbine serves to rotate and direct the flow of hot gas to the rotating turbine with the most favorable incident angle. There is no centrifugal load on the nozzle segment. The combination of bending loads a thermal gradient caused by cooling of the nozzle results in high stationary operating stresses on the nozzle [22]. The first stage nozzle may experience damage mechanisms such as creep, fatigue-creep, oxidation, corrosion, and mechanical damage during its service life [23]. The microstructural evaluation is one of the most important tools in assessing the current condition of the nozzle segment for its correlation with the service conditions experienced by the component. The microstructural evaluation can point out strategies for repair and/or heat treatments for rejuvenation and recover the mechanical properties and extend the useful life of the alloy.

The evaluated component is a segment of the first stage nozzle of a 60 MW gas turbine; gas inlet temperature to



(a)



(b)

FIGURE 3: (a) General view of the nozzle vane. (b) Analysis regions and internal temperature distribution on the nozzle vane transversal section in the cutting plane at 50% height (section of maximum temperature).

TABLE 1: Chemical composition of FSX-414 superalloy (wt%).

Alloy	C	Cr	Ni	Co	W	Fe	B
FSX-414	0.25	29	10	52	7.5	1.0	0.01

the turbine is 1086°C. The full nozzle consists of 32 vanes and is cooled by air extracted from the compressor discharge. The microstructural evaluation was performed after 54,000 operating hours in mode of base load. The nozzle is made of a conventional cobalt-based FSX-414 superalloy by means of conventional investment casting (equiaxial grains) and without coating; its chemical composition is shown in Table 1. The gas turbine operates with natural gas. An overview of the nozzle segment (two vanes) is shown in Figure 3(a); the vanes have cooling passages on the pressure surface, and, in Figure 3(b), the different operating temperature zones are indicated on a section of the nozzle block. The maximum service temperature (Figure 3(b)) is recorded in the leading edge of the nozzle blade and the temperature distribution was obtained by numerical analysis using Computational Fluid Dynamics (CFD) with the code Star CD V 3150 [24]. Figure 4 shows some cracks detected near the cooling cavities of the nozzle, close to the trailing edge.

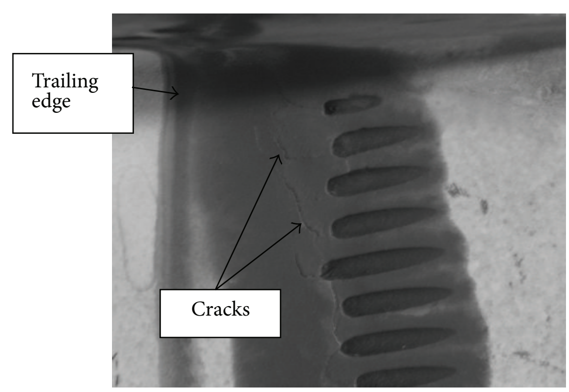
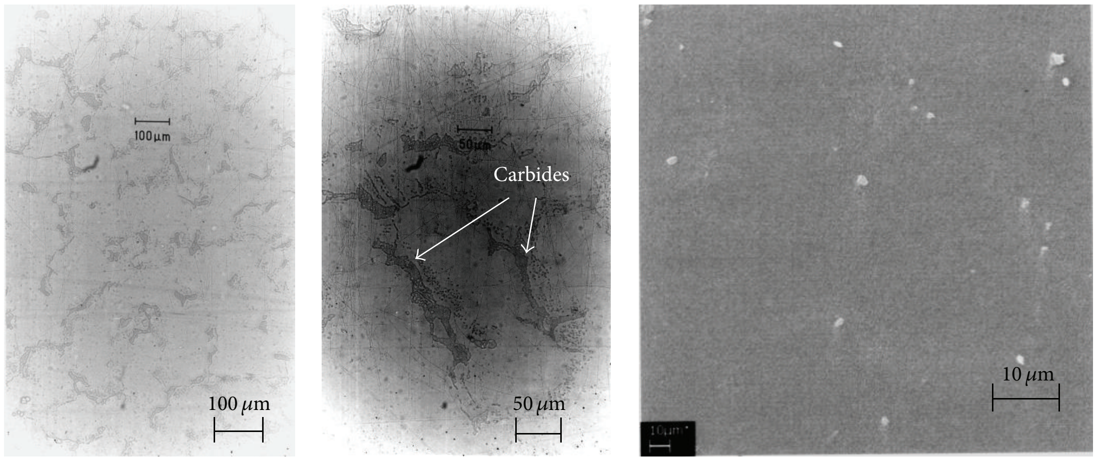


