



Original Research Article

Evaluation of Hot Corrosion of Hot Dip Aluminized Coated Superalloy IN738LC in Melted Na₂SO₄-25wt% NaCl Salt

Mohammad Sajjadnejad^{1,*} , Vahid Tavakoli Targhi² , Seyyed Mohammad Saleh Haghshenas³ 

¹ Department of Materials Engineering, School of Engineering, Yasouj University, Yasouj, Iran

² Department of Materials and Metallurgical Engineering, Amirkabir University of Technology (Tehran Polytechnic), Tehran, Iran

³ Department of Materials Science and Engineering, School of Engineering, Shiraz University, Shiraz, Iran

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ABSTRACT

Superalloy IN738LC is categorized as one of the most frequently utilized nickel base superalloys in the production of hot section components due to its multiphase microstructure maximizing its strength under elevated temperatures. In this study, a hot dip diffusion coating of aluminum was employed on the nickel-base superalloy Inconel 738LC substrate to enhance the hot corrosion resistance required for high-temperature applications, such as turbine blades. The aluminizing salt bath included Al powder with a particular composition, NaCl, KCl, Na₃AlF₆, and NaF. A thickness of about 48 μm was attained by applying the coating for 30 minutes at 720 °C. Bare and aluminized coated specimens were subjected to hot corrosion assessment in molten salt, with a composition of Na₂SO₄-25wt% NaCl at 720 °C being exposed for 60 and 140 hours. Scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD) were conducted on the coating sample to ensure the successful deposition of the hot dip aluminized layer. The aluminized sample exhibited excellent corrosion resistance owing to the formation of an Al₂O₃ layer, which meant that after 140 hours of testing; very little coating deterioration was detected. In contrast, the naked sample suffered severe degradation and showed poor hot corrosion resistance. It was thought that the aluminized sample's superior hot corrosion resistance resulted from the uniform and dense growth of an Al₂O₃ protective scale without any cracks on the superalloy surface

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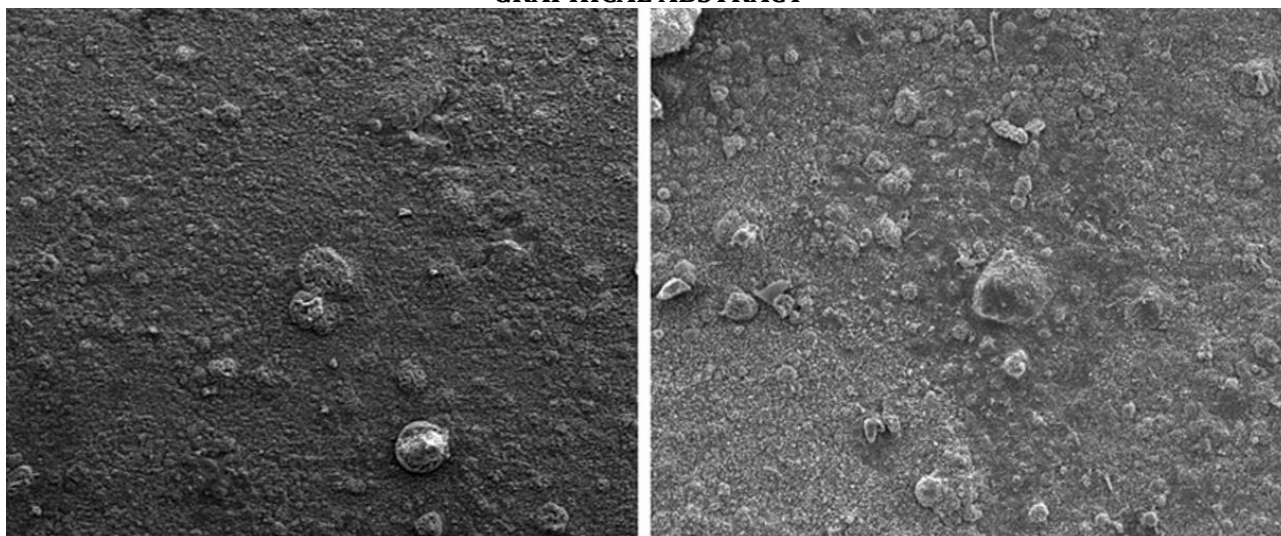
* Corresponding author: Mohammad Sajjadnejad

