

CREEP RUPTURE PROPERTIES OF BARE AND COATED POLYCRYSTALLINE NICKEL-BASED SUPERALLOY RENE®80**M.M. Barjesteh ^{a,*}, S.M. Abbasi ^a, K. Zangeneh-Madar ^a, K. Shirvani ^b**^a Malek Ashtar University of Technology (MUT), Faculty of Material and Manufacturing Technologies, Tehran, Iran^b Iranian Research Organization for Science and Technology (IROST), Department of Advanced Materials and New Energies, Tehran, Iran

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Abstract

Creep deformation is one of the life time limiting reasons for gas turbine parts subjected to stresses at elevated temperatures. In this study, creep rupture behavior of uncoated and platinum-aluminide coated Rene®80 has been determined at 760°C/657 MPa, 871°C/343 MPa and 982°C/190 MPa in air. For this purpose, an initial layer of platinum with a thickness of 6µm was applied on the creep specimens. Subsequently, the aluminizing coatings were formed in the conventional pack cementation method via the Low Temperature-High Activity (LTHA) and High Temperature-Low Activity (HTLA) processes. Results of creep-rupture tests showed a decrease in resistance to creep rupture of coated specimen, compared to the uncoated ones. The reductions in rupture lives in LTHA and HTLA methods at 760°C/657 MPa, 871°C/343 MPa and 982 °C/190 MPa were almost (26% and 41.8%), (27.6% and 38.5%) and (22.4% and 40.3%), respectively, compared to the uncoated ones. However, the HTLA aluminizing method showed an intense reduction in creep life. Results of fractographic studies on coated and uncoated specimens indicated a combination of ductile and brittle failure mechanisms for all samples. Although the base failure mode in substrate was grain boundary voids, cracks initiated from coating at 760°C/657MPa and 871°C/343. No cracking in the coating was observed at 982°C/190MPa.

Keywords: Rene®80; Platinum-aluminide; Creep rupture; Larson-Miller parameter; Fractography**1. Introduction**

Cast polycrystalline nickel based superalloy Rene®80 (a well-known turbine blade alloy-GE-trademark), is extensively used for manufacture of rotating blades of aero-turbo jet engines. This superalloy is employed at temperatures between 760°C and 982°C. Many design applications depend on alloys that are subjected for a long time to high-temperature conditions. High temperature creep under elevated centrifugal stress leads to cracking of grain boundaries perpendicular to the principal stress axis that limits the life of the rotating blades. The temperature regime, for which creep is important in alloys is $0.5T_m < T < T_m$, where, T_m is the melting temperature of the alloys [1, 2].

Although the superalloys have comparatively favorite mechanical properties at elevated temperatures, their oxidation and corrosion resistance are not satisfactory at high temperatures. The aluminide diffusion coating is deposited on the surface of airfoil turbine blades in order to enhance the oxidation resistance of the base alloys at high

temperatures. Modification of diffusion aluminide coatings by platinum has been known to significantly improve their resistance to high-temperature oxidation [3-5]. To form the platinum-aluminide (Pt-Al) coatings, an initial layer 5–10 µm of platinum was electroplated, and then heat-treated to diffuse the platinum into the substrate prior to aluminizing. Diffusion coatings play a remarkable role in the mechanical behavior of the substrate at different temperatures. Effect of Pt–Al coating on the tensile properties of a nickel-base, single crystal superalloy was evaluated by Parlikar et al. [6]. The results of the mentioned work indicated that the application of Pt-Al coating on the superalloy led to a decline in the strength properties of the alloy (yield and ultimate tensile strength) at temperatures between ambient and 1100 °C. On the other hand, the coated superalloy exhibited more ductile behavior than the bare alloy. Influence of diffusion coatings on the creep life of the superalloy is equivocal and there are a few reports about the creep behavior of coated superalloys. Detrimental effect on creep life in Pt-Al coated AM1

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(a nickel-based superalloy) at the temperature range of 850-1100 °C has been reported by Ref [7]. While Haynes 75 and CMSX4 coated by Pt-Al, lean in Al, showed an increase in creep strength at lower temperatures, because of a higher volume fraction of the spread γ' ($\text{Ni}_3(\text{Al,Ti})$) phase (strengthening precipitates) than types that are richer in Al [8]. The contents of Al and Pt in Pt-Al coatings have a significant effect on The Ductile/Brittle Transition Temperature (DBTT). Depending on working temperature, below or above DBTT, negative or positive effects of the coating on mechanical properties of the base alloys are reported [9, 10].

Today, Turbine Inlet Temperature (TIT) has been increased by designers for improving the performance of turbojet engines. As a result, the creep behavior of coated rotary/stationary components, such as turbine blades and vanes, has been affected by temperature variations. It should be mentioned that creep tests measure the magnitude of creep strain as a function of time, while creep rupture tests measure the time to fracture for an assumption temperature and stress levels [11]. Although there are some investigations of the mechanical behavior of the bare nickel-based superalloy Rene®80 [12-14], no research has been done to study the effect of Pt-Aluminide coating on the creep behavior of this superalloy. In light of the above, in this study, the influence of the high and low activity Pt-Al coatings on the creep rupture life of Rene®80 has been evaluated.

2. Experimental procedure

The substrate superalloy utilized in this work was a cast nickel-based superalloy (Rene®80) with the nominal chemical composition of Ni-0.16C-13.81Cr-9.69Co-4.23Mo-4.02W-3.02Al-4.87Ti-0.12Fe-0.05Zr-0.05V-0.03Mn-0.02B-0.02Si (in wt. %).

Creep specimens were manufactured by machining cylindrical bars of 10 mm in diameter and 60 mm in length obtained from the investment casting according to Figure 1. Where R_z , R , d_0 , L_0 , d_1 , h , L_c , and L_t are required surface finish, radius, test piece width, original gauge length, width of gripped ends, length of gripped ends, parallel length, and total length of the test piece, respectively (DIN50125). It should be mentioned that to make sure there were no surficial (cracks, etc.) or internal (pores, etc.) defects, all specimens were inspected by the Fluorescent Penetration Inspection (FPI) and X-ray test method.

At the next task, all of the rods were solutionized at 1205 °C for 2 h and aged at 1095 °C for 4 h [15]. Both the solution and aging heat-treatments were performed in a vacuum furnace. Then, specimens were machined from the solutionized rods. Afterwards, some of the creep specimens were subjected to a two-procedure aging heat-treatment,

i.e., 2 h at 1050°C and after that 16 h at 845 °C, to attain the favorite γ - γ' micro-structure (fully heat treatment). The remaining specimens were used for preparation of the coated samples. After grit blasting and cleaning the specimens with acetone, an intermediate layer of nickel with a thickness of 1-2 μm was applied on the specimens for decreasing the negative effect of the chromium on the lack of adhesive property of platinum. Then, the specimens were electroplated with a layer of Pt. Before electroplating of Pt, the specimens were blasted with the alumina grits, and then cleaned with acetone and ethanol. Electroplating of Pt was conducted in an electrolyte solution containing P salt (di-nitro diamino platinum), calcium carbonate, sodium acetate, and distilled water at 90°C. The electroplating current was 0.2-0.4 A/dm² and the deposition rate was about 1 $\mu\text{m}/\text{h}$. Prior to electroplating, the pH value was adjusted about 10.5 [16]. In order to achieve a platinum layer with a thickness of 6 μm , the time of 360 min was considered for the plating process. The platinum layer was subjected to heat treatment at 1050°C for 2 hours to enhance cohesion and the distribution of the platinum into the substrate, followed by cooling the specimens in a furnace at 400°C and then air-cooled [17]. The conventional pack cementation method via the LTHA (at 750 °C for 4 h followed by post aluminizing for 2 h at 1050 °C) and the HTLA method (at 1050 °C for 2 h) used for aluminizing. Aluminizing packs in the current study were composed 2NH₄Cl-12Al- 86Al₂O₃ (wt %) and 1NH₄Cl-4Al-95Al₂O₃ (wt %) for LTHA and HTLA methods, respectively. Subsequently, a final aging treatment was performed at 845 °C for 16 h in a vacuum [15].

