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ANALYSIS OF PITTING CORROSION ON AN INCONEL 718 ALLOY SUBMITTED TO AGING HEAT TREATMENT*

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Abstract

Inconel 718 is one of the most important superalloys, and it is mainly used in the aerospace field on account of its high mechanical strength, good resistance to fatigue and creep, good corrosion resistance and ability to operate continuously at elevated temperatures. In this work the resistance to pitting corrosion of a superalloy, Inconel 718, is analyzed before and after double aging heat treatment. The used heat treatment increases the creep resistance of the alloy, which usually is used up to 0.6 T_m. Samples were subjected to pitting corrosion tests in chloride-containing aqueous solution, according to ASTM-F746-04 and the procedure described by Yashiro *et al*. The results of these trials show that after heat treatment the superalloy presents higher corrosion resistance, i.e., the pitting corrosion currents of the as received surfaces are about 6 (six) times bigger (~0.15 mA) than those of double aged surfaces (~0.025 mA).
Keywords: Double aging heat treatment; Pitting corrosion; Inconel 718.

ANÁLISE DE CORROSÃO POR PITE NA LIGA INCONEL 718 SUBMETIDA A TRATAMENTO TÉRMICO DE ENVELHECIMENTO

Resumo

O Inconel 718 é uma das superligas mais importantes, sendo utilizada principalmente no setor aeroespacial devido a alta resistência mecânica, boa resistência a fadiga e fluência, boa resistência a corrosão e capaz de operar continuamente em temperaturas elevadas. Neste trabalho a resistência à corrosão por pite da superliga Inconel 718 é analisada antes e após o tratamento térmico de duplo envelhecimento. O tratamento térmico utilizado aumenta a resistência à fluência desta superliga, que normalmente é utilizada até 0,6 T_m. Amostras foram submetidas ao teste de corrosão por pite em solução de cloreto de sódio, de acordo com a norma ASTM-F746-04 e o procedimento descrito por Yashiro *et al*. Os resultados mostram que a superliga apresenta maior resistência a corrosão após o tratamento térmico, isto é, a corrente de corrosão por pite da condição como recebida é 6 vezes maior (~0,15mA) do que a envelhecida (~0,025 mA).
Palavras-chave: Duplo envelhecimento; Corrosão por pite; Inconel 718.

*Dedicated to the memory of Prof. Carlos de Moura Neto.

1 INTRODUCTION

Since 1930 nickel base superalloys are mainly used in aerospace applications, due to their high mechanical resistance, good creep, fatigue and corrosion properties and potential to work in elevated temperatures. One of the most important superalloys, Inconel 718, has been used in nuclear, cryogenic, oil and mostly aerospace industries. For example Inconel 718 is the backbone of jet turbines, both civil and military. This superalloy has a Ni-Fe-Cr matrix and alloying elements that produce secondary phases such as $Ni_3(Al, Ti)$ (γ') gamma prime; Ni_3Nb (γ") gamma double prime, also eta (η) hexagon close packed Ni_3Ti; delta (δ) orthorhombic Ni_3Nb and other topologically close packed phases such as Laves and μ. Heat treatments such as double aging promote increase in its structural stability by inducing the precipitation of γ' and γ", which confers the mechanical resistance of Inconel 718 [1,2].

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Due to the presence of chromium and aluminum and formation of their respective stable oxides, the passivation of Inconel 718 surfaces is achieved, conferring its characteristic corrosion resistance. Metals that form passive layers are susceptible to localized corrosion (pitting), which consists in the formation of short extension and deep cavities. This kind of corrosion initiates due to the break of passivation film, usually in defects such as inclusions, dislocations, grain boundaries or other interfaces.

This work studies the pitting corrosion potential of the superalloy Inconel 718, before and after double aging heat treatments. The samples were submitted to corrosion tests by adaptation of the methods of pitting corrosion potential suggested in Yashiro *et al*. [3] and ASTM F746-04 [4].

2 EXPERIMENTAL PROCEDURE

The superalloy Inconel 718 was furnished by Multialloy Co. Its chemical composition is described in Table 1. The material was produced via VIM/VAR, forging and solution treated, with an ASTM grain size of 8.20 [5].