✉ E-mail: m.sajjadnejad@yahoo.com; m.sajjadnejad@yu.ac.ir

☎ Tel number: +989378730497

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GRAPHICAL ABSTRACT



Introduction

In recent decades, nickel and nickel-based superalloys have been extensively applied in critical multi-areas including aviation, space, chemical and petrochemical industries [1-4]. Because of their notable high strength at high temperatures, significant resistance to oxidation and corrosion, high creep strength, heat resistance, excellent processability, and remarkable weldability, nickel-based superalloys are well-known as a class of high-performance alloys [5-7]. Nickel-based superalloys have introduced themselves as the enhancer of the life and efficiency of turbine engines under harsh conditions imposed by the turbine environment. They are thus widely used in gas turbine blades and other parts because of their superior high-temperature mechanical qualities [8-12], and above all these segments must demonstrate a high level of oxidation and corrosion resistance under the combustion environment conditions [13-15]. To be more specific, thermochemical surface treatments have been broadly applied to provide oxidation resistance [16]. Superalloy IN738LC is one of the most frequently utilized nickel base superalloys in the production of hot section components [4,17-19]. IN738LC is well-

recognized for its multiphase microstructure, which enhances its strength at

high temperatures. This is achieved by the presence of small particles of the $L1_2$ type ordered γ' [$\text{Ni}_3(\text{Al,Ti})$] phase, which precipitate coherently inside the (FCC) γ matrix [20]. Surface coatings are broadly employed for these gamma prime materials, as the alloying requirements leads to a reduction in corrosion resistance. The primary concept of this technology involves choosing a substrate alloy with high strength to endure stress and applying a surface coating to provide optimal protection against corrosion from the environment [13]. Numerous techniques have been employed to fabricate a protective coating on Ni-based superalloys such as laser surface modification [21,22], high velocity oxy-fuel (HVOF) [23], ion implantation [24], electro-discharge coating [25,26], plasma spraying APS, LPPS, VPS, AXPS [27], and pack cementation [28]. The hot dip coating procedure is favorable among many surface modification methods due to its simplicity, speed, and cost-effectiveness [29]. Hot corrosion is a significant cause of failure in high temperature components

of essential technical systems, including aviation and engine-power turbines [30]. Several varieties of protective coatings have been shown to provide exceptional resistance to high temperature oxidation and hot corrosion [31-34]. Diffusion aluminide coatings are often used to effectively preserve jet engine components [35]. The development of an adhering layer of Al_2O_3 in diffusion aluminide coatings is a viable way for providing protection to super-alloys against corrosion. This method is widely utilized and highly desired [36-42]. An Al_2O_3 protective layer is well recognized for its superior oxidation resistance compared to other layers like Cr_2O_3 . In addition, it remains intact without flaking even at high temperatures approaching 1300 °C. Furthermore, Al_2O_3 is very likely to provide sufficient resistance to oxidation for nickel-base superalloys [43-45] and it serves as a diffusion barrier that shields the substrate from fast deterioration [46]. Several methods are being employed to fabricate such an aluminide layer including electroplating, thermal spray coatings, chemical vapor deposition (CVD), pack aluminizing, laser cladding [47], electron-beam powder bed fusion [48], Reactive air aluminizing (RAA) [49], and hot dip coating [16]. According to the results of the study done by Barwinska *et al.* [50], hardness, fatigue response, service life, and high-temperature corrosion resistance of Inconel 740 nickel alloy were significantly improved by aluminizing through the chemical vapor deposition (CVD) process. This involved the use of AlCl_3 vapors, a hydrogen protective atmosphere, the temperature of 1040 °C, 8 hours, and an internal pressure of 150 hPa. Mottaghi Golshan *et al.* [51] examined the laser process parameters, including laser power, laser scanning speed, and powder feeding rate, to determine their impact on the geometrical characteristics of aluminized Inconel 738 superalloy produced through laser cladding. The findings of this study could serve as a valuable reference for aluminizing In738 using laser

cladding technology. The cementation pack aluminizing process is often used to enhance the oxidation resistance of turbine blades made from Ni-based superalloys. The formation of a continuous, adherent, and slow-growing layer of $\alpha\text{-Al}_2\text{O}_3$ is considered as crucial and very effective in achieving significant corrosion resistance for aluminide coatings. Aluminizing the surface of a nickel-based superalloy is a well-established and successful technique for creating and maintaining a protective Al_2O_3 scale [52]. The $\beta\text{-NiAl}$ aluminide coating is susceptible to brittleness and sulfur-induced separation, mostly caused by grain boundary corrosion. This separation ultimately weakens the oxide-metal interface, leading to exfoliation [53]. Among the various techniques mentioned, Hot Dip Aluminizing (HDA) [54-58], is a diffusion coating method often used to provide high temperature oxidation- and corrosion-resistant coatings on stainless steels and low-alloy steels. The first use of the hot dip method for aluminizing Ni-base turbine blades occurred in 1952 [59]. The distinguished privileges of this technique are remarkable yield, low operating costs and significant applicability for large-size work-pieces [43,60] making hot-dip aluminizing a suitable technique [61,62], to develop a thick and adherent oxide layer of Al_2O_3 on the surface of aluminized components when they are subjected to an oxidizing environment [63-65] regulating the external infiltration of the alloying elements, while impeding the internal infiltration of corrosive substances [52].