Microstructural characterization and fractography studies were conducted using a light microscope (LEICA MEF4A) and a Scanning Electron Microscope (SEM) Model Zeiss Supra 55 equipped with Energy Dispersive Spectroscopy (EDS) Oxford Model, both prior to performing creep tests (to ensure the quality of the coating) and after the tests (to investigate fracture surfaces). X-ray diffraction

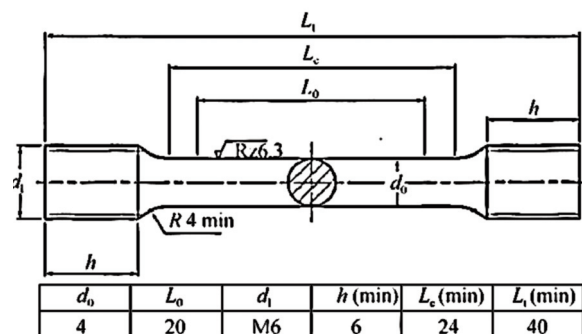


Figure 1. Dimensions of creep test specimen according to DIN50125 standard (mm) [2]

analysis was performed using an Inel Equinox 6000 with X'Pert High Score Plus v2.0, Cu K α 1 with Graphite monochromator, $2\theta = 16^\circ$ to 93° , to determine the distributions of different phases across the coating thickness and measure the residual stress of the coating. Micro-hardness testing was performed normal to the alignment of the Pt-Al coating and the substrate according to ASTM E384 using an automatic Akashi micro-hardness tester equipped with the Clemex software through applying 50gf. The creep rupture tests on the Pt-Al coated and uncoated specimens were undertaken uninterrupted per the ASTM E139 in the air and constant load, using an ATM CR-100KN machine equipped with an electrical furnace that could apply temperatures up to $1000^\circ\text{C} \pm 1$. The creep strains in the gauge section were measured by a linear variable differential transformer attached to an extensometer frame which was also attached to the creep specimen. The temperatures were measured by three thermocouples attached to the specimens. The test temperatures and nominal stresses applied were $760^\circ\text{C}/657\text{ MPa}$, $871^\circ\text{C}/343\text{ MPa}$ and $982^\circ\text{C}/190\text{ MPa}$. All testing started after a 30 min soak to ensure that the specimens obtained the required temperature.

3. Results and discussion

3.1. Microstructural characterization

Fig. 2(a) shows the optical microstructure of the polycrystalline cast alloy Rene[®]80, showing the typical dendritic structure. The inter-dendritic regions exhibited in the Fig. 2(b) at the higher magnification. These regions remained un-dissolved even after the solution treatment. The main microstructure of the Rene[®]80 consisted of a γ -phase matrix, carbides, borides, primary, and secondary γ' [18].

Microstructural studies have indicated that there are no changes in the cubic morphology, size and distribution of the $\gamma'(\text{Ni}_3(\text{Al,Ti}))$ phase (strengthening precipitates) in both coated and uncoated substrates (Fig. 2(c) and Fig. 2(d)). Microstructures consisted of dual primary cubic γ' with $460 \pm 40\text{ nm}$ in ledge size and the secondary spherical γ' with average diameter of $65 \pm 15\text{ nm}$. Volume percent of the primary γ' was 38 ± 4 and for secondary γ' was 12 ± 2 (measured with Clemex image analyzer v3.5). Hardness of the coated and uncoated substrates was measured as 404 ± 5 and $408 \pm 8\text{ HV}$, respectively. The average grain size of the coated and uncoated substrate was found to be $0.4\text{--}0.6\text{ mm}$ according to the ASTM E-112-96 standard.

Fig. 3(a) and Fig. 3(b) show the cross-sectional microstructures of the coatings used in the present study ($6\mu\text{m Pt/LTHA}$ and $6\mu\text{m Pt/HTLA}$). Both of the coatings exhibited the typical three-layer structure.

As shown in these figures, the outer layer

contained $\xi\text{-PtAl}_2$ (bright contrast) and $\beta\text{-(Ni, Pt)Al}$ (gray contrast). The presence of $\xi\text{-PtAl}_2$ and $\beta\text{-(Ni, Pt)Al}$ phases, in both aluminizing conditions, was also confirmed from XRD analysis (Fig. 4). The intermediate layer constituted single-phase $\beta\text{-(Ni,Pt)Al}$ and final zone named inter-diffusion zone (IDZ, the coating-base alloy interface). The thicknesses of the outer layer, the intermediate layer and IDZ were measured 44 ± 4 , 92 ± 7 and $4 \pm 1\text{ }\mu\text{m}$, for $6\mu\text{m Pt/LTHA}$ and 44 ± 4 , 39 ± 7 and $13 \pm 1\text{ }\mu\text{m}$, for $6\mu\text{m Pt/HTLA}$, respectively (three segments were evaluated, and the average quantity was announced). It can be observed that the $\beta\text{-(Ni,Pt)Al}$ zone in LTHA method was thicker than this layer in HTLA. The thickness of $\beta\text{-(Ni,Pt)Al}$ layer is strongly dependent on the Al availability in the pack as well as the duration of aluminizing. The thickness of IDZ layer was also reported to be lower in LTHA in comparison with HTLA.

Al, Ni and Pt EDS concentration line scan-profiles of these coatings ($6\mu\text{m Pt/HTLA/LTHA}$) are indicated in (Fig. 5 (a) and 5 (b)). Results of EDS line scan analysis are indicative of inward diffusion of Pt and Al and outward diffusion of Ni. As shown in these graphs, the surface Pt concentration is about 40-43 wt. % for both LTHA and HTLA methods. Based on the report by Pedraza et al. [17], at least 28 wt. % of platinum in the surface of Pt-Al coating is necessary to constitute a dual-phase structure. Chromium is one of the important elements that is present in the coating chemical composition. An increase in the chromium content led to the formation of Topologically Closed Packed TCP phases [19]. Regarding Fig. 5 (C), it is clear that the chromium concentration in $\beta\text{-(Ni,Pt)Al}$ layer and especially in IDZ is higher in HTLA aluminizing method than LTHA.

Hardness in the distance of $15\text{ }\mu\text{m}$ from the surface of $6\mu\text{m Pt/LTHA}$ coating was measured 1011 HV , while this value was measured 728 HV in the depth of $125\text{ }\mu\text{m}$ from the coating surface (Fig. 5 (d)). Hardness of $6\mu\text{m Pt/HTLA}$ coating in the same distance, was also measured 935 HV and 503 HV , respectively. This high value of hardness shows the brittle nature of Pt-Al coatings.