The double aging heat treatment is described in Table 2. The first step is the solid solution, which promotes homogenization and redistribution of alloy elements and increases the grain size (ASTM grain size of 7.23). The aging step promotes the precipitation of secondary phases, mainly γ' and γ".

Conventional metallographic procedures of grinding and polishing were used, before each corrosion testing. For the corrosion test an electrolytic cell with three electrodes was used: a working electrode (Inconel 718 with an exposed surface of 0.785 cm^2), a reference electrode of Ag/AgCl and a counter electrode of platinum. The corrosion medium was a 3.5% wt NaCl aqueous solution, without stirring, at ambient temperature and pH 6.05. The corrosion tests were conducted using an AUTOLAB potentiostat/galvanostat, model PGSTAT30, interfaced to a microcomputer via the USB-IF030 interface and controlled by the GPES software. In order to characterize the pitting formation, the surfaces were analysed using a Tescan – Vega 3XMU electron microscope equipped with EDS system of microanalysis. In order to characterize the pitting formation, SEM/EDS analysis were done in as received and aged samples after cyclic voltammetry tests. In these tests the potential was varied from zero up to 3 V, and the current levels reached values up to 10^{-2} A/cm^2. Cyclic voltammetry and chronoamperometric experiments were conduct to determine the breakdown potentials (E_b), potential at which a significant increase in the current was observed, and the pitting potentials (E_p), respectively (each experiment was repeated 5 times). The cyclic voltammetry experiments were performed starting from the open circuit potential (OCP), after 01 hour of exposition to the electrolyte solution, varying the working electrode potential with 5 mV/s rate up to (OCP+400) mV, and reverting the potential to the starting one. The chronoamperometric experiments were also performed after 01 hour of exposition of each surface to the electrolyte solution, starting with an applied potential E_1 ($E_1 \approx E_b$ - 30 mV) and holding this potential for 120 s. Following this first experiment, the working electrode surface was freshly polished, exposed to the electrolyte solution for 01 hour, a potential $E_2 = (E_1 + 20mV)$ was applied and held for 120 s. Step 02 was repeated, increasing the applied potential at each step by 20 mV until and an increase in the current was observed.

Table 1. Chemical composition (wt. %) of Inconel 718

Inconel 718 (wt. %)								
Cr	Ni	Si	Mo	Ti	Nb	Fe	Al	C
18.94	54.47	0.04	1.35	1.05	5.89	17.99	0.27	0.03

Table 2. Double aging heat treatment steps

Heat treatment steps	Parameters
Solid solution	1095°C; 1.0 h/AC
Double aging step	955°C; 1.0 h/AC
	720°C; 6.5 h/FC
	720°C; 1.5 h/FC
	620°C; 8.0 h/AC

Where: AC – Air cooling; FC – Furnace cooling.

3 RESULTS AND DISCUSSION

Initially the procedure ASTMF746-04 was used to determine the pitting corrosion potential. Following this procedure, firstly the OCP is determined, after that the potential is shifted to (OCP+0.8) V and then the values of current against time are recorded. If localized corrosion is not stimulated within the initial 20 s, the potential is kept at +0.8 V (SCE) for an additional 15 min. If localized corrosion cannot be stimulated even afetr 15 min, the test is terminated, and one can only conclude that the critical pitting potential (E_p) is > (OCP+0.8) V. In order to precisely determine the critical pitting potencial the procedure described by Yashiro *et al*. [3], an chronoamperometric procedure, was then conducted. But before that the breakdown potential (E_b) was obtained by cyclic voltammetry. This potential was used as starting potential in the chronoamperometric experiments.

Figure 1 presents representative cyclic voltamograms of the as received and double aged Inconel 718 surfaces exposed to the chloride containing aqueous solutions. The breakdown potential of the as received Inconel 718 surfaces ($E_{b',(AR)}$) was (1.03±0.03) V while that of the double aged surfaces ($E_{b,(DA)}$) was (0.98±0.01) V.

The similar values of E_b potentials indicate that thermodynamically there is no significant difference in corrosion trend of Inconel 718, before and after the heat treatment.

Figures 2 and 3 show results of the chronoamperometric experiments performed for as received and double aged Inconel 718 surfaces, respectively, exposed to the sodium chloride solution.

