Influence of thermal shocks at high temperatures on microstructure and hardness of RENE 41 alloy

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Abstract. Rene 41, super alloy nickel base with aging hardening, presents an exceptional strength at high temperatures, find used at temperatures above 871°C. After aging, the microstructure of the alloy is made up of solid solution and precipitates and the mechanical properties show a significant variation with the aging temperature. The researches presented in this paper aims to determine the influence of thermal shocks at 1000 ° C on the alloy microstructure and the hardness. The heat shocks were applied in the surface, with the aid of solar energy, in 3 and 6 cycles of 30s. Samples were characterized, in surface and in section, by hardness measurements, scanning electron microscopy and EDX. The variations in the hardness characteristics were correlated with the structural transformations induced by the thermal shocks applied to the surface.

1. Introduction

Ni-based superalloys are characterized by high mechanical properties and high corrosion resistance at high temperatures. They Cr contain in large proportions ensures high resistance to oxidation at high temperatures. Co, Mo, Ti and other small quantities in addition elements controls the microstructure and mechanical properties. Small additions of B, C lead to the formation of borides and carbides. The microstructure of Rene 41 superalloy is made up of a high Y, with high concentrations of cobalt, chromium and molybdenum, with FCC network, and Ni3Al precipitated with FCC network, mostly coherent with the matrix. The secondary phases are distributed at the grain boundary [1]. Carbon-free elements, such as cobalt, influence carbide morphology and carbide distribution. Although MC solvus is well above 1200°C, MC particles degenerate to aging temperatures after the following reactions [2]:

\[
\text{matrix } \gamma \rightarrow \text{carbides} + \gamma^* \\
y^*(Ni, Cr, Al, Ti) + MC (Ti, Nb, C) \rightarrow Y'[Ni3 (Al, Ti)] + M_{23}C_6[(CrMo)_{23}C6]
\]

The process of separation of carbides depletes the neigboring regions of Cr and Mo.

Rene 41 is used for applications requiring high mechanical characteristics at high temperatures. It has a high resistance in the temperature range of 649-982°C: its traction resistance is 1420 MPa at ambient temperature, 621MPa at 871°C and 400MPa at 921°C [2, 3].
2. Material and experimental techniques

The chemical composition of the Rene 41 alloy is: C-0.04%, Cr-18.6%, Co-10.4%, Mo-9.3%, Fe 3.6%, Ti-3.3%, Al-1.7%, Si 0.1%, Mn-0.04%, Ni-balance. In the initial state, the alloy microstructure presents large polyhedron beads but precipitation at the grain boundary and inside them (figure 1).

Thermal shock testing was carried out in the PROMES Solar Furnace from Odeillo - Font Romeu, France [10], on parallelepiped samples with dimensions (10x10) of thickness 8 mm. The thermal cyclic was realized in air with 2 treatment regimes: 3 and 6 successive thermal shock cycles of 30s. The treatment diagrams are shown in figure 2.

![Figure 1](image1.png)

**Figure 1.** Rene 4 alloy initial microstructure, glyceregia attack.

![Figure 2](image2.png)

**Figure 2.** Thermal shock application charts at 1000 °C with a duration of 30 seconds for the Rene 41 alloy: a-3 cycles, b-6 cycles.

Samples are well characterized by EDX scanning electron microscopy and hardness measurements.

3. Experimental results

The microscopic analysis of the samples treated with 3 heat shock cycles highlights the layers formed by the application of thermal shock, with areas where spatial development is evident (figure 3, a and b).

Under the superficial layer, in the section, we see the formation of an area where the microstructure has a negligible number of precipitations (figure 4a), the number of them rising from the surface treated to the core of the sample (figure 4b).

