A Study on the Cyclic Oxidation Behavior of Detonation-Gun-Sprayed Ni-5Al Coatings on Inconel-718 at 900 $^{\circ}$ C

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Cyclic oxidation behavior of detonation-gun-sprayed Ni-5Al coating on Inconel-718 is discussed in the present study. Oxidation studies were carried out on both bare and coated superalloy substrates in air at 900 °C for 100 cycles. The thermogravimetric technique was used to establish kinetics of oxidation. X-ray diffraction, FESEM/EDAX, and x-ray mapping techniques were used to analyze the oxidation products of bare and coated samples. The weight gain of bare superalloy was higher than the Ni-5Al-coated superalloy. Both bare and Ni-5Al-coated superalloys followed nearly parabolic oxidation behavior. The Ni-5Al coating was able to reduce the overall weight gain by 26.2% in comparison with bare superalloy in the given environment. The better oxidation resistance of Ni-5Al coating may be due the formation of protective oxides phases such as NiO, Al_2O_3 , and Ni Al_2O_4 on the oxidized coating and Cr_2O_3 at the coating-substrate interface. The Ni-5Al coatings obtained from detonation-gun-spraying process showed very little porosity and low surface roughness values.

Keywords coatings, D-gun, Ni-5Al, oxidation, superalloys

1. Introduction

Nickel-based superalloy Inconel-718 (In-718) is one of the most important and widely used alloys for high-temperature parts of gas and steam turbines, as it has excellent mechanical properties such as tensile strength, creep resistance, and low-cycle fatigue strength up to 650 \degree C. The alloy currently accounts for 45% of wrought nickel-based superalloy production (Ref [1\)](#page-8-0). However, the presence of high-temperature and combustion atmosphere with excess air or oxygen constitutes an extreme environment, and oxidation and/or hot corrosion is inevitable when alloys are used for long periods of time (Ref [2\)](#page-8-0). Oxidation is one of the most important high-temperature corrosion reactions (Ref [3](#page-8-0)). During oxidation attack, a porous, nonprotective oxide scale is formed at the surface, and the material is consumed at an unpredictably rapid rate, leading to premature failure of the components (Ref [4\)](#page-8-0). One of the effective solutions to counter the problem of oxidation is to coat the alloy with a protective layer using various surface-treatment techniques (Ref [5](#page-8-0), [6\)](#page-8-0). The coatings offer cost-effective ways to combat against corrosion and wear, without sacrificing the bulk properties of the material. Further, coatings can aggregate value to products up to 10 times their cost (Ref [7,](#page-8-0) [8](#page-8-0)).

Among the currently available thermal spray techniques, the detonation-gun (D-gun)-spraying process is one of the most promising and useful methods for depositing a wide variety of coatings to enhance the performance of materials in degrading environment such as corrosion and wear (Ref [8](#page-8-0), [9\)](#page-8-0). During D-gunspraying process, the powder particles are accelerated to very high velocities (800-1200 m/s), and as a result, there is minimum decomposition of feed powder, and the coatings obtained exhibit excellent morphologic features such as low porosity, and high density with fine grain structure along with superior mechanical properties such as high hardness and bond strength (Ref [10-13\)](#page-8-0). Senderowski et al. (Ref [14-19](#page-8-0)) have reported extensively on Fe-Al coatings deposited on NiAl and NiCr intermediate layers using D-gun-sparying process on carbon steel substrate material. They found that, D-gun technology has advantageous effects for spraying self-decomposing Fe-Al powders, and the deposited coatings were characterized by low porosity, high-temperature corrosion and oxidation resistance, reduced negative gradients of stress, and temperature influencing the substrate and increasing adhesion strength. Sundarajan et al. (Ref [8](#page-8-0)). and Kamal et al. (Ref [10](#page-8-0)) have reported that the porosity values of D-gun-sprayed coatings are less than that of the coatings deposited by other thermal spray techniques. Hence, the D-gun-spraying technique has been widely used in many fields such as gas and steamturbines, aero-engines, oil-refining industry, heat treatment rolls, and coal burning boiler tubes (Ref [20](#page-8-0)).