Surveying the literature brings us about remarkable findings conducted on hot dip aluminizing alone and also modified aluminide coating type for hot corrosion resistance enhancement of Ni-based superalloys [66]. Accordingly, there have been scarce studies conducted on utilization of hot-dip aluminizing process through a solution containing Al powder and $\text{KCl/NaCl/NaF/Na}_3\text{AlF}_6$ molten salt. Thus, in this work, a novel hot-dip aluminizing process

has been employed to deposit aluminide coating on Ni-base superalloy Inconel 738. Finally, further characterizations including XRD, SEM and EDS are performed to investigate the hot corrosion behavior of the obtained specimens.

Experimental

Materials and specimens

A commercial, nickel-base superalloy, IN738LC, (15.96% Cr–1.63% Mo– 3.31% Ti–3.52% Al–0.69% Nb–1.83% Ta–3.08% W–8.53% Co–0.12% C, and Ni balance in wt.%), plate was used as the substrate material. Four specimens in the shape of a rectangle were cut to dimensions of 10×10×3 mm through a water-cooled cutting machine for corrosion tests. Before hot dip aluminizing, the specimens were degreased in an acetone bath subsequently cleaned by ultrasonic in an ethanol bath, and dried in air. The mixture of 12.0% aluminum powder and 88% salt (25.0% KCl–25.0% NaCl–28.0% Na₃AlF₆–10% NaF, in wt.%) was mixed and homogenized using a ball mill for 15 minutes. The mixture then was melted in an alumina crucible at 720 °C. Two of the specimens were hung by stainless steel wires and inserted into the crucible containing molten salt. After 30 min of insertion, the specimens were pulled out and air-cooled to room temperature. A mixed aqueous solution of nitric acid, phosphoric acid, and water in a 1:1:1 volume ratio at 25 °C was utilized to clean the hot-dipped specimen.

Hot corrosion tests

A supersaturated aqueous solution was prepared by mixing a corrosive medium consisting of a salt combination of Na₂SO₄-25%wt NaCl (in a ratio of 3:1) with distilled water. To achieve optimal adherence of the salt to the specimens, the samples were first subjected to a temperature of about 200 °C in an oven. Subsequently, the warm specimens were immersed in a supersaturated solution, resulting in the homogeneous deposition of a blended salt coating. The salt concentration on the surface of the samples was

maintained at an average of 3 mg/cm². The specimens were positioned and dehydrated in a furnace, thereafter subjected to a temperature of about 720 °C, and maintained for durations of 60 and 140 hours. The specimens were extracted from the furnace and, at intervals of 20 hours, their weight was measured and they were subjected to salt exposure and subsequent testing. During the last step, the objects were immersed in hot water to separate any remaining salts. Weight measurements were conducted to assess the rate of hot corrosion. Table 1 indicates the samples coding.

Table1. Coding of the samples used in this study

Sample	Corrosion time	Coating
A60	60 h	Aluminized
A140	140 h	Aluminized
B60	60 h	Bare
B140	140 h	Bare

Characterization methods

X-ray diffraction (XRD) using EQUIPOX3000 model (INEL Co. France) with Cu-K_α radiation ($\lambda = 1.540510 \text{ \AA}$) radiation was employed in order to identify the phase structures of the specimen surface. The X-ray diffraction (XRD) instrument tube operated at a voltage of 40 kilovolts (kV) and an electric current of 30 milliamperes (mA). The specimens were analyzed by using scanning electron microscopy (SEM) in conjunction with energy-dispersive spectrometry (EDS) to analyze their cross-sections and surface morphologies. The coating's thickness was determined by analyzing microscopic inspection and EDS data, which included graphing the concentrations of elements against the depth from the surface.

Results and discussion

XRD characterization

Figure 1 depicts the XRD patterns of the IN738LC alloy in its original state (bare) and after exposure to hot corrosion at a temperature of 720 °C for 60 hours (B60) and 140 hours (B140). Based on the acquired X-ray patterns, it is

evident that the surface of the B60 sample shows the presence of Cr_2O_3 , TiO_2 , and NiCr_2O_4 phases after 60 hours of corrosion. While Cr_2O_3 exhibits a high level of corrosion resistance, TiO_2 and NiCr_2O_4 show a relatively low level of resistance to corrosion [41].

However, the XRD spectra of the B140 sample do not even exhibit any peaks corresponding to the Cr_2O_3 phase. To be more specified, the existence of Cr_2O_3 peaks in the XRD pattern of the coating sample exposed to the corrosive molten salt for 60 h is not necessarily sufficient for withstanding the corrosion attack due to the appearance of TiO_2 and NiCr_2O_4 compounds possessing low levels of corrosion resistance, and this shaky corrosion resistance was worsened by exposing for 140 hours as a result of disappearance of Cr_2O_3 peaks. Consequently, bare IN738LC lacks the ability to withstand hot corrosion conditions. Figure 2 shows the XRD patterns of the aluminized coated samples before and after hot corrosion at 720 °C for 60 (A60) and 140 hours (A140). The existing peaks for aluminized

coating before the corrosion test, at 32, 45, 55, and 67 °C correspond to (100), (110), (111), and (200) planes, respectively, which is attributed to NiAl phase. The obtained XRD pattern is in good consistency with the obtained XRD spectrum of NiAl phase in Aghayar's research [67].

