



## Diffusion mechanisms and microstructure development in pack aluminizing of Ni-based alloys

Francesco Bozza<sup>a,\*</sup>, Giovanni Bolelli<sup>a</sup>, Carlo Giolli<sup>b</sup>, Andrea Giorgetti<sup>b</sup>, Luca Lusvarghi<sup>a</sup>, Paolo Sassatelli<sup>a</sup>, Andrea Scrivani<sup>c</sup>, Alessia Candeli<sup>a</sup>, Martin Thoma<sup>d</sup>

<sup>a</sup> Department of Engineering “Enzo Ferrari”, Università di Modena e Reggio Emilia, Via Vignolese 905, 41125 Modena (MO), Italy

<sup>b</sup> Formerly in Turbocoating SpA, Via Mistrali 7, Rubbiano di Solignano (PR), Italy

<sup>c</sup> Turbocoating SpA, Via Mistrali 7, Rubbiano di Solignano (PR), Italy

<sup>d</sup> MT Consulting, Munich, Germany

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### ABSTRACT

Despite the large industrial use of pack aluminizing processes for the protection of parts (e.g. cooling channels) of gas turbine blades, systematic studies relating the formation mechanisms and the chemical composition of pack aluminized layers to important process parameters, including the nature of the base alloy and the temperature, are scarce. In this study, 4 different alloys (pure Ni, Ni–20Cr, Inconel 738 and directionally solidified CM247LC) were subjected to a pack aluminizing process at three different temperatures (950 °C, 1000 °C, 1040 °C), using a pack mix containing a fluorine-based activator; the results were compared to those obtained with a chemical vapor deposition (CVD) aluminizing process performed at 1040 °C with the same fluorine-based gaseous precursor.

The microstructural characterization, performed by SEM + quantitative EDX analysis, XRD and nanoindentation testing, shows that, during the heating stage of the pack aluminizing process, Al is transported to the sample surface at temperatures too low to allow significant simultaneous diffusion of Ni; therefore, a  $\delta$ -Ni<sub>2</sub>Al<sub>3</sub> outer layer is formed by inward Al diffusion below the alloy surface, and its growth then continues during the isothermal stage as well. As a result, the chosen isothermal treatment temperature does not affect growth mechanisms, although it modifies the overall thickness of the aluminized layer.  $\delta$ -Ni<sub>2</sub>Al<sub>3</sub> is converted to  $\beta$ -NiAl after a subsequent vacuum heat treatment at 1120 °C. In a CVD process, where gaseous precursor are introduced only after attaining the isothermal treatment stage, Al and Ni diffuse simultaneously from the very beginning of the aluminizing process and  $\beta$ -NiAl is directly developed.

Less mobile species (heavy atoms, such as W) in the alloy composition hinder all diffusion phenomena, both during pack aluminizing and during subsequent vacuum heat treatment: after aluminizing, precipitates are developed within the  $\delta$ -Ni<sub>2</sub>Al<sub>3</sub> outer layer and, after vacuum heat treatment, the resulting  $\beta$ -NiAl layer exhibits a compositional gradient.

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### 1. Introduction

The modern land-based gas turbine plants operate in very demanding conditions, where severe mechanical and thermal stresses (exposure to high temperatures and strong thermal gradients) are made more critical by the presence of a corrosive environment, which may cause oxidation and hot corrosion. The components employed therefore require the use of bulk materials capable to resist mechanical stresses at high temperatures. These materials are typically Ni-based superalloys, whose chemical composition is optimized to reach the best mechanical performance and creep resistance, even if this causes limited resistance to corrosion and oxidation in the previously mentioned environment.

As a result, without the use of protective surface treatments, these degradation mechanisms could seriously damage the components, resulting in complete system shutdown. Therefore the use of protective coatings has a twofold positive effect: preventing damage to expensive superalloy components and avoiding production slowdown due to maintenance needs [1,3].

Depending on the specific environment and process temperature, different coating solutions are available to protect the base alloy from oxidation and/or hot corrosion. In the land-based gas turbine field, the state of the art consists in the protection of the outer surfaces of blades and vanes with a layered thermal barrier coating system based on an external ceramic layer (YPSZ), providing thermal insulation in order to decrease the service temperature of the superalloy, and an inner bond coat (MCrAlY, where M = Ni, Co), which prevents oxidation and corrosion on account of its sufficiently large content of elements able to form a stable oxide scale, such as chromium and aluminum [4–7].

\* Corresponding author. Tel.: +39 0592056281; fax: +39 0592056243.

E-mail address: [francesco.bozza@unimore.it](mailto:francesco.bozza@unimore.it) (F. Bozza).