Nickel aluminides (Ni-Al) possess many attractive properties such as high melting point, excellent resistance to environmental degradation, low density, and good mechanical and chemical stability at high temperatures (Ref [21,](#page-9-0) [22\)](#page-9-0). Accordingly, Ni-Al have received considerable attention due to its potential for a wide range of engineering applications such as, high-pressure turbine blades and vanes, high-temperature corrosion-protective coatings, microelectronics, surface catalysts, high-current vacuum circuit breakers, and electronic metallization in semiconductor heterostructures (Ref [21-24](#page-9-0)). Thermal-sprayed Ni-5Al coatings are widely used as bond coat between the superalloy substrate and top

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coat due to its good oxidation and abrasion resistance at high temperatures (Ref [25](#page-9-0)). Ni-5Al coatings are also used as dense bond coats on gas turbine components over which abradable coatings are deposited. The abradable coatings have inherent porosity through which the corrosive species reach the bond coat, and hence the Ni-5Al coatings should have good hot corrosion and oxidation resistance (Ref [26\)](#page-9-0). In addition, the difference in the coefficients of thermal expansion of Ni-5Al intermetallic $(15.1 \times 10^{-6} \text{ m/m K})$ and Inconel-718 $(14.4 \times 10^{-6} \text{ m/m K})$ is very small, thereby reducing the chances of spallation and cracks at coating-substrate interface in high-temperature environment. Many researchers have evaluated hot corrosion and oxidation behaviors of Ni-Al coatings deposited using plasma and HVOF-spray process (Ref [25](#page-9-0), [27-29\)](#page-9-0). However, there is very little literature on the oxidation behavior of D-gun-sprayed Ni-Al coatings on superalloys. Therefore, in the present study, the high-temperature cyclic oxidation behavior of detonation-gun-sprayed Ni-5Al coating on nickel-based superalloy In-718 is discussed. Oxidation studies were carried out at 900 °C for 100 cycles in air under cyclic heating and cooling conditions on bare and coated superalloys. The cyclic oxidation studies create aggressive test conditions, which simulate the conditions prevailing in actual service environment of the superalloy components. The thermogravimetric technique was used to establish kinetics of oxidation. X-ray diffraction, FESEM/ EDAX and x-ray mapping techniques were used to analyze the oxidation products of bare and coated samples.

2. Experimental Procedure

2.1 Substrate Material and Coating Formulation

Nickel-based superalloy In-718 used in the present study was procured from Narendra Steels, Mumbai, (India), in rolled sheet form. The nominal chemical composition (wt.%) of the substrate material is given in the Table 1. Each specimen measuring approximately 20 mm \times 15 mm \times 5 mm was cut from the rolled sheet. The specimen were polished and grit blasted (grit 45) with alumina prior to D-gun coating. Commercially available Ni-Al powder of nominal composition Ni-5Al (wt.%) (MEC 906[®] Powder), supplied by Metallizing Equipment Company Pvt. Limited (MECPL) Jodhpur (India) was used in this study. The particle size of the Ni-5Al powder used was in the range of 50-80 μ m (Fig. 1). D-gun (Model: Awaaz) process was used to apply coatings on all six sides of the superalloy at SVX Powder M Surface Engineering Pvt. Ltd., New Delhi (India). The spray parameters used during coating deposition are given in Table [2](#page-2-0). All the process parameters, including the spray distance, were kept constant throughout the coating process.