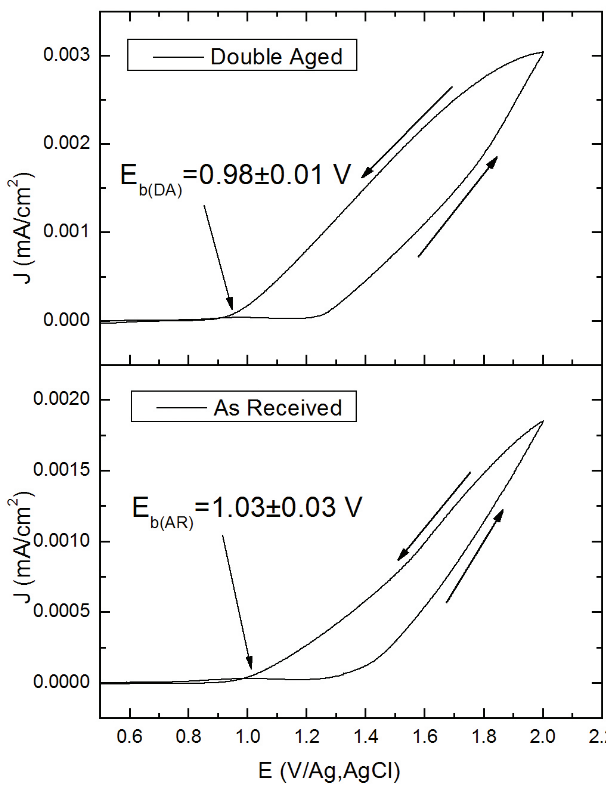

Figure 1. Cyclic voltamograms of double aged and as received Inconel 718 surfaces exposed to sodium chloride aqueous solution. Scan rate= 5 mV/s.

From the chronoamperometric results it can be observed that for the as received Inconel 718 the pitting corrosion potential is (1.21±0.03) V while for the double aged sample it is (1.13±0.01) V. The values of E_p potentials indicate that E_b is not a good parameter to decide over the thermodynamic pitting trend of Inconel 718 in chloride containing media. Despite the similar values of E_b of as received and double aged Inconel 718 surfaces, the pitting potential (E_p) of as received is nobler than that of the double aged surfaces. Therefore, thermodynamically, the double aged surfaces are more susceptible to pitting formation than the as received ones. Moreover, at the pitting corrosion potential, the induction time for pitting corrosion observed on double aged Inconel 718 surfaces is twice longer (~40 seconds) than that of the as received surfaces (~20 seconds). However, when the corrosion starts at the respective pitting potential, the pitting corrosion current on the as received surfaces is 6 (six) times higher (0.15 mA) than that of the double aged surfaces (0.025 mA). This means that when pit formation starts it is more severe in the as received Inconel 718 (solution treated) than in the double aged.

A previous study of the microstructure evolution [6] from the as received to the double aging condition on Inconel 718, performed in our laboratory, showed that after double aging, secondary phases basically composed by Ni/Ti and Ni/Nb, finely dispersed all over the Ni-Cr-Fe matrix, precipitates in Ni-Cr-Fe matrix, whereas in the as received microstructure clusters of Ti and Nb were observed. The formation of sigma (σ) and α-Cr, phases generally associated with chromium segregation, was not observed. As observed by Wlodek and Field [7], who analyzed Inconel 718 submitted to long time exposure to

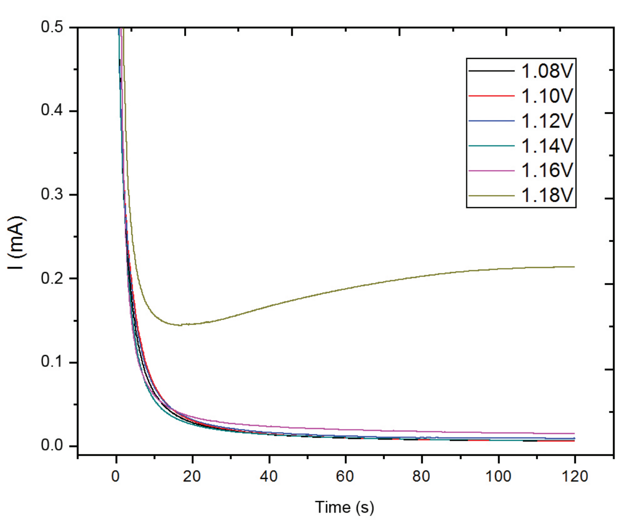

Figure 2. Chronoamperometric tests for as received Inconel 718 surfaces exposed to chloride containing aqueous solution.