3.2. Creep rupture properties of coated and uncoated specimens

Creep rupture tests are evaluated on coated and uncoated specimens at three combinations of temperatures and stresses. The results, including failure time, tensile elongation and reduction in the area at the failure location are exhibited in Table 1. Creep rupture tests at different conditions were performed on two uncoated specimens (after full heat treatment, according to AMS5403 [15]) and two coated ($6\mu\text{m Pt/LTHA/HTLA}$) specimens. It should



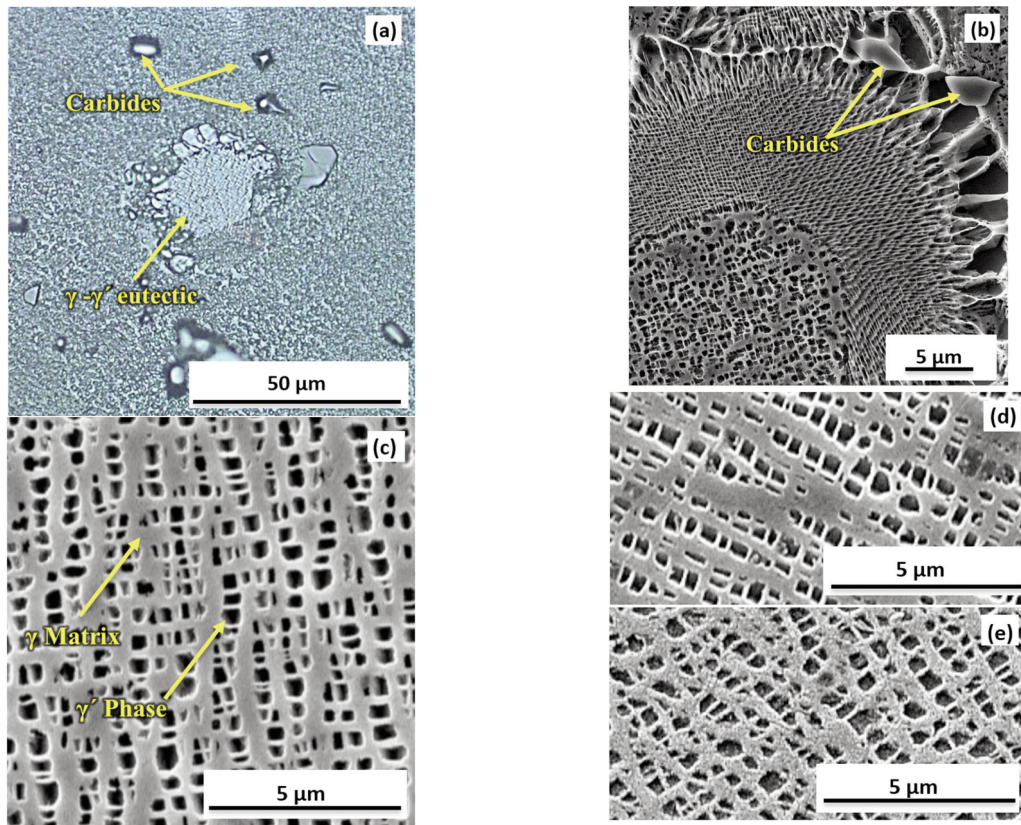


Figure 2. OM/SEM images of (a) γ - γ' eutectic region in the Rene[®]80. (b) Magnified view of γ - γ' eutectic region. (c) SEM image indicating the cubic γ' in γ matrix (uncoated). (d) Cubic γ' in γ matrix (coated/LTHA). (e) Cubic γ' in γ matrix (coated/HTLA)

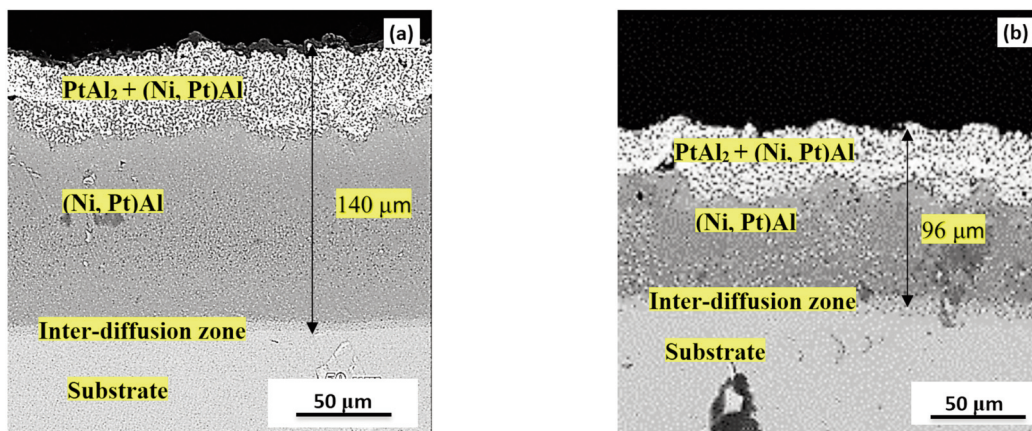


Figure 3. Backscattered SEM image showing Pt-Al coating for platinum layer thicknesses of 6 μ m (a) LTHA and (b) HTLA

be mentioned that in accordance with AMS 5403, minimum rupture life of 23h at 982°C/190 MPa is necessary for acceptance of uncoated Rene[®]80.

As it can be observed from the Table 1, the application of the Pt-Al coating in both aluminizing methods resulted in a decrease in the rupture life of Rene[®]80. The reductions in rupture lives in LTHA and HTLA methods at 760°C/657 MPa, 871°C/343 MPa and 982 °C/190 MPa were almost (26% and 41.8%),

(27.6% and 38.5%) and (22.4% and 40.3%), respectively as compared to the uncoated ones. Typical creep curves for bare and (6 μ m Pt/LTHA/HTLA) coated Rene[®]80 that were tested at different conditions are also shown in Fig. 6.

All of the curves were divided into three stages: I, primary or transient; II, secondary or constant rate, and III, tertiary [20]. During the test procedure, in stage I, when the creep stress was applied, a small

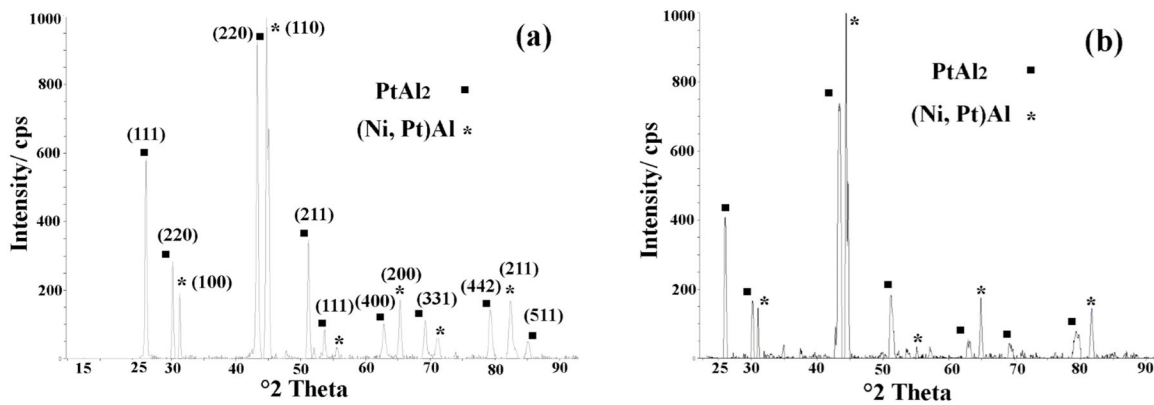


Figure 4. Picks of XRD analysis on Pt–Al coating for platinum layer thicknesses of 6 μm (a) LTHA and (b) HTLA