According to Figure 2, XRD patterns of the A60 specimen confirm the existence of Al_2O_3 and NiAl diffraction peaks on the surface of the sample. The existence of Al_2O_3 leads to good corrosion resistance. Likewise, there was no trace of any other phases representing the destruction of the oxide layer. Furthermore, the X-ray pattern of the A140 specimen shows the presence of the NiAl_2O_4 phase, that reflects a very mild destruction in the surface [68].

Microstructure analysis

Figure 3 clearly illustrates the cross-sectional BSE images of the aluminized coating prior to the hot corrosion investigation. A diffusion coating, about $48 \pm 0.2 \mu\text{m}$ thick, is present between the aluminum layer and the substrate. This is shown by the vertical green line in the illustration.

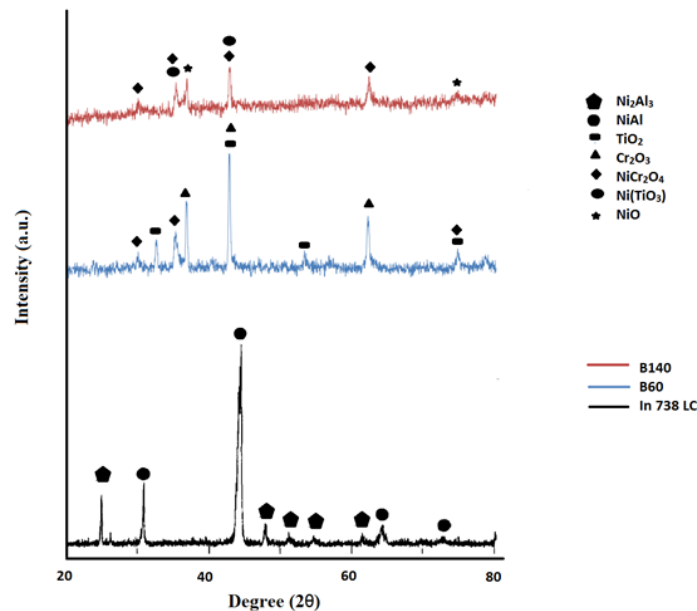


Fig 1. XRD patterns of the bare IN738LC before and after corrosion at 720 °C for 60 and 140 hours in the salt of Na_2SO_4 -25wt% NaCl

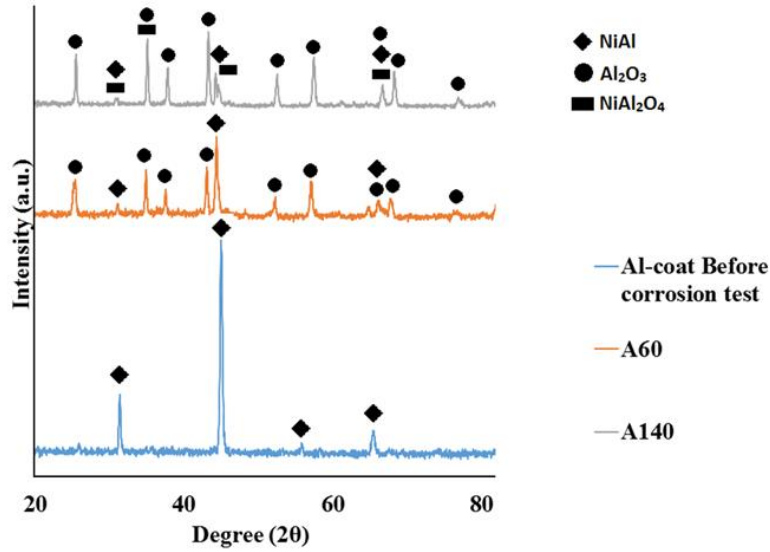


Fig 2.XRD patterns of the aluminized coating before and after corrosion at 720 °C for 60 and 140 hours in the salt of Na₂SO₄-25wt% NaCl