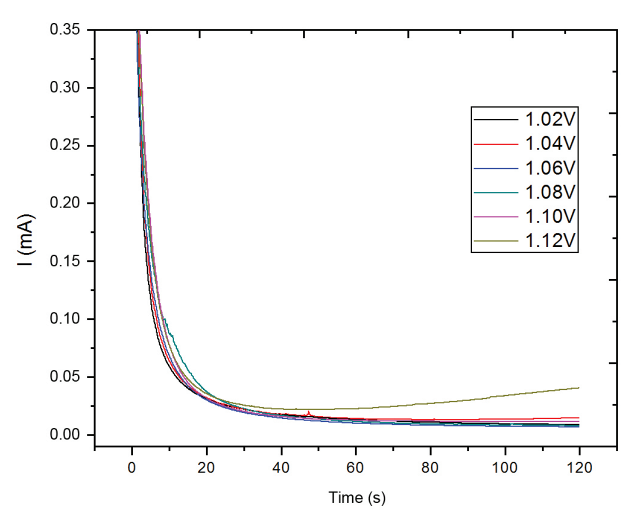

Figure 3. Chronoamperometric tests for double aged Inconel 718 surfaces exposed to chloride containing aqueous solution.

the temperature of 649°C, the formation of phases enriched in chromium (σ and α-Cr) can be observed only after long hours and their precipitation is associated with delta (δ) formation.

The pitting formation was investigated via SEM images after induction of pitting formation on cyclic voltammetry tests at same test conditions. The microstructure of the as received and double aged condition before corrosion tests can be found in [6]. Figure 4 shows one pit over the surface of Inconel 718, with approximately 50 μm diameter. The EDS map scan is also shown in this figure and reveals a depleted chromium zone at the border of the pit, while the same area is apparently enriched with molybdenum, niobium, titanium, aluminum, and oxygen.

Figure 5 details one pit near a grain boundary triple point on Inconel 718 double aged surface. The diameter of the pit showed in Figure 5, 8 μm approximately, is smaller than that of the pit in Figure 4 (as received condition). The pitting formation in both conditions represents the entire surface of the samples. The map scan performed on double aged sample also showed a depleted chromium and titanium zone at the border of the pit and an enrichment of this zone with aluminum, molybdenum, niobium and oxygen. For the analysis of double aged specimen surface area, the thick oxide layer covering most of the area had to be first removed by polishing to reveal the pit.

Therefore, the EDS map scans showed in Figures 4 and 5, allow to conclude that the pitting formation on as received Inconel 718 surfaces could be mainly associated to the low capability to form thick chromium oxide over these surfaces, while in the double aged surfaces, the pitting formation can be associated to the grain boundaries precipitated phases, specially of titanium rich phases, which preferentially oxidize together with chromium.

Similar research was previously conducted on Inconel 718 surfaces submitted to mill finishing and machine hammer peening [8]. In that research the authors concluded that the higher corrosion resistance of heat treated surfaces was due to the aging treatment itself, which promotes surface smoothing and larger compressive residual stress. Another aspect that could entail the different corrosion resistance on Inconel 718 before and after aging treatment could be related to the grain