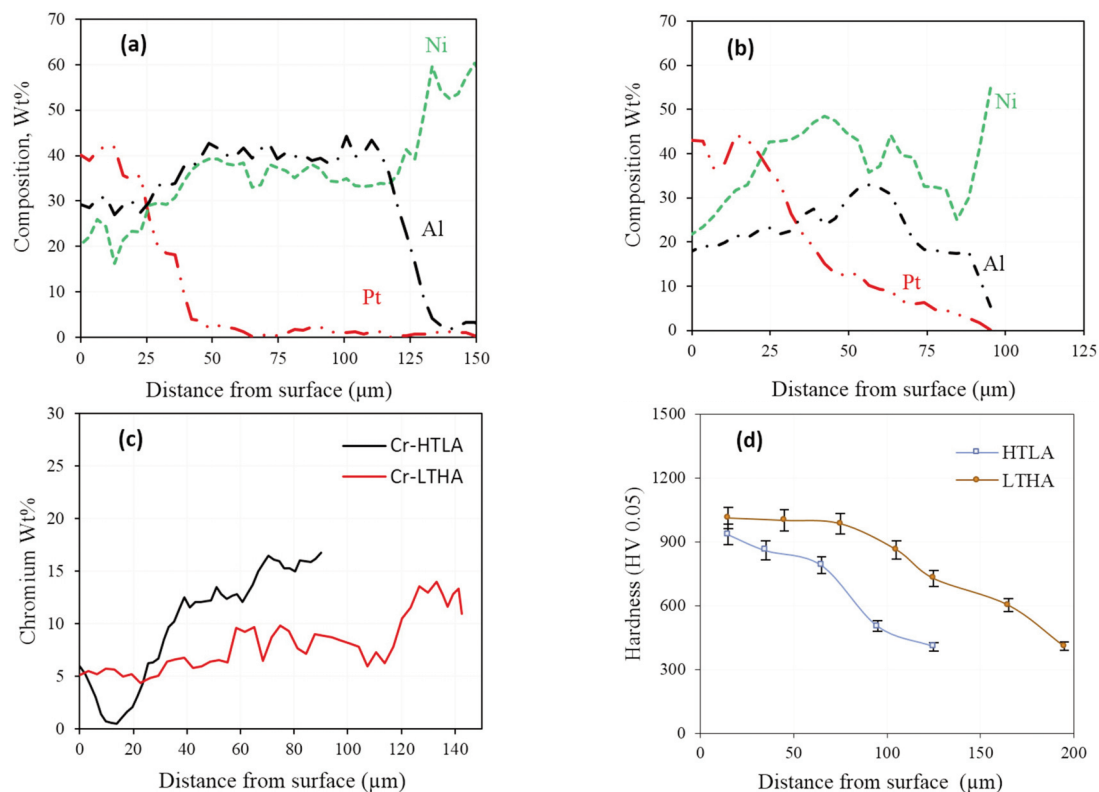


Figure 5. (a) Quantitative SEM EDS line scan profile of Pt, Al and Ni in 6 μm Pt/LTHA (b) the same as result in 6 μm Pt/HTLA (c) Comparison of variations of the chromium content across coatings created under 6 μm Pt/LTHA/HTLA (d) Variation of micro-hardness from surface to depth of present coatings

loading strain was created. After that, increasing strain led to a reduction in the creep rate. A constant value of the creep rate occurs at stage II. The steady state region showed the upper limit of time during the creep test because the strain rate was the lowest. Finally, in stage III, increasing the creep rate led to eventual rupture of the specimen (internal voids begin to form during this stage).

The Larsen– Miller Parameter (LMP) graphs versus the stress rupture properties of bare and coated (6 μm Pt/LTHA/HTLA) Rene[®]80 are shown in Fig. 7.

The Larson–Miller equation has the form $T (\log t_r + C) = \text{LMP}$ [20], where T is temperature in degrees Kelvin, t_r is time to rupture in hours, and the quantity C is specified as an alloy constant, but is roughly 20 for the superalloys [1]. Fig. 7 also shows a reduction in stress rupture properties of the coated alloy compared to the bare specimen, where the profile for the bare Rene[®]80 is located above that for the Pt–Aluminide coated Rene[®]80.

The decline in creep/stress rupture behavior of coated Rene[®]80 samples can be assigned to the

strength of the Pt-Al coating. Regarding to the existence of hard and brittle (Fig 5 (d)) phases of $PtAl_2$ and β -(Ni, Pt)Al (Fig. 4) in Pt-Al coatings, the fracture stress of the substrate is higher than that of the coating. The yield and ultimate strength of Pt-Al coatings have been measured 200 and 300 MPa at a temperature range of 800–900 °C, respectively by Alam et al. [21]. These values are lower than tensile properties of Rene®80 (UTS=701MPa and YS=590MPa [2]). Therefore, applied tensile stress causes some cracks on the coating. This phenomenon led to a decrease in the load bearing cross-section of coated creep specimens. The rest cross section will tolerate lower load and finally, coated specimens will be ruptured earlier than uncoated ones.

As it can be concluded from Fig. 7, the lowest rupture life belongs to the sample which was aluminized by HTLA method. This could be related to coating stoichiometric constitution, elemental diffusion in the coating/substrate and residual stresses.

The β -(Ni, Pt)Al phase has an important effect on the strength properties of the coatings [22]. The reason of that is related to the stoichiometry of the β -(Ni, Pt)Al phase and the resultant defect structure of this phase affect its strength characteristics. It is known that if the percentage of Al in the β -(Ni, Pt)Al composition is high, this phase will be hyper-stoichiometric (Al-rich). On the other hand, if the

percentage of Ni in this phase is high, intermediate layer will be hypo-stoichiometric (Ni-rich). Phase stoichiometry plays an effective role on the type of structural defects. Vacancy imperfections are more evident in hyper-stoichiometric coatings, while the

Table 1. Results of creep rupture test on coated and uncoated Rene®80 alloy at 982 °C /190 MPa

Test Conditions			Time (h)	%El	%RA
760±1 °C 657±2 MPa	Uncoated	Sample 1	58	8.5	6.2
		Sample 2	55.5	8	7.3
	6µm Pt/ LTHA	Sample 1	43	4.5	8.5
		Sample 2	41	4.2	8
	6µm Pt/ HTLA	Sample 1	34	3.8	6.2
		Sample 2	32	3.6	6
871±1 °C 343±2 MPa	Uncoated	Sample 1	100	8.3	16
		Sample 2	97	7.5	15.7
	6µm Pt/ LTHA	Sample 1	72	5.6	15.5
		Sample 2	70.5	5.5	15.5
	6µm Pt/ HTLA	Sample 1	61	5.2	12.3
		Sample 2	60	4.3	12.3
982±1 °C 190±2 MPa	Uncoated	Sample 1	35	7.3	18.5
		Sample 2	32	7	18
	6µm Pt/ LTHA	Sample 1	27	6.3	16.4
		Sample 2	25	6.2	16.2
	6µm Pt/ HTLA	Sample 1	21	5.5	13.7
		Sample 2	19	4.7	13.1