2.2 Characterization of the Coatings

XRD analysis of the samples was carried out using a Bruker AXS D-8 Advance diffractometer with Cu K α radiation for the phase identification. The surface and cross-sectional analysis of the samples were analyzed using field emission scanning electron microscope (FESEM, FEI, Quanta 200F Company) with EDAX Genesis software attachment. The surface porosity of the coatings was measured with a Zeiss Axiovert 200MAT inverted optical microscope, fitted with imaging software Zeiss AxioVision release 4.1, which is developed based on ASTM B276. The cohesive and adhesive porosity values were assessed by photomicrograph quantitative analysis carried out with JEOL-JSM 5610LV (SEM). The cohesive porosity values were calculated based on the ratio of the sum of pore surfaces to the total surface of the specimen. On the other hand, the adhesive porosity (in the coating substrate joint) was defined as a ratio of the sum of the lengths of pore chords to the length of measuring length on the specimen surface (Ref [16\)](#page-8-0). The microhardness of the coating was measured using a Leitz miniload 2 (Germany) hardness tester. A load of 15 g (147.1 mN) was provided to the needle for penetration, and the hardness value was calculated from the relation $Hv = 1854.4 \times F/d^2$ (where "F" is load in grams and ''d'' is the mean penetrated diameter in micrometer). Surface roughness tester (Mitutoyo SJ-201, Japan) was used to measure the surface roughness (Ra) of the as-sprayed coatings at five different locations. The centre-line average (CLA) method was used to obtain the Ra values.

2.3 Oxidation Studies Under Cyclic Conditions

The oxidation studies on bare and Ni-5Al-coated In-718 superalloys were performed in air at $900 \degree C$. The specimens were kept in alumina boats and were subjected to oxidation studies for 100 cycles. Each cycle consisted of 1 hour heating at 900 °C followed by 25-min cooling at room temperature. The weight change measurements of the specimens were made at the end of each cycle using a microbalance (model BSA224S-CW, Sartorius) with a sensitivity of 0.1 mg. The thermogravimetric technique was used to establish kinetics of oxidation.

Fig. 1 SEM image showing the morphology of Ni-5Al powder

After exposure, the scale formed on the surface of the oxidized samples were characterized by XRD, FESEM/EDAX, and x-ray mapping techniques.

3. Results

3.1 Surface SEM/EDAX Analysis of As-sprayed Coating

The SEM micrographs of the as-sprayed Ni-5Al coating deposited using D-gun-spraying process are shown in Fig. 2. It is observed that the coating is dense and uniform, with very little surface porosity (Fig. 2a). The dense microstructure of the coating can be attributed to the high velocity of impinging molten powder particles during D-gun spraying. Further, the cross-sectional morphology of coating shows characteristic lamellar structure of splats (Fig. 2b) obtained by D-gunspraying process. The splats are rich in Ni with little Al and negligible amount of oxygen (O), while the intersplat regions

Spray parameters	
Acetylene (C_2H_2) flow rate	2.88 m^3/h
Oxygen (O_2) flow rate	2.24 m^3/h
Carrier gas (N_2) flow rate	$0.96 \text{ m}^3/\text{h}$
Frequency of shots	3 shots/s
Spot size (diameter)	20 mm
Spraying distance from nozzle	160 mm
Powder flow rate	$1-2$ g/shot
Length of the Barrel	1350 mm

are rich in Al along with O and Ni. The presence of oxygen near intersplat region indicates the formation of oxides as a result of oxidation of in-flight particles during D-gun spraying. The EDAX analysis of the as-sprayed coatings shows light and dark gray regions (Fig. 2c). The light gray region is rich in Ni, while the dark gray region is rich in Al (Fig. 2c). Very little oxygen is present on the surface indicating minimum decomposition of feed powder during D-gun-spraying process.

3.2 Porosity, Surface Roughness, Micro Hardness, and Thickness of the Coating

Table 3 presents the porosity, surface roughness, and microhardness values of D-gun-sprayed Ni-5Al coating on In-718 superalloy. The average thickness of the Ni-5Al coating was found to be $175 \mu m$.