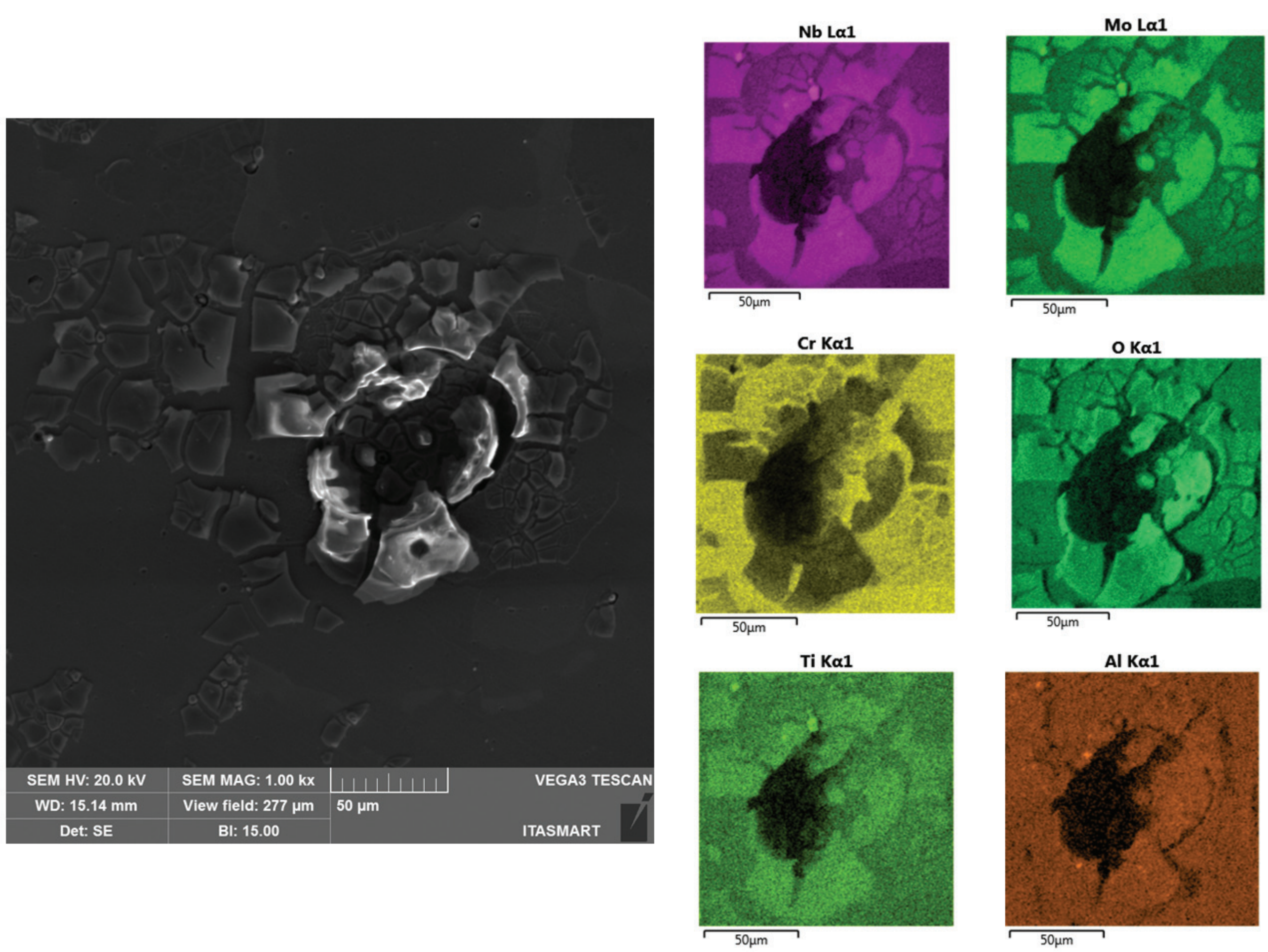

Figure 4. Image of a pit in Inconel 718 as received, and the EDS map scan of the main elements around this pit.