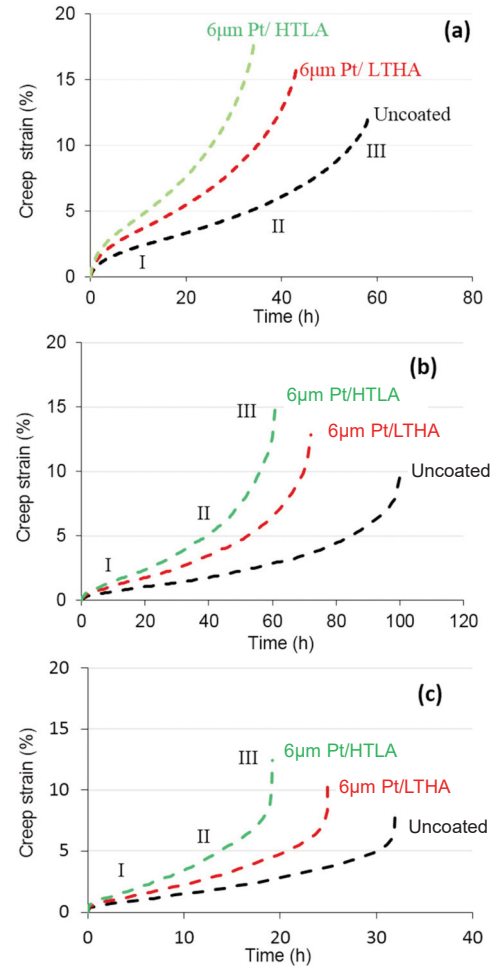


Figure 6. Creep rupture curve of uncoated and coated Rene®80 (a) 760°C/657 MPa; (b) 871°C/343 MPa and (c) 982°C/190 MPa

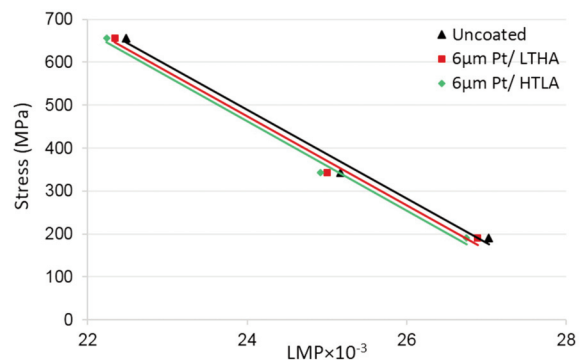


Figure 7. Larsen Miller Parameter (LMP) as a function of stress for air tested bare and two types of Pt-Al coated Rene®80

substitutional imperfections become visible in hypo-stoichiometric coatings [6, 22]. Vacancy defects show higher hardness in comparison with substitution defects [6]. According to Fig. 5 (d), the micro-hardness values of LTHA were greater than those of HTLA. Hence, it can be resulted that in LTHA method, β -(Ni, Pt)Al is hyper-stoichiometric and in HTLA method, this phase is hypo-stoichiometric. The strength of hyper-stoichiometric phases is more than hypo-stoichiometric [6, 21]. Thus, coating which has been formed by LTHA method is stronger than the HTLA coating and higher stresses can be tolerated by it.

Another reason for more reduction of the rupture life in HTLA compared to LTHA, is the diffusion of elements from the coatings to the base alloy (or from the substrate to the coatings) and the formation of deleterious phases. Some of the unfavorable phases the same as TCP (intermetallic phases), are formed across the IDZ. According to Fig. 3, it is seen that the thickness of the IDZ layer is thinner in LTHA. On the other side, as observed in Fig. 5 (c), the percentage of Cr in HTLA method is higher than LTHA. Cr can contribute to $\text{Cr}_{61}\text{Co}_{39}$ (the σ phase) formation and can also increase the DBTT. The solubility of elements Cr and Co is much lower in β -(Ni, Pt)Al and γ' than in γ , and therefore the precipitation of these elements in TCPs is predictable [19].

Residual stress was also measured in two conditions of HTLA and LTHA by XRD technique. XRD peaks were analyzed using Rietveld analysis. By consideration of the crystal properties of the present phases in the coatings, the elastic strain of ($6\mu\text{m Pt/LTHA}$) and ($6\mu\text{m Pt/HTLA}$) specimens were reached as 1.5×10^{-3} and 1.9×10^{-3} , respectively (with the help of X'pert high-score plus software and utilize of stress-less sample of Yttrium oxide). Regarding coating elastic modulus 99 and 93 GPa for LTHA and HTLA, respectively [21] and the application of Hook's Law ($\sigma = E\varepsilon$) [23], the value of the residual stresses were calculated as 148.2 MPa for LTHA and 176.7 MPa for HTLA, respectively. As seen, the value of residual stress in HTLA method is greater than LTHA, indicating another reason for the reduction in the rupture life in HTLA. Watanabe et al. [24] also have reported the value of residual stress equal to 140MPa for a high activity Pt-Al coating.

3.3. Fractography

The evaluation of fracture surfaces of coated and uncoated Rene[®]80 was performed by SEM. Fracture features of uncoated failed creep specimens at 760 °C/657 MPa and 982°C /190 MPa are shown in Fig. 8.

It should be mentioned that a similar fracture surface feature was observed in the coated samples.

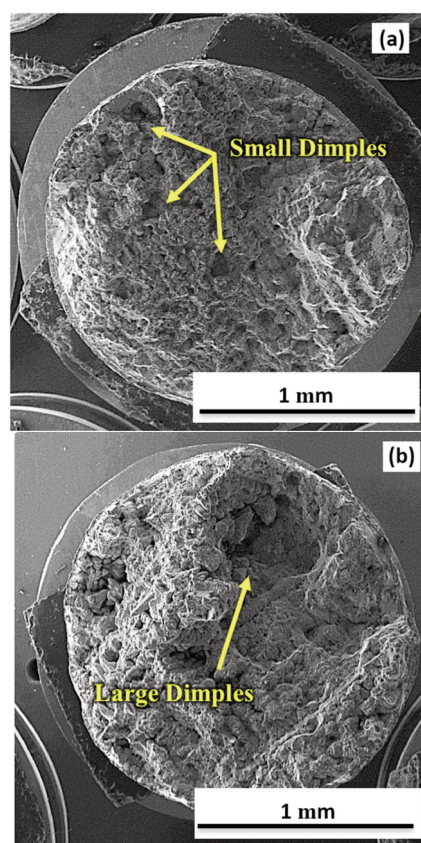


Figure 8. SEM images of uncoated Rene[®]80 creep rupture tested at (a) 760 °C/657 MPa (b) 982°C/190

As shown in Fig. 8, at 760 °C fracture surfaces of the substrate of the specimens showed small dimples, while at higher temperature (982°C), deep dimples were observed. On the other hand, the specimens (coated and uncoated) tested at 982°C/190 MPa indicated appreciable necking in comparison with the specimens that were tested at 760 °C/657 MPa and 871°C/343 MPa.