3.3 Visual Observations

Photographs of bare and Ni-5Al-coated superalloys subjected to cyclic oxidation in air at 900 °C for 100 cycles are shown in Fig. [3](#page-3-0). The color of the oxide scale formed on the Table 2 Spray parameters used to deposit Ni-5Al coating bare In-718 was dark brown up to 10 cycles, and during

Table 3 Porosity, surface roughness, and microhardness of Ni-5Al coating

Ni-5Al	Porosity, %			
coating			Surface (Avg. values) Sf Kh Ad roughness, Ra, μ m	Micro hardness, Hv
		$1.2 \quad 0.8 \quad 0.9$	4.8	296

Sf: surface porosity; Kh: cohesive porosity; Ad: adhesive porosity

Fig. 2 SEM/EDAX analyses of (a) polished surface (b) cross-sectional, and (c) un-polished as-sprayed Ni-5Al coating

Fig. 3 Photographs of (a) bare and (b) Ni-5Al-coated superalloys subjected to oxidation studies in air at 900 °C for 100 cycles

Fig. 4 (a) Weight gain/area vs. number of cycles plot and (b) (weight gain/area)² vs. number of cycles plot for bare and Ni-5Al-coated superalloys subjected to oxidation studies in air at 900 °C for 100 cycles

subsequent cycles, gray patches started to appear. By the end of 100 cycles, the entire surface turned to gray color (Fig. 3a). The scale formed was consistent and without any cracks. On the other hand, the color of the scale formed on Ni-5Al-coated superalloy was dark gray with light green patches during initial few cycles, and during subsequent cycles, the entire surface turned to light green color, which remained the same until the end of 100 cycles (Fig. 3b). The scale formed on coated superalloy was adherent, uniform, and without any cracks. As seen in Fig. 3, both bare and Ni-5Al-coated

Fig. 5 X-ray diffraction pattern of (a) bare and (b) Ni-5Al-coated superalloys subjected to oxidation studies in air at 900 °C for 100 cycles

superalloys showed negligible spalling of oxide scale during the entire study.

3.4 High-Temperature Cyclic Oxidation Studies in Air

The plots of the weight gain per unit area versus the number of cycles for bare and coated samples are shown in Fig. [4](#page-3-0)(a). The bare superalloy shows higher weight gain when compared with Ni-5Al-coated superalloy. The cumulative weight gain at the end of 100 cycles for bare and Ni-5Al-coated superalloys were found to be 6.5 and 4.8 mg/cm^2 , respectively. The bare and coated superalloys followed nearly parabolic rate law as evident from Fig. $4(a)$ $4(a)$. The (weight gain/area)² versus the number of cycles data are plotted to show conformance with the parabolic rate law (Fig. [4b](#page-3-0)). The parabolic rate constant K_c was calculated by a linear least-square algorithm to a function in the form of $(\Delta W/$ $A^{2} = K_{p}t$, where $\Delta W/A$ is the weight gain per unit surface area $(mg/cm²)$, and t is the oxidation time in seconds. The parabolic rate constants (K_p in 10⁻¹⁰ g²/cm⁴ s) for bare and Ni-5Al-coated superalloys are found to be 1.17 and 0.64, respectively.

3.5 XRD Analysis of Oxide Scales

The x-ray diffraction patterns of surface oxide scale of bare and Ni-5Al-coated superalloys after cyclic oxidation in air at 900 °C for 100 cycles are shown in Fig. 5. The main phases identified in case of bare superalloy are NiO, Cr_2O_3 , Fe₂O₃ and $NiCr₂O₄$. On the other hand, Ni-5Al-coated superalloy showed NiO, α -Al₂O₃, AlNi, Al₃Ni₂, NiAl₂O₄, NiCr₂O₄ and Cr₂O₃ as main phases.

3.6 SEM/EDAX Analysis

3.6.1 Surface-Scale Analysis. The SEM micrographs with EDAX spectrum at selected points of interest on the surfaces of the bare and Ni-5Al-coated superalloys after cyclic oxidation in air at 900 \degree C for 100 cycles are shown in Fig. [6.](#page-5-0) The surface scale developed on bare superalloy is nodular, continuous, and without any cracks. The nodules are rich in Cr_2O_3 along with a considerable amount of Fe_2O_3 (points 1 and 2). The major phases identified by EDAX analysis are $Cr₂O₃$, $Fe₂O₃$ and NiO along with minor quantities of MnO and TiO₂. The scale formed on Ni-5Al-coated superalloy is dense with dark gray and white regions. The dark gray region mainly consists of Al_2O_3 along with NiO (point 3), while the white region predominantly consists of NiO (point 4). The major phases indicated by EDAX analysis are NiO and Al_2O_3 along with small amounts of Cr_2O_3 , Fe_2O_3 , MnO, and TiO₂. The presence of above phases on the surfaces of bare and coated superalloys is also supported by XRD analysis.