According to the literature [1, 2, 6, 8, 11, 12], in the microstructure of nickel base superalloys coexist several types of carbides, distributed inside austenitic type matrix or dispersed in discontinuous and granular network at grain limit. Their distribution is dependent on the heat treatment parameters [9, 11-13]. Ramadan Kayacan and others [9] show that precipitation of $M_6C$ type carbides have the granular form, and those of the $M_{23}C_6$ type have the form of chains, the grain boundary network. The $M_6C$ type carbide solution is at 1149 °C and the $M_{23}C_6$ carbide is at 982 °C. [2]
Figure 3. The microstructure of the surface layer formed at the sample at 1000 °C with the shock duration of 30s, for 3 thermal shocks (a, b) and for 6 thermal shocks (c, d).

For samples subjected to 6 thermal shock cycles, the surface oxide layer is more continuous and overlapped (figure 3c and d).

Figure 4. Microstructure in the Rene 41 alloy sample section subjected to 3 shocks with 30 duration of the temperature 1000°C: a- near the surface, b-core.

Considering the temperature of 1000 °C of the thermal shocks applied to the surface and the short times, it can be appreciated that their application caused the modification of the alloy structure immediately beneath the superficial layer is done by matrix dissolution of (CrMo)3C6 carbides.

EDX characterization of the samples highlighted a strong diffusion of alumina to the surface, a phenomenon specific to aluminium - containing superalloys, and the formation of a layer composed mainly of aluminium oxide, in which the majority alloying elements are preserved in a proportion similar to that of the alloy, figure 5.
Figure 5. Alloy elements concentration profile after 3 thermal shocks at 1000°C.

Figure 6. Distribution of alloying elements in a precipitated area for the sample subjected to 6 thermal shock cycles, at 1000 °C, 30s.

The precipitated microstructure areas have high Mo, Cr and C concentrations relative to the nickel-rich matrix (figure 6). The proportion of alloying elements in the thick oxide layer respects that of the alloy composition (figure 7).

Figure 7. Alloy elements concentration profile after 6 thermal shocks at 1000°C, 30s.

The micro hardness of the sample with 3 thermal shocks shows a slight variation with the increase in hardness from the surface of the shock application, where there are few precipitations to the core (figure 8). For 6 cycles of thermal shock, there is a considerable cure of hardness (figure 9).
Figure 8. The hardness variation in relation with the treated surface of the sample subjected to 3 thermal shock cycles at 1000 °C.

Figure 9. Hardness variation in sample section for 6 thermal shock cycles, at 1000°C, 30s.

The phenomenon could be explained by carbides precipitation, the increase of the number of precipitates inside the grains and the increase of the continuity of the intergranular carbide network (figure 10) due to the decrease of the thermal transfer through the surface with the increase of the thickness of oxide layer.

Figure 10. Sample microstructure subjected to 6 cycles of thermal shocks.
4. Conclusions
Rene41 alloy samples were exposed to thermal shock cycles at 1000 °C. The characterization of the samples highlighted the characteristics of the oxide layers, the influence of the thermal shocks on the microstructure, the chemical composition and the alloy hardness.

The layers formed after three thermal shocks, discontinuous and thin, have high alumina content, well above the aluminium concentration in the alloy, which shows a potenti diffusion of aluminium to the surface. In case of the 6 cycles of thermal shock, the oxide layer grows in thickness and continuity, ultimately retaining a distribution of alloying elements similar to the alloy.

The application of 3 thermal shocks at high temperature, above the temperature stability of some complex carbides of the Rene alloy 41 causes their partial dissolution in the area immediately below the surface of the shock application, microstructural change that is associated with the decrease of the hardness, making a softening of the area. After 6 thermal shock cycles at 1000 °C, the hardness increases, especially in the core, by reducing of thermal transfer.

The experimental data were compared with literature results [4, 9, 11, 12].

The study of the effect of high temperatures shock on the Rene41 alloy by application of short thermal shock cycles, allows for a gradual assessment of the structural changes induced by the heating of the material in comparison with the long term treatment at high temperatures.

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