Fig. 9 shows the microstructure of IDZ layer in failed creep rupture samples in both aluminizing methods. The limited solubility of Cr as well as Co, Mo, W in the β -(Ni,Pt)Al phase produces precipitates of TCPs forming an interdiffusion zone (IDZ) exactly under the β -(Ni,Pt)Al layer. The degradation in the micro-structure of the coating/base alloy interface (IDZ) caused the reduction in creep life. Several related researches have reported the microstructural degradation of Pt-Al coatings which was started from IDZ [25, 26].

As shown in Fig. 9, the thickness of this layer in HTLA method is higher than LTHA. As previously mentioned, in HTLA aluminizing method, Ni diffuses outward to the surface rapidly. This phenomenon creates an obstacle against the diffusion of refractory elements such as Cr, Co, W, and Mo from the substrate to β -(Ni, Pt)Al. The limited solubility of Cr

as well as Co, Mo, and W in the β -(Ni, Pt)Al phase produces precipitates of TCP phases, forming a thicker inter-diffusion zone under the β -(Ni, Pt)Al phase layer in HTLA method. On the other hand, in LTHA aluminizing method, Al diffuses inward to the substrate. Substrate allowing elements such as Cr, Co, W and Mo are selectively diffusing at the coating/substrate interface. These elements can diffuse from IDZ to β -(Ni, Pt)Al, and therefore precipitation of TCP phases in inter-diffusion zone of the coating which has been manufactured by LTHA method is lower. Thus, a thinner inter-diffusion zone in LTHA method will be produced. EDS analyses of IDZ are provided and the results have been reported as 16 Cr, 9 Co, 6 Mo, 6.5 W, 6 Ti, 4 Al, 52.5Ni (in wt %) and 20.3 Cr, 9.9 Co, 5.3 Mo, 4.3 W, 3.0 Ti, 52.7 Ni, 2.5 Al (in wt %) for LTHA and HTLA methods, respectively. According to these results and also line scan EDS report Fig. 5 (c), it is clear that the percentage of Cr in the HTLA method is higher than LTHA. Therefore, the formation of needle-like σ - (Co, Cr) is more possible in the HTLA. This brittle phase may induce negative effects on the mechanical behavior of the superalloy, such as loss of ductility and reduce of creep strength. TCPs also play an effective role in stress concentrations within an alloy because of their structure [27]. Thus, another reason for the reduction in rupture life of HTLA coated Rene[®]80 samples can be attributed to the higher thickness and faster degradation in the micro-structure of the IDZ in HTLA method.

In order to determine the cracks nucleation locations and their growth direction in uncoated and coated Rene[®]80, longitudinal sections of ruptured samples were evaluated. As the creep rupture test was done at an elevated temperature the brittle and hard oxide film on the surface of the uncoated specimens could collaborate in the nucleation and propagation of

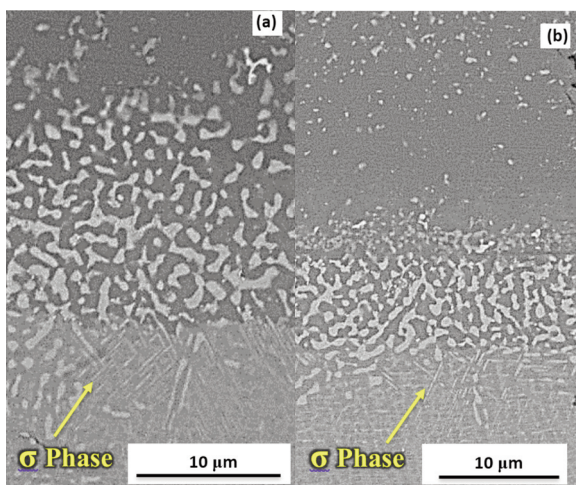


Figure 9. SEM images of IDZ microstructure (Pt-Al coatings) in fractured creep rupture specimens tested at 871 °C/343 MPa: (a) HTLA (b) LTHA

cracks. Optical micrographs of the fracture surfaces of specimens which were tested at 760 °C/657 MPa and 982 °C/190 MPa are provided in Fig 10(a) and Fig. 10 (b), respectively.

As evident from these figures, at both conditions the initial cracks nucleated from brittle oxide layer normal to the applied stress axis, and propagated through cavities, intergranularly. It is clear that the density and width of cracks at 982 °C/190 MPa was higher than that at 760 °C/657 MPa. Raising the thickness and quantity of cracks, led to a decrease in the cross section of load bearing. Consequently, the effective stress experienced by the specimens during the creep test increases and hence the rupture will occur earlier.

The longitudinal sections of the failed coated samples, at different conditions, are illustrated in Fig. 11. As shown in Fig. 11 (a) and (b), at 760 °C/657 MPa, in both aluminizing conditions (HTLA and LTHA), the crack nucleation started from the dual-phase zone of the coating on the alloy surface and grew within (Ni, Pt)Al zone, and entered the substrate once passed through IDZ zone. At 760 °C/657 MPa and in HTLA method, the crack density was higher than LTHA method. This was because the test

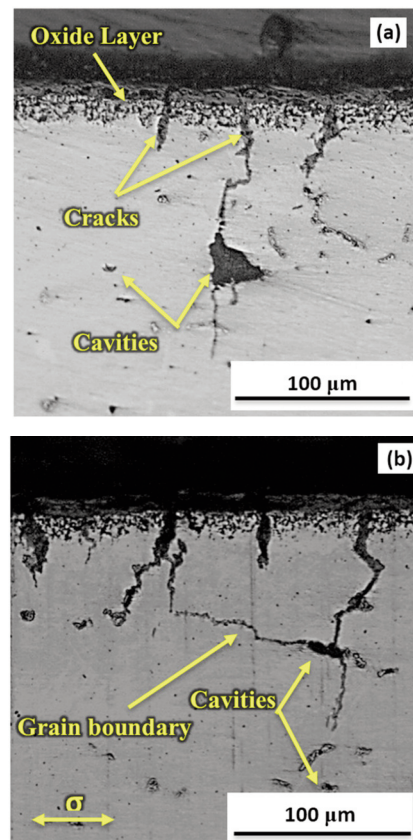


Figure 10. Optical images exhibiting voids and cracks in uncoated specimens after creep rupture testing at (a) 760 °C/657 MPa and (b) 982 °C/190 MPa

temperature was near the DBTT of the coating. Studies of Alam and his colleagues [28] showed the relationship between DBTT of Pt-Al coating and initial platinum layer thickness according to the following equation:

$$T_{DBTT} = 698 + 10 t_{Pt} \quad (1)$$

Where T_{DBTT} stands for ductile-to-brittle temperature ($^{\circ}\text{C}$) of the coating and t_{Pt} indicates the initial Pt layer thickness (in μm). By means of this equation, the DBTT of use coating was equal to 758°C . According to this calculation, the temperature

of 760°C , is too close to DBTT. Also, cleavage fracture (brittle mode) morphology with very small dimples (ductile mode) can be seen in Fig. 12 (a) and (b). This kind of fracture surface morphology approved that the testing temperature (760°C) was under or close to DBTT. The morphology of cleavage fracture (brittle fracture) in HTLA could be observed in the larger region. Under constant loading, the crack initiated from the coating and propagated across it very easily. On the other hand, the creep rupture failed in both coated and uncoated, took place fundamentally by formation, growth, and the coalescence of the voids created at eutectic γ' areas