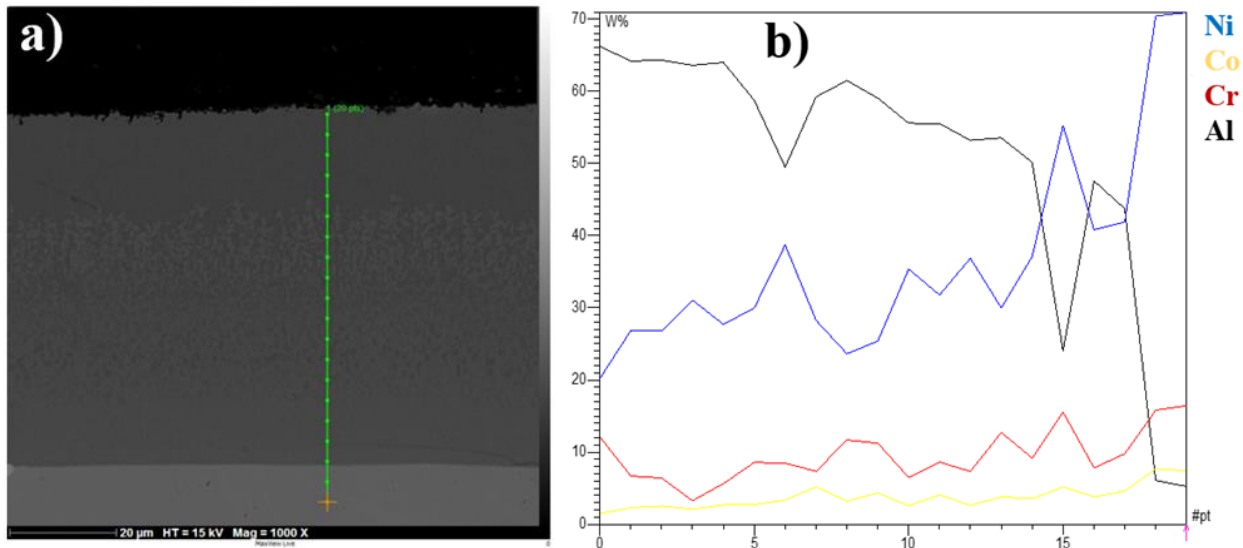


Fig 3.Cross-sectional BSE images of the aluminized coating specimens with a thickness of about 48 μm before the corrosion test

The coating consists of both exterior and interior zones. The interior region has a compact and homogeneous composition, while the surrounding region displays a porous composition. The figure demonstrates that the coating possesses a continuous and solid structure, with the major parts equally distributed and no visible fractures. In addition, the coating displays strong adherence to the

substrate. Figure 3 shows the diffusion curve over the coatings' thickness, indicating the outward diffusion of the substrate components. Spots 1-4, 5-17, and 18-20 corresponded to the aluminum topcoat, diffusion layer, and the IN738LC superalloy substrate, respectively. Table 2 displays the allocation and proportion of various constituents in the coating. There is 66% Al and 20% Ni in the exterior layer of the coating,

while in the coating-substrate interface, 44% Al and 42% Ni were found. X-ray diffraction of the coated specimen showed that the coating was composed mainly of one phase which was NiAl. Hence, this is in good agreement with the EDS results and Ni-Al phase diagram [69], which showed the downward diffusion of Al.

The NiAl synthesis on the substrate in hot-dip aluminize coating in the molten salt is facilitated by the generation of Al³⁺ ions in a mixture of molten chloride alkali metals, Al powder, and Na₃AlF₆ in which the reaction (1) would be written as below [70]:



The obtained Al⁺ ions can react with elements possessing higher levels of electronegativity. Since the substrate used in this study is a Ni-based superalloy, the NiAl phase could be obtained as a result of the reaction (2):

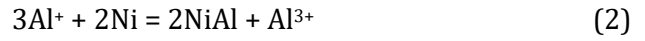


Figure 4a demonstrates the cross-sectional BSE image of the bare IN738LC before the hot corrosion test. According to the EDS results (Fig 4b), the white-colored deposit in the coating (point A) is mainly composed of Ti, Ta, and W. This also implies that the Al elements had diffused into the substrate.

Table 2. Distribution and the relative content of various elements in the coating

Distance from surface coating (μm)	Ni content (%)	Al content (%)	Cr content (%)	Co content (%)
0	20	66	12	1.5
10	27	64	5.6	2.6
20	39	49	8.5	3
30	37	53	7	2
40	41	47	8	4
47	42	44	9	5