FIGURE 4: Cracks on the nozzle vane near the internal shroud close to the trailing edge.

Microstructural Characterization of Nozzle Blade. The nozzle microstructure was evaluated at a zone corresponding to a height of 50% of the flow channel on the low and high temperature section. The characterization included grain size and carbide precipitation. In order to evaluate the extent of damage in the superalloy, the microstructure in the low temperature zone (zone B) was compared with the high temperature region (zone D). The microstructure of the low temperature zone can be taken as a reference or initial condition of the alloy, because at that temperature microstructural changes are insignificant. The microstructure of the low temperature zone is shown in Figure 5; this consists of equiaxed grains of the γ phase matrix (Figure 5(a)) and at higher magnification (Figure 5(b)) dispersed carbide particles in the grain boundaries and matrix can be observed. Figure 5(c) shows the unit area quantized to determine the percentage of precipitates. This microstructure is characteristic of cobalt-base superalloys [25–28].

Table 2 shows the grain size and the volume fraction at the different zones in the nozzle pressure side in cross section. Volumetric fraction of carbides in each area was determined



(a) (b) (c)

Figure 5: (a) General microstructure, (b) magnified microstructure of the low temperature zone (zone B), and (c) minor precipitation of carbide particles.

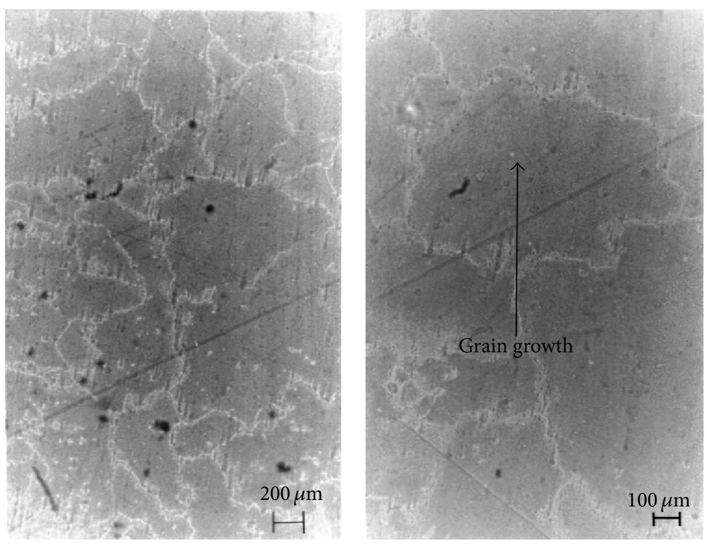


Figure 6: Grain growth in the high temperature (zone D).

Table 2: Quantitative microstructure in different zones of the nozzle vanes.

Microstructural parameter	Zone A	Zone B	Zone C	Zone D
	Temperature [°C]			
	693	560	907	934
Average grain size [μm]	313	54	401	531
Volume fraction of carbides [%]	7.36	0.72	10.22	12.96

taking into account the area ratio of carbides in μm^2/total measured area also in μm^2.

As shown in Table 2, the extent of deterioration of the superalloy (grain growth and higher amount of precipitates) depends directly on the metal temperature. The micrographs in Figure 6 show a larger grain size for the area where the temperature is high (zone D), and the micrograph of Figure 7 shows a higher amount of precipitates (area D) compared to the “cold” or reference; see Figure 5. The grain size ratio between area A (693°C) and the high temperature zone D (934°C) is 0.6 and the grain size ratio between the reference area and the high temperature area is 0.1. The growth of grain size (coarse growth) is one of the main symptoms of microstructural worsening of nozzle’s material. This is explained because the material is exposed to gas at high temperature and velocity.