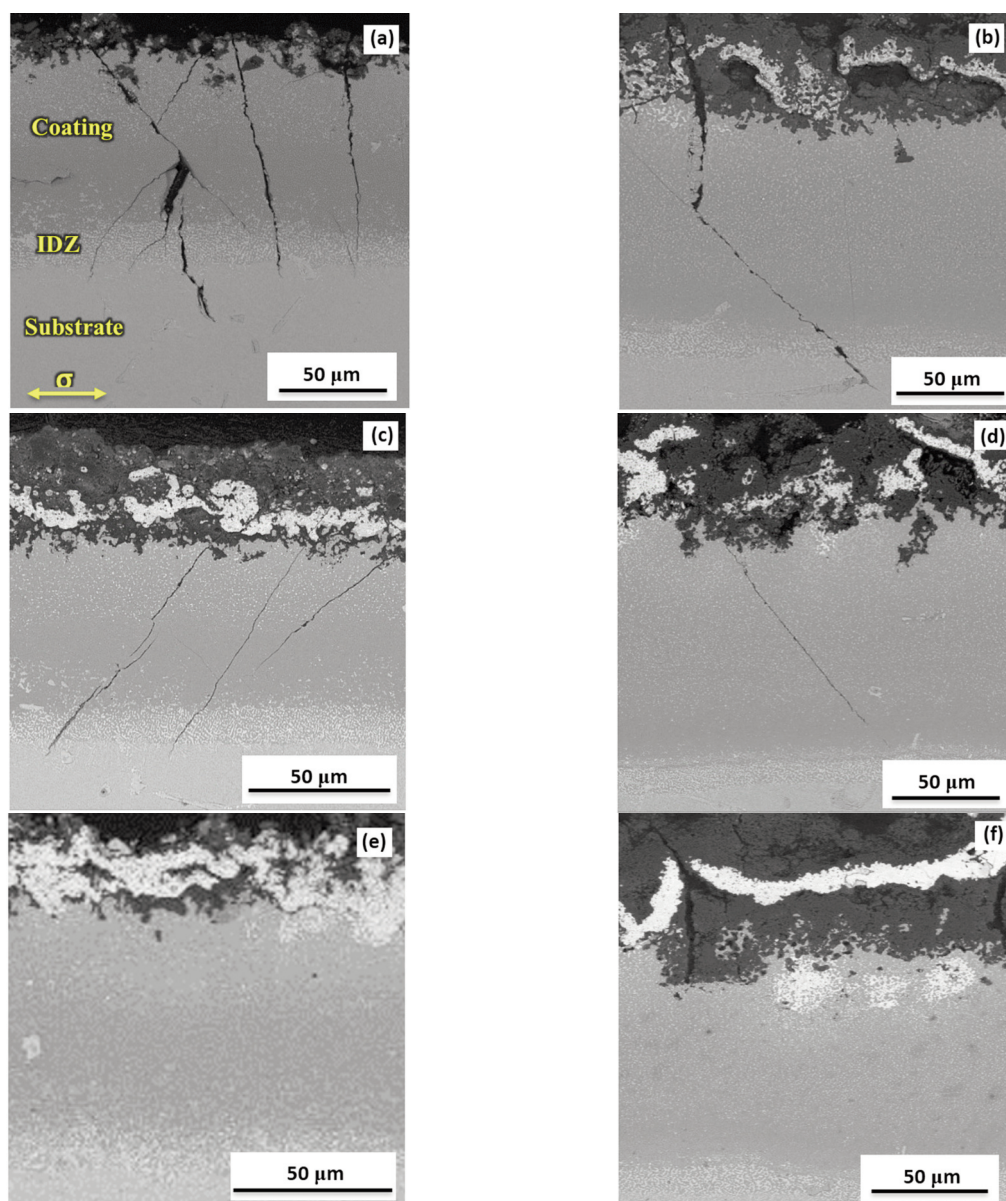


Figure 11. Backscattered SEM image of crack behavior in the coating after creep rupture test at (a) $760^{\circ}\text{C}/657\text{ MPa}$, in HTLA (b) $760^{\circ}\text{C}/657\text{ MPa}$, in LTHA (c) $871^{\circ}\text{C}/343\text{ MPa}$, in HTLA (d) $871^{\circ}\text{C}/343\text{ MPa}$, in LTHA (e) $982^{\circ}\text{C}/190\text{ MPa}$, in HTLA (f) $982^{\circ}\text{C}/190\text{ MPa}$, in LTHA

inside the base alloy (Fig. 10). Very suitable path for crack propagation will be provided by these voids. Fig. 11(c) and (d) show the longitudinal section of the failed as-coated specimens (HTLA/LTHA) that have been tested at 871 °C/343 MPa. As evident from these figures, although cracks initiated from coatings, in both aluminizing methods, cracks remained in the coating range in LTHA.

The presence of the cracks in the coating and no penetration of them to the substrate show that the creep rupture test was performed near the DBTT. Similar to the specimens which were tested at 760 °C/657 MPa, the fracture surface analysis of specimens that tested at 871 °C/343 MPa showed a mixed mode fracture morphology (cleavage and small dimples). Although the fracture surfaces of these specimens were similar, the cleavage area of the specimen which were coated in HTLA condition and tested at 760 °C/657, was larger than the others.

The fracture surface of failed coated specimens (HTLA/LTHA) that were tested at 982 °C/190 MPa are exhibited in Fig. 11 (e) and (f). As shown in these figures, no cracking in the coating was detected. The absence of any cracks in the coating region shows that the coating was ductile enough at the creep rupture test temperature of 982 °C to resist cracking up to fracture. It means that the test temperature was well above the DBTT. However, various voids had formed

within the IDZ of the coating by de-cohesion of precipitates from the matrix phase of the coating. Also, large dimples (ductile fracture) can be seen in the fracture surface of the specimens in both aluminizing methods (Fig. 12 (c) and (d)). A few cleavage facet planes were also noted. Although at 982 °C/190 MPa, both of HTLA and LTHA specimens showed a mixed mode fracture, the ductile area in both of them was greater than in the specimens which were tested at 760 °C/657 MPa and 871 °C/343 MPa.

As shown in Fig. 11, in comparison with LTHA, the number of cracks were higher in HTLA specimen that were tested at 760 °C/657 MPa and 871 °C/343 MPa. As mentioned previously, increasing the density of cracks led to a reduction in the cross section of load bearing, and hence the cracks can easily propagate toward the substrate.