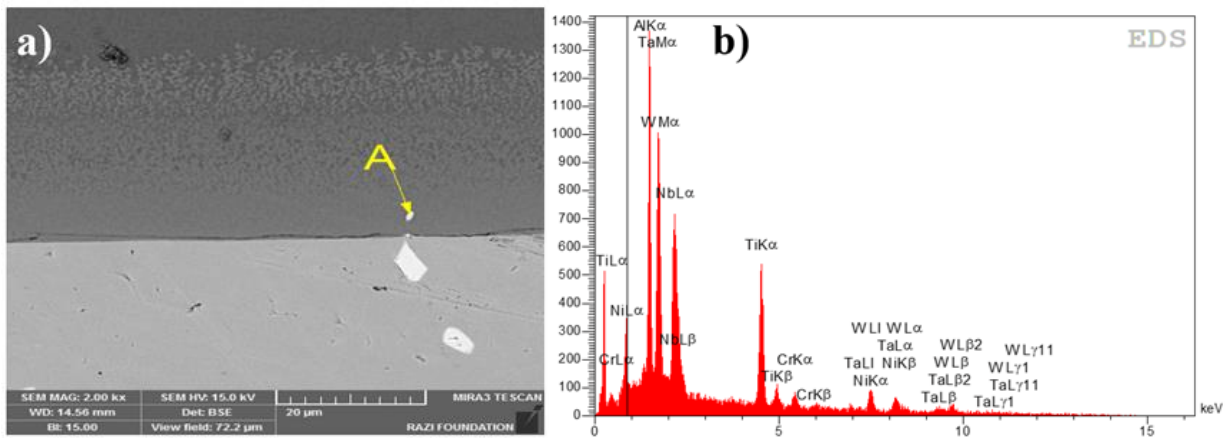


Fig 4. SEM images (a) and EDS analysis of A point (b) in bare IN738LC before the hot corrosion test

Hot corrosion behavior

Figure 5 displays the cross-sectional view of the A60 and A140 specimens. The coating and the interface between the coating and substrate remain intact, with no visible cracks even in the A140 sample. This indicates that the coating effectively protects IN738LC against hot corrosion. In addition, after 140 hours of heat

corrosion, the microstructure of the layers and coating surface became flattened. The EDS findings further indicate that the substrate components are well-shielded from hot corrosion due to the existence of a protective layer composed of Al₂O₃.

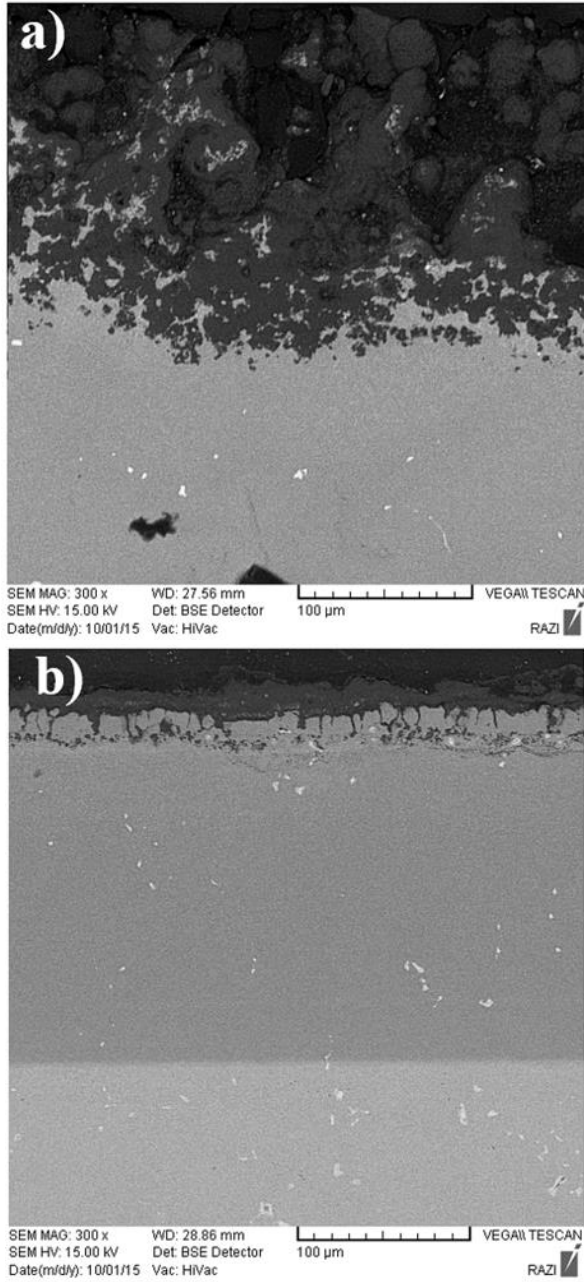


Fig 5. Line scanning images on the cross-sections of the aluminized coating specimen after corrosion at 720 °C for (a) 60 and (b) 140 hours in the salt of Na_2SO_4 -25wt% NaCl

Figure 6 depicts the cross-sectional view of the B60 and B140 specimens. Upon observing the sample surface after 60 hours of heat corrosion, it is evident that corrosion has occurred and the resulting corrosion product has suffered some minor damage.