3.6.2 Cross-Sectional EDAX Analysis of the Scale. The oxidized samples of bare and Ni-5Al-coated superalloys were cut along the cross sections and mounted in epoxy resin, mirror polished, and gold coated to study the cross-sectional details using FESEM/EDAX analysis.

The cross-sectional EDAX analysis of oxidized bare and Ni-5Al-coated superalloys is shown in Fig. [7](#page-6-0). Analysis of top scale of bare superalloy (Fig. [7a](#page-6-0)) shows mainly the presence of Ni, Cr, Fe, and oxygen along with small amounts of Al, Nb, and Ti (point 1). The percentages of Ni and Cr are less in the top scale compared with the subscale region (point 2). The oxygen has penetrated into the subscale region (points 2 and 3), and its presence is almost negligible further into the substrate material (points 4-6). In case of Ni-5Al coating (Fig. [7](#page-6-0)b), the top scale mainly consists of Ni, Al, and oxygen (point 1). The splats are rich in Ni, while Al is present predominantly near the splat boundaries (points 2-4). Point 5 indicates the presence of high percentages of Cr and oxygen near the coating-substrate interface. Point 6 indicates the composition of the substrate material. The Cr and Fe are present in traces in the oxidized coating and have diffused to the top of the scale. The scale formed on the surface of the coatings was found to be uniform,

Fig. 6 Surface scale SEM/EDAX analysis of (a) bare and (b) Ni-5Al-coated superalloys subjected to oxidation studies in air at 900 °C for 100 cycles

continuous, adherent, and without any cracks. It is believed that some alumina particles might be retained in the asperities during grit blasting of the substrate material prior to the deposition and appeared at the coating-substrate interface as seen in the Fig. [7b](#page-6-0).

4. X-ray Mapping

The x-ray mapping analysis of Ni-5Al coatings subjected to oxidation studies (Fig. [8](#page-7-0)) shows that the splats mainly consist of Ni. The top scale is a very thin oxide scale (few microns) and is a combination of oxides of Ni and Al. The Ni-rich splats in the subscale region are mostly unoxidized. The region surrounding

splat boundaries are rich in Al, Ni, and oxygen thereby indicating the formation of their oxides. The Al is mainly concentrated near the intersplat region. Cr and oxygen coexist as a thin layer near the coating-substrate interface indicating the formation of Cr_2O_3 . Small amounts of Cr, Fe, and Mn have diffused into the coating from the substrate material as seen in Fig. [8](#page-7-0).

5. Discussion

The D-gun-spraying process can be successfully used to deposit Ni-5Al coatings on In-718 superalloy. Figure [2](#page-2-0) shows

Fig. 7 Oxide scale morphology and variation of elemental composition across the cross sections of (a) bare and (b) Ni-5Al-coated superalloys subjected to oxidation studies in air at 900 $^{\circ}$ C for 100 cycles

the surface morphology of the as-sprayed coating, which exhibits characteristic splat like structure with very little surface porosity. The cohesive and adhesive porosity values of D-gun-sprayed coating (Table [3\)](#page-2-0) are almost similar to the finding of Senderowski and Bojar (Ref [16](#page-8-0)). Further, the surface roughness of the coating is less, and, therefore, can be used without the post-coating surface-finish operation in some applications. The microhardness values of D-gun-sprayed coatings are found to be slightly higher than those of nickel aluminide coatings deposited using other thermal-spray processes (Ref [30-32\)](#page-9-0). These findings can be attributed to the significantly higher particle velocities and low thermal energy of the powder particles during D-gun-spraying process (Ref [16](#page-8-0)). Figure [3](#page-3-0) shows that the oxide scale formed on Ni-5Al coating is light green in color, which may be due to the presence of NiO in the oxide scale (Ref [27\)](#page-9-0).