On the other hand, grain-boundary weakening is generally responsible for the creep rupture failure of polycrystalline alloys [11]. As shown in Fig.10, not only cracks initiated from the oxide layer, but also some cavities were created along the grain boundaries. For polycrystalline superalloys (e.g. Rene®80) the addition of few ppm carbon, by producing carbides along grain boundaries, can improve the resistance of grain-boundary sliding [11]. According to the composition measurement by EDS two types of carbides were analyzed in Rene®80:

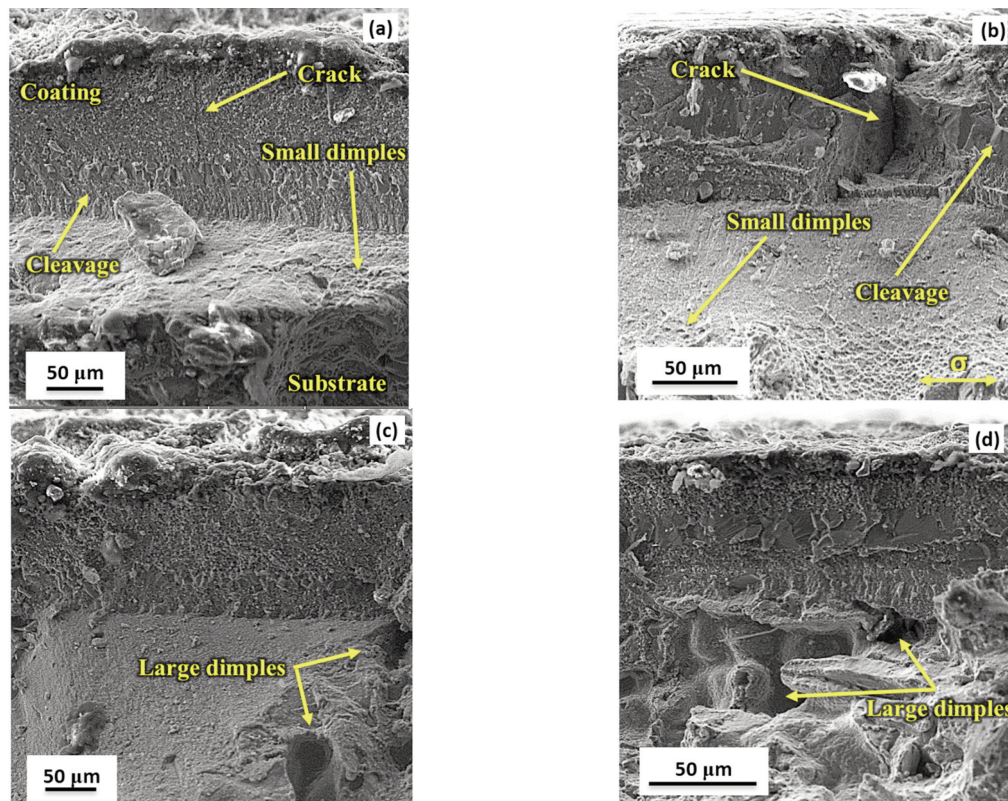


Figure 12. SEM images (Secondary electron mode) of Pt-Al coated specimens after creep rupture test (a) LTHA-760 °C/657 MPa (b) HTLA-760 °C/657 MPa (c) LTHA- 982 °C/190 MPa and (d) HTLA- 982 °C/190 MPa

(Cr,W)-rich $M_{23}C_6$ (with a chemical composition of 27.34Cr-4.55Co-17.5Mo-8.7W-5.3Al-4.7Ti-32Ni (in at. %)) and (Ti)-rich MC (with a chemical composition of 2.9Cr-0.9Co-11.5Mo-13.5W-2Al-64Ti-4.4Ni (in at. %)). $M_{23}C_6$ was only detected along the grain boundaries while MC was common both along the grain boundaries and inside the grains. During the coating process some alloy elements such as Ni, Cr, Ti, and W, diffuse from the substrate to the coating outwardly and Al and Pt diffuse from the coating to the base alloy inwardly. This phenomenon will continue during the creep test due to high temperature and enough time. Therefore, near the surface some grain boundaries became poor from Cr and W and carbides detached from the grain boundary. In this case, the grain boundary not only is a suitable site for void nucleation, but a preferential path for crack propagation as well. Therefore, the creep rupture life in the coated specimen is lower than in the uncoated ones. As seen in Fig. 5(c), the percentage of Cr in the composition of the coating in HTLA method is more than LTHA. It means that the distribution of $Cr_{23}C_6$ along the substrate grain boundaries in HTLA method is lower than LTHA. Thus, it can be expected that the creep rupture life of the substrate which has been coated by HTLA method tends to be more decline.

Despite the reduction in the creep properties of coated Rene®80, coatings are necessary to enable the superalloy aviation/industry parts to service at elevated temperatures in a modern gas turbine engine [29, 30]. If bare turbine blades are exposed to the operation condition of gas turbine engines, they will be damaged fast by several of the phenomena such as oxidation, hot corrosion, and heat damage. Therefore, the investigation of the influence of coatings on the mechanical properties of the base alloy (substrate) at high temperatures will enable the designer to choose an optimum coating that has the highest life under certain running conditions of turbine engines.

4. Conclusions

The experimental data of this work showed that the resistance to creep rupture decreased after applying the Pt-Al coating in both methods of LTHA and HTLA.

The fracture surface evaluation indicated that a mixed mode (ductile and brittle) of failure in both uncoated and coated alloy under all creep rupture conditions occurred. In the coated specimens, the dimples (ductile fracture) were observed more and larger in the fracture surface of LTHA specimen which was tested at 982 °C/190 MPa, and cleavage area (brittle fracture) was larger in fracture surface of HTLA specimen that was tested at 760 °C/657 MPa.

Although no cracking was observed in both HTLA

and LTHA coatings at 982 °C/190 MPa, several cracks were detected in both HTLA and LTHA at 760 °C/657 MPa. These cracks passed the IDZ and entered the substrate. Crack started from HTLA coating at 871 °C/343 MPa and entered the base alloy, while it initiated from LTHA coating and remained in the coating range.

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OSOBINE STATIČKE IZDRŽLJIVOSTI POLIKRISTALNE SUPERLEGURE RENE®80 NA BAZI NIKLA SA I BEZ PREMAZA

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Apstrakt

Deformacija puzanjem je jedan od razloga zašto delovi gasnih turbina koji su izloženi naprezanjima pri visokim temperaturama imaju ograničen vek trajanja. U ovom radu, ponašanje loma usled puzanja kod Rene®80 legure bez prevlake i sa Pt-aluminidskom prevlakom ispitivano je na 760°C/657 MPa, 871°C/343 MPa i 982°C/190 MPa u vazduhu. Za ovu svrhu, početni sloj platine primenjen na uzorcima puzanja je bio debljine 6µm. Naknadno je formirana aluminizacija konvencionalnom metodom cementacije putem niska temperatura-visoka aktivnost (LTHA) i visoka temperatura-niska aktivnost (HTLA) procesa. Rezultati testova puzanja pokazali su smanjenje otpornosti na lom usled puzanja kod uzoraka sa prevlakom u poređenju sa uzorcima bez prevlake. Smanjenje vremena do loma kod LTHA i HTLA metoda pri 760°C/657 MPa, 871°C/343 MPa i 982 °C/190 MPa bile su gotovo (26% i 41.8%), (27.6% i 38.5%) i (22.4% i 40.3%), pojedinačno, u poređenju sa uzorcima bez prevlaka. Ipak, HTLA metod aluminizacije pokazao je znatno smanjenje vremena puzanja. Rezultati fraktografskih ispitivanja na uzorcima sa i bez prevlaka ukazali su na kombinaciju krtoeg loma i duktilnog metoda otkazivanja za sve uzorke. Iako je osnovni način otkazivanja u supstratu nastajao zbog mikrošupljina između granica zrna, mikropukotine su su inicirane od prevlake pri 760°C/657MPa i 871°C/343 MPa. Pri 982°C/190MPa nije primećeno pucanje u prevlaci.

Ključne reči: Rene®80; Platinski aluminid; Lom usled puzanja; Larson-Miller parametar; Fraktografija