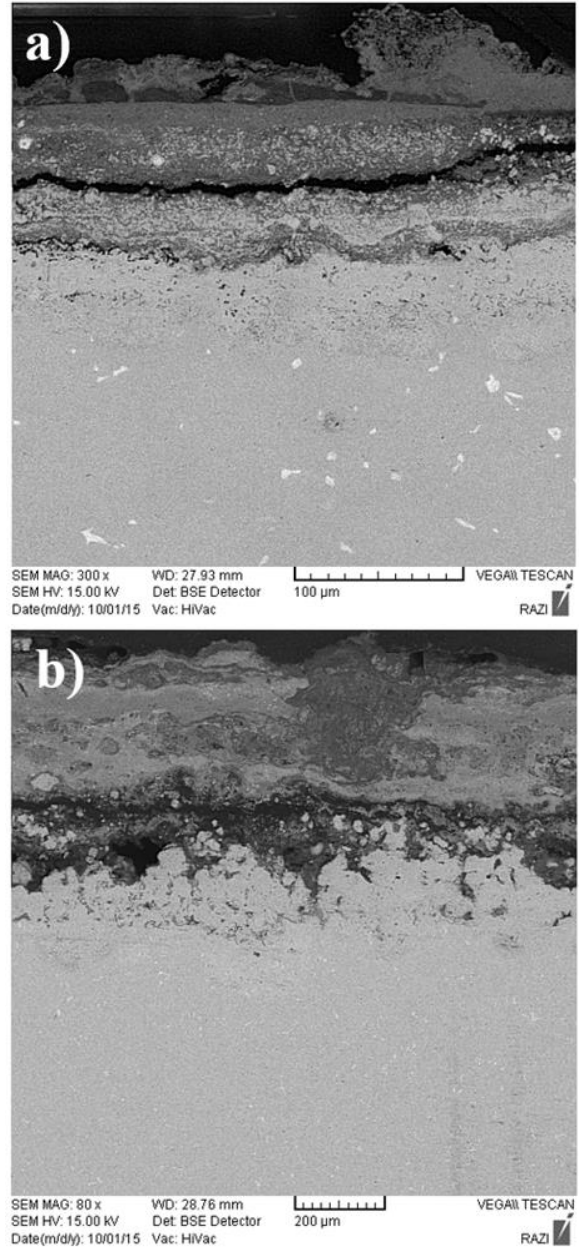


Fig 6. Cross-section of the hot corroded uncoated sample after corrosion at 720 °C for (a) 60 and (b) 140 hours in the salt of Na_2SO_4 -25wt% NaCl

Furthermore, when the corrosion period increases, the spread of corrosive components and the depth of corrosion diffusion progressively increase. The corrosion process involves the gradual production of layers of reaction products along the contact, resulting in the creation of a laminar structure. These layers exhibit rapid growth and are readily separated

from the surface. Moreover, the Energy Dispersive Spectroscopy (EDS) analysis of this sample reveals that essential components like Nickel (Ni) and Chromium (Cr) from the substrate are involved in the corrosion processes. The damage process is similar to thermal oxidation, but the presence of corrosive salts in molten form intensifies the damage, leading to the development of corrosion

phenomena. This intensified corrosion damage is clearly seen at the grain boundaries. Figure 7a and 7b show the SEM images of the A60 and A140 specimens. Obviously, the Al_2O_3 layer is formed after 60 hours which is consistent with the XRD analysis. By increasing the corrosion time to 140 h, no particular change was observed in the morphology; however, the oxide layer is marginally scaled.