Figure [4](#page-3-0) shows the weight change data after the cyclic oxidation studies at 900 $^{\circ}$ C. It is clear from the plots that the weight gained by bare superalloy is more than that of Ni-5Al coating (Fig. [4a](#page-3-0)). Both bare and Ni-5Al-coated superalloys followed nearly parabolic rate law (Fig. [4b](#page-3-0)). A small deviation from the parabolic rate law may be due to formation of inhomogeneous oxides such as NiO and $Cr₂O₃$ during oxidation process. Mahesh et al. (Ref [25\)](#page-9-0). and Choi et al. (Ref [33\)](#page-9-0) also reported the formation of similar oxides during their study. The

weight gain of the Ni-5Al-coated superalloy during cyclic oxidation studies was high initially, and then it becomes nearly constant (Fig. [4](#page-3-0)a). The higher weight gain may be due to the rapid formation of oxides at the surface and splat boundaries of the coating, and within open pores. These oxides create a protective phase that remains adherent and form thermally grown oxides on the metallic coatings by reacting with the environment (Ref [26\)](#page-9-0). As all the internal surfaces get oxidized, the coating becomes dense, and the diffusion of oxidizing species to internal portions of the coatings slows down, while the growth of the oxides becomes limited mainly at the surface of the specimen. Therefore, the weight gain and the oxidation rate becomes steady with time (Ref [34](#page-9-0)). It was found that the overall weight gain of Ni-5Al coating is 26.2% less in comparison to that of bare superalloy.

The XRD analyses (Fig. [5](#page-4-0)) of bare and Ni-5Al-coated superalloys after cyclic oxidation studies indicate the presence of NiO, Cr_2O_3 , Fe₂O₃ and NiO, Al_2O_3 , Ni Al_2O_4 as major phases, respectively. The coated superalloy also shows the presence of Cr_2O_3 and Ni phases. In the oxidizing environment, nickel aluminides undergoes the following chemical reactions $(Ref 35):$ $(Ref 35):$ $(Ref 35):$

$$
2Al + (3/2)O_{2(g)} = Al_2O_{3(s)}
$$
 (Eq 1)

$$
Ni + (1/2)O_{2(g)} = NiO_{(s)}
$$
 (Eq 2)

Fig. 8 Compositional image and x-ray mapping of the cross sections of Ni-5Al-coated superalloy subjected to oxidation studies in air at 900 °C for 100 cycles

$$
Ni + 2Al + 2O_{2(g)} = NiAl_2O_{4(s)}
$$
 (Eq 3)

These oxides are very protective in nature and acts as a solid diffusion barrier that inhibits interaction of oxygen with underlying coating (Ref [27](#page-9-0)). This may be the reason for the good oxidation resistance of Ni-5Al coating.

The surface EDAX analysis of bare superalloy subjected to cyclic oxidation studies shows mainly the presence of Cr_2O_3 , Fe_2O_3 and NiO. The percentage of Fe_2O_3 in the oxide scale is between 11.5 and 12.5% (points 1-2 Fig. [6](#page-5-0)a). $Fe₂O₃$ is a porous, nonprotective oxide phase and allows oxygen to penetrate through the oxide scale (Ref [3](#page-8-0), [34\)](#page-9-0). This may be the reason for the low oxidation resistance of bare superalloy in the given environment. In case of Ni-5Alcoated superalloy, the main phases present were NiO and $Al₂O₃$. The presence of these phases on the surface scale is also supported by XRD analysis. Also, small quantities of Cr_2O_3 , Fe₂O₃, and MnO present on the surface scale indicate the interdiffusion of these elements from the substrate into the coating. The scale formed on the surface of the coating is adherent and uniform.