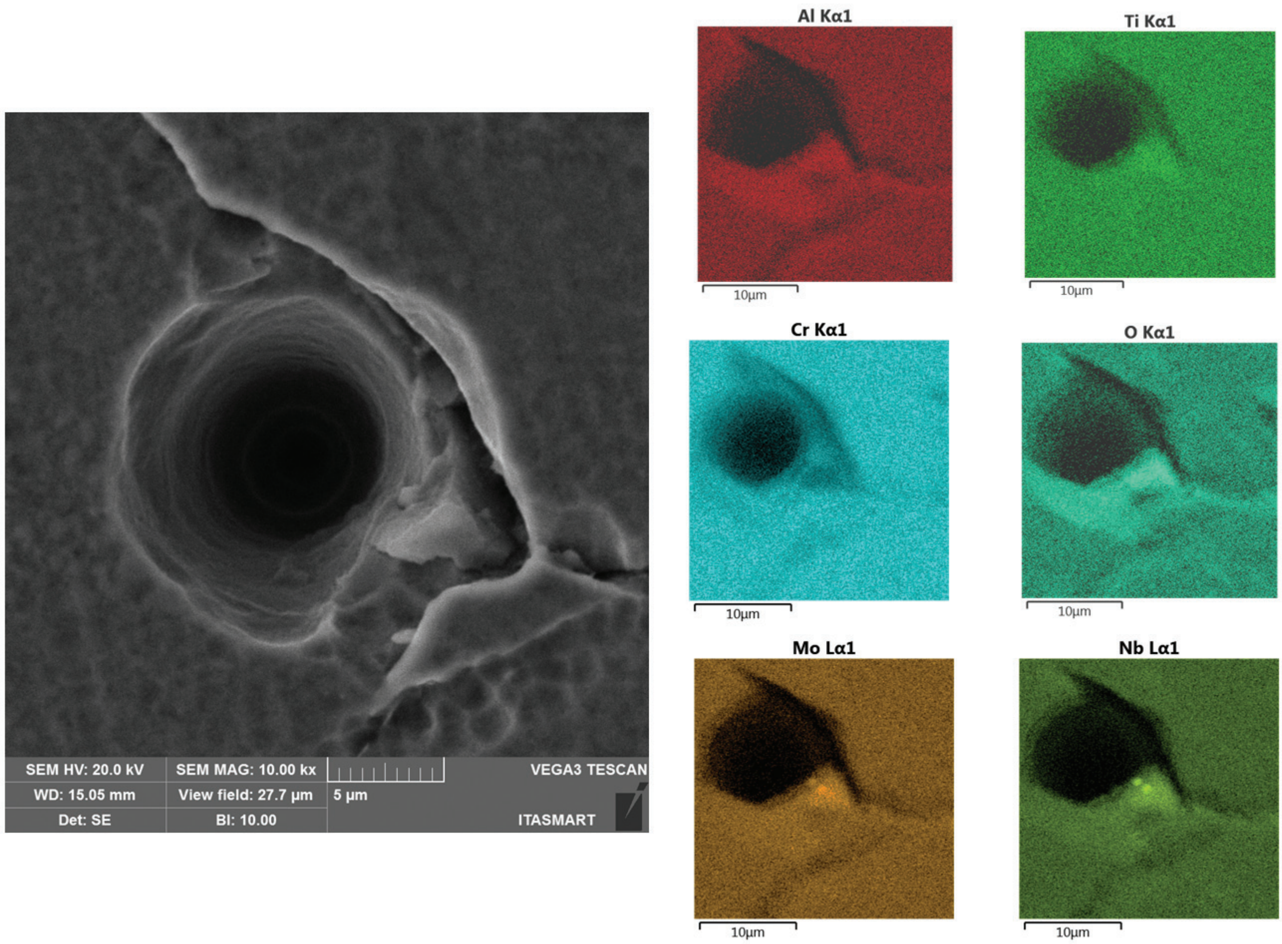

Figure 5. Image of a pit in Inconel 718 double aged, and the map scan of main elements around this pit.

size, which has increased from ASTM 8.20 (as received) to ASTM 7.23 (double aged). That means that the as received condition has larger area of grain boundary per unit volume.

4 CONCLUSIONS

From the presented results, it can be concluded that:

- The procedures suggested in standard ASTM F746-04 [4] and Yashiro *et al*. [3] separately are not adequate to obtain information about pitting corrosion potential of the Inconel 718 in chloride containing aqueous solution, probably due to the elevated corrosion resistance of this alloy. However, it is possible to obtain pitting corrosion information by combining the procedures suggested in these two standard procedures;
- The as received Inconel 718 has a pitting corrosion potential of (1.21±0.03) V, whereas the double aged condition has a potential of (1.13±0.01) V. However, when the pit formation starts the pitting corrosion rate on the as received Inconel 718 (solution treated) surface is higher than in double aged. The pitting corrosion currents on the as received surfaces are about 6 (six) times bigger (~0.15 mA) than on double aged surfaces (~0.025 mA). The lower pitting corrosion resistance of as received Inconel 718 is probably attributed to the larger grain boundary area per unit of volume. Besides that, after double aging this superalloy shows higher corrosion resistance probably due to surface smoothing, larger compressive residual stress and capability to form thick oxide layer. SEM/EDS results showed at the pitting border of both conditions a chromium depleted zone enriched with molybdenum, niobium, titanium and aluminum.

Acknowledgments

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