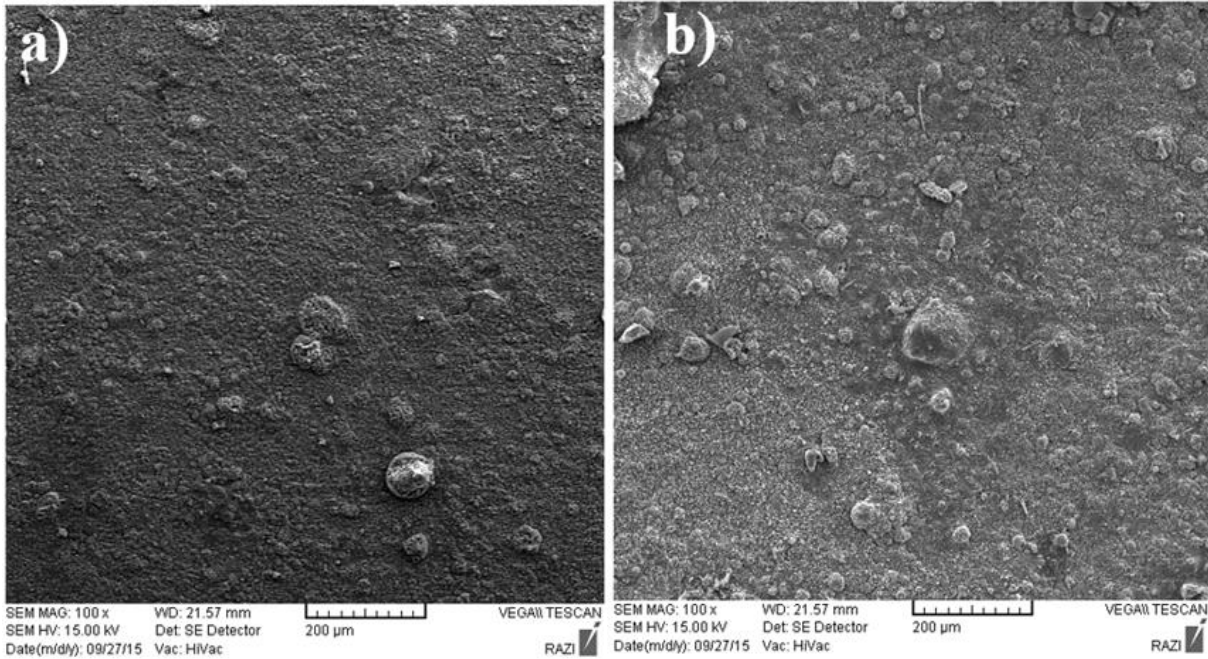


Fig 7. SEM images of the hot-dip aluminized sample after hot corrosion test for (a) 60 and (b) 140 hours

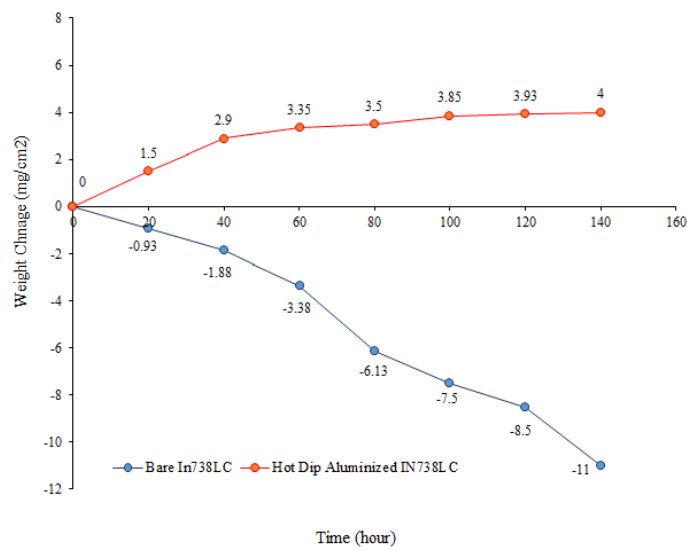


Fig 8. Weight change of hot corrosion test for coated and bare specimens

Corrosion kinetics

The hot corrosion kinetic figures of the bare and the aluminized coated IN738LC are illustrated in Figure 8.

The mass change involves an increase in mass (reaching a weight limit of 4 mg/cm² after 120 hours) due to the development of scales, and a decrease in mass (with a progressively steeper slope) caused by the shedding of scales. Therefore, the change in mass of the specimen in molten salt reflects the combined influence of these two processes.

The uncoated and the aluminized coated IN738LC alloy exhibit distinct corrosion characteristics when exposed to molten salt. According to the information shown in Figure 8, the IN738LC material exhibits poor resistance to heat corrosion, which may be related to the volatile nature of chloride. Due to the low melting temperatures and high vapor pressure of most metal chlorides, chlorine has a tendency to rapidly diffuse out through the oxide scale. It then reacts with alloying elements such as aluminum and chromium at the oxide/metal interface, resulting in the formation of volatile chlorides [41,71]. Moreover, the Cr₂O₃ creation might contribute to weight reduction due to the evaporation of chromium oxides and hydroxides, resulting in the loss of Cr from the surface. Moreover, the significant decrease in the curve might be attributed to the existence of low-melting-point phases located at the interfaces between grains, which serve as efficient pathways for diffusion [13]. The molten salt may quickly disperse and react with the alloy via these pathways. The use of an aluminized coating enhanced the alloy's resistance to heat corrosion. The first stage of exposure to the molten salt combination resulted in a significant rise in mass, reaching a maximum gain of 2.9 mg/cm² after about 40 hours. Subsequently, the mass gain stabilized at a relatively low level, indicating a stable state in the second stage. It may be inferred that during the second stage, the rate at

which the oxidation film grew was equal to the rate at which it peeled off.

Conclusion

An aluminized coating was applied to the Ni-based superalloy IN738LC by immersing it in a combination of Al powder and molten salt consisting of KCl, NaCl, NaF, and Na₃AlF₆. The molten salt facilitates the formation of a dense and homogeneous coating with a high concentration of aluminum. The data obtained show that the coating consists mostly of a NiAl matrix. Subsequent experiments revealed that the uncoated Ni-based superalloy IN738LC had a low resistance to corrosion in high-temperature corrosive conditions, while the aluminized coating, with a thickness of about 48 μm, exhibited exceptional resistance to corrosion in the Na₂SO₄-25wt% NaCl salt combination at 720 °C. This resistance may be attributed to the creation of the Al₂O₃ phase.

ORCID

Mohammad Sajjadnejad

<https://orcid.org/0000-0001-5112-1791>

Vahid Tavakoli Targhi

<https://orcid.org/0000-0002-3934-4682>

Seyyed Mohammad Saleh Haghshenas

<https://orcid.org/0000-0001-5878-8214>

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