The cross-sectional EDAX analysis of bare superalloy indicates that the oxygen has penetrated through the top scale and reached the subscale region (points 1-4 Fig. [7a](#page-6-0)). This may be due to the presence of porous $Fe₂O₃$ phase in the top scale (point 1). In case of Ni-5Al coating, the top scale mainly consists of Ni, Al, and oxygen (point 1 Fig. [7](#page-6-0)b) indicating the formation NiO , Al_2O_3 and $NiAl₂O₄$ phases, as given by the reactions ([1](#page-6-0)-3). The sub-scale region shows that the Ni-rich splats are mostly un-oxidized and therefore reveal the as-sprayed nature of the coating (point 4). The presence of Al along with oxygen in the intersplat region indicates the formation of Al_2O_3 . Al_2O_3 has excellent high-temperature corrosion resistance, as it has a high melting point and stability at high temperature (Ref [36](#page-9-0)). This might have blocked the penetration of oxidizing species along the splat boundaries. Further, it is also evident that oxygen could not penetrate easily beyond the subscale region (points 3 and 4). It can be seen from Fig. [7b](#page-6-0) that the Ni-5Al coatings are dense with very little porosity and hence provided very good oxidation in the given environment.

The x-ray mapping of the Ni-5Al coating subjected to cyclic oxidation studies reveals that the top scale is partially oxidized, where Ni, Al, and oxygen coexist indicating the formation of NiO, NiAl₂O₄, and Al₂O₃ on the top scale (Fig. [8](#page-7-0)). The Ni-rich splats are mostly un-oxidized, and Al is mainly present along the splat boundaries. Further, Cr and oxygen coexist as a thin layer near the coating-substrate interface, indicating the formation of $Cr₂O₃$. The above findings are also confirmed by the crosssectional EDAX analysis (Fig. [7](#page-6-0)b). Due to the formation of protective oxide phases such as NiO, $NiAl₂O₄$ and $Al₂O₃$ in the top and subscale regions and along the splat boundaries, the Ni-5Al coating acted as solid diffusion barrier that decreased the availability of oxygen with underlying substrate material. This in turn favors the formation of thermally grown oxide $Cr₂O₃$ near the coating and substrate interface. The Cr_2O_3 forms a dense, continuous, and adherent layer that grows relatively slowly and acts as a solid diffusion barrier that inhibits interaction of corrosive species with the underlying substrate material (Ref 20, [26](#page-9-0)). Small amounts of Cr, Fe, and Mn have diffused from the substrate material into the coatings. This is also supported by XRD, surface, and cross-sectional EDAX analysis.

Therefore, it may be mentioned that, based on the present investigation, D-gun-sprayed Ni-5Al coating can provide very good oxidation resistance at high temperature when compared with bare superalloy.

6. Conclusion

The high-temperature cyclic oxidation behavior of D-gunsprayed Ni-5Al coating on Ni-based superalloy In-718 has been studied, and the following conclusions are drawn.

- 1. D-gun-spraying process can be used successfully to deposit Ni-5Al coating on In-718 substrate material. The deposited coating is dense and uniform with very little decomposition of feed powder during D-gun-spraying process. The surface, cohesive, and adhesive porosity values of the as-sprayed coatings was found to be 1.2, 0.8, and 0.9%, respectively.
- 2. The Ni-5Al-coated superalloy was able to reduce the overall weight gain by 26.2% in comparison with bare superalloys. The bare as well as coated superalloys nearly followed parabolic oxidation behavior after exposure at 900 $^{\circ}$ C for 100 cycles.
- 3. The formation of NiO, Al_2O_3 and Ni Al_2O_4 phases in the oxide scale of Ni-5Al coating has provided better protection against oxidation to the substrate material. The formations of these oxides are confirmed by XRD, EDAX, and x-ray mapping.
- 4. A thin layer of thermally grown Cr_2O_3 oxide is formed at the coating-substrate interface, which might have acted as diffusion barrier and enhanced the protection to the substrate material. The presence of $Cr₂O₃$ is confirmed by cross-sectional EDAX and x-ray mapping analyses.
- 5. Small amounts of Cr, Fe, Mn, and Ti oxides were present on the top scale of the coated samples, indicating the diffusion of these elements from the substrate to the top scale during the study.

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