The nozzle microstructural investigation revealed the presence of a continuous band of precipitated carbides in grain boundaries and a rise in the volume fraction of carbides up to 50%. This occurs because of the transformation of M_6C carbides to $M_{23}C_6$ carbides. Carbide transformation is

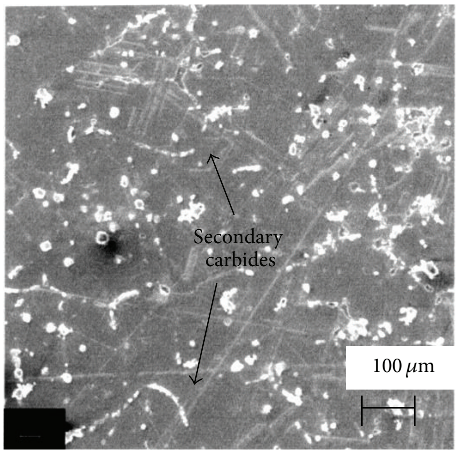


Figure 7: Precipitated carbides in grain boundaries of FSX-414 superalloy at intermediate temperature zone (area D).

encouraged by the high operating temperature of the nozzle, mainly at the blade leading edge; the latter type of secondary carbides takes place abundantly in the Co-base superalloys with more than 5% Cr [29]. Precipitation of these secondary phases in grain boundaries reduces material creep resistance.

This dense and continuous network of carbides observed mainly in the area D reduces the ductility and toughness of the alloy by up to 30% of its initial value and may facilitate initiation and propagation of cracks due to grain boundaries brittleness; all this leads to decreasing the useful life of the alloy. Additionally, such grain boundary precipitation reduces the material creep resistance [1, 27]. The microstructural characterization of FSX-414 superalloy revealed that the grain size increased considerably; see Figure 6. This may also reduce the material fatigue life [1, 30]. Also, the average grain size increment in the vane body reduces alloy fatigue life [1].

An analysis of thermal stress was performed which is not indicated in this work, but the results showed that the maximum tension stresses at steady-state were located near the cooling holes and blade profile on the pressure side of the nozzle.

In addition, because the gas turbine nozzle is a fixed component, its operational stresses are generated only by the gas flow pressure and by thermal loads due to temperature gradients through the nozzle elements. These stresses and temperature gradients cause fatigue damage during transient and steady-state operation; this thermal stress induces the initiation and propagation of cracks.

From the metallurgical evaluation carried out and cracks detected by nondestructive testing, the nozzle segment analyzed is a candidate for repair. It is noteworthy that there are virtually no limits for their rehabilitation by welding, because this component remains fixed during operation and is not exposed to concentrated mechanical stresses caused by the centrifugal force.

Repair may include use of conventional welding and/or brazing, subsequently applying a postweld heat treatment including a solution heat treatment at a temperature of 1150°C followed by rapid cooling and then an aging cycle at a temperature of 980°C followed by cooling. In the event that the nozzle blocks have no coating and in order to decrease the effect of elevated temperature on the microstructure of the blades, the use of a thermal barrier coating (TBC) should be considered to improve corrosion and oxidation resistance.

4. Conclusion

A microstructural study to determine the extent of damage in terms of microstructural deterioration can be used to identify and therefore determine the type of repair (heat treatment, welding, conventional or brazing) to the nozzle block segment. Comparing deterioration parameters discussed above and the temperature distribution over the nozzle block, a direct relationship between the magnitude of damage of the superalloy and the metal temperature can be established. Therefore, metallurgical analysis of main components of a gas turbine is a very useful tool that provides information needed to make decisions about the possibility of repair, establish risk of fracturing and evaluate the operating conditions of the equipment. Consequently, metallurgical characterization should be incorporated into maintenance schedules. It is noteworthy to mention that any metallographic study should be complemented by a stress and temperature distribution analysis in order to corroborate or determine the nozzle failure mechanism.

Competing Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

Financial support from Consejo Nacional de Ciencia y Tecnología (CONACYT, Mexico) (Projects 196205, 159898, and 159913) is gratefully acknowledged.

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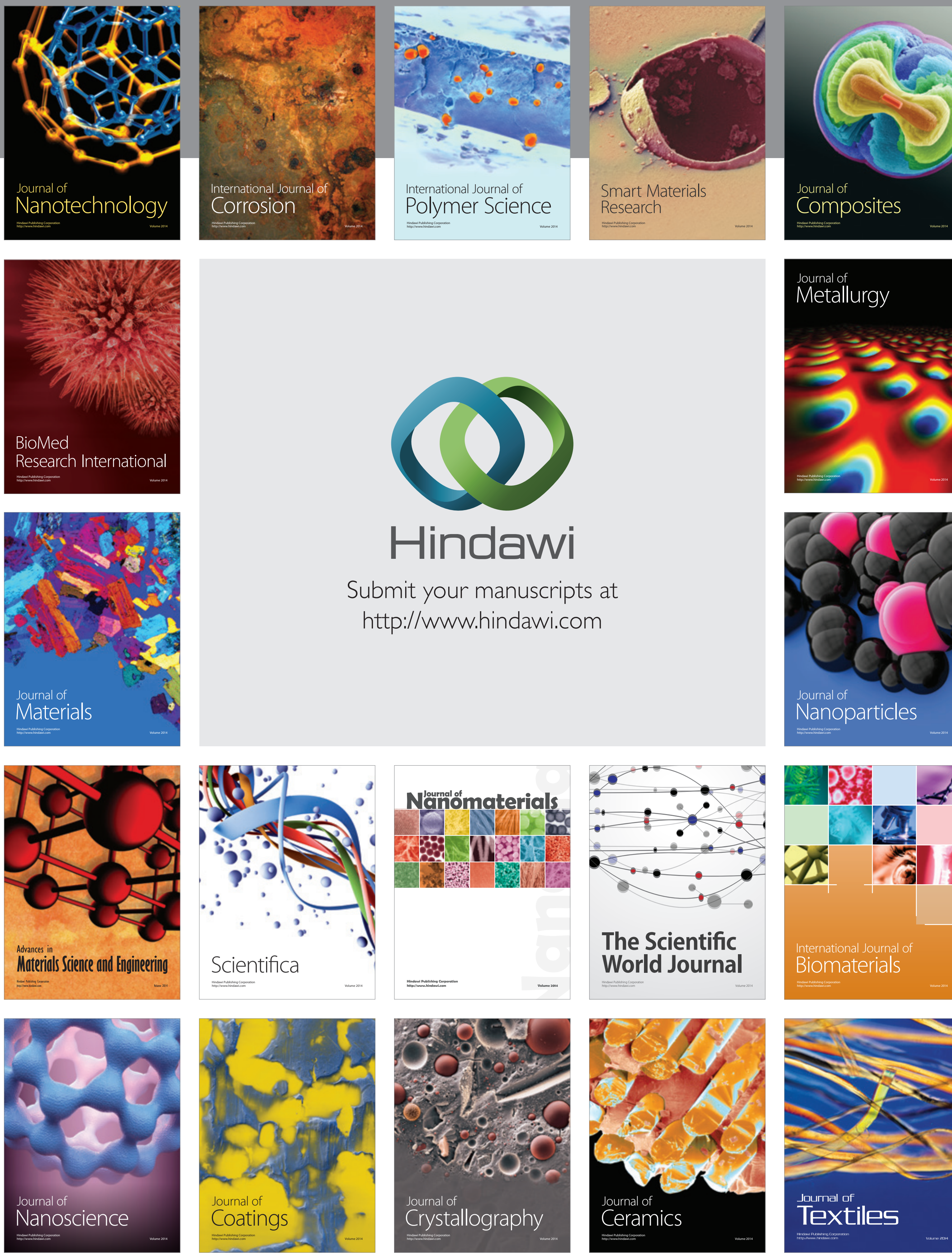
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