**Table 1**  
Nominal chemical composition (wt.%) of the four substrate alloys employed in this study.

	Pure nickel	Ni20Cr	Inconel 738	CM274LC
Ni	Bal.	Bal.	Bal.	Bal.
Co	/	/	8.00–9.00	9.00–9.20
Cr	/	20	15.70–16.30	8.00–8.10
W	/	/	2.40–2.80	8.50–9.00
Al	/	/	3.20–3.70	5.00–5.60
Ta	/	/	1.50–2.00	3.00–3.20
Ti	/	/	3.20–3.70	0.07–0.70
Zr	/	/	0.05–0.15	0.015
Mo	/	/	1.50–2.00	0.05–0.50
B	/	/	0.005–0.015	0.015–0.15
C	/	/	0.15–0.20	0.05–0.07
Others	/	Si: 1.5	Nb: 0.60–1.10	Hf: 1.00–1.50

**Table 2**  
Pack-mix composition.

Role	Al source	Activator	Inert filler
Chemical formula	CoAl	AlF <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>
Amount (wt.%)	10	0.3	Bal.

The continuous rise in turbine inlet temperatures, necessary to improve the production efficiency, also caused the need for internal cooling systems. Small amounts of air, diverted from the compressor, are forced to flow through thin channels inside the turbine blades and vanes. Due to the high service temperature of the blade ( $\approx 800$ – $900$  °C) the air that flows through the channels can easily oxidize the internal surface of the component, also leading to the consequences described previously.

In order to protect these critical internal surfaces from oxidation and hot corrosion, the state of the art involves the use of diffusion coatings, formed by chemical vapor deposition (CVD), by pack-cementation, by above-the-pack diffusion processes and/or by slurry cementation [8–10]. The protection is achieved by enriching the surface layer of the component in oxide scale formers, such as aluminum, chromium and/or silicon, through diffusion mechanisms. These elements react with the substrate material to form secondary phases and intermetallics that act as a reservoir of scale formers during the working life. In particular, aluminum enhances the oxidation resistance of components exposed at high temperature ( $>900$  °C) because of the formation of a stable Al<sub>2</sub>O<sub>3</sub> oxide film, whereas layers based on Al + Cr or Al + Si improve the resistance against hot corrosion phenomena occurring at temperatures between 700 °C and 900 °C [2,10–12].

In chemical vapor deposition processes, the component is exposed to volatile precursors at high temperature, and the coating is produced by chemical interactions taking place in the vapor phase, leading to the deposition of reduced metallic species onto the substrate surface and to subsequent interdiffusion between the substrate and the deposited layer [8]. The vapor phase enabling the formation of diffusion coatings generally consists of gaseous halides of the desired elements. During the manufacturing of CVD diffusion coatings, the

vapor halides can reach the surface of the components only when the process temperature is reached, avoiding undesired transient phenomena that may occur during the heating and the cooling phases. The main disadvantage of this technique is the high cost of the CVD apparatus.

Pack-cementation processes are comparatively cheaper, cost-effective techniques in which the component is embedded in a mixture of powders (pack-mix) based on a metal source (which acts as a reservoir of scale former), an activator (which develops a vapor phase by reacting with the metal source and transports the vapor containing the metal phase onto the component surface) and an inert filler, which prevents sintering and agglomeration phenomena of the metal source powders [1,2,9,13].

The present investigation studies the mechanisms of formation of aluminum diffusion coatings by pack cementation. During this process, the aluminum deposited onto the component surface from the vapor phase reacts with nickel (the main constituent of many superalloys employed in gas turbine components) to form intermetallics [13,14]. The main phase of the coating depends chiefly on the aluminum activity, because this parameter influences the location of the reaction front [8,15–17].

In “high activity” processes, the formation takes place due to the inward diffusion of the aluminum deposited by the vapor phase onto the component surface. The coating contains Ni<sub>2</sub>Al<sub>3</sub> ( $\delta$ -phase) as main constituent; moreover, its overall composition strongly depends on the chemical composition of the substrate, because alloying elements typically precipitate out of the Ni matrix as it reacts with inward-diffusing Al, developing secondary phases into the  $\delta$  phase. In this case, the coated component requires a further heat treatment (in a vacuum atmosphere) to allow further transformation of the metastable  $\delta$ -phase into the stable  $\beta$ -phase (NiAl): this phase is indeed less hard and more ductile than Ni<sub>2</sub>Al<sub>3</sub>, therefore it is more protective because it is less susceptible to cracking during service; moreover, it is thermodynamically stable and its thickness does not change upon further exposure to high temperatures [18]. A disadvantage of coatings obtained from the “high activity” process is the fact that the  $\delta$ - $\beta$  transformation is accompanied by an increase in the overall thickness of the surface modified layer which is difficult to control.

In “low activity” processes, the coating grows by the outward diffusion of nickel, which reacts with the aluminum halides to form  $\beta$ -NiAl. The nickel depletion caused by the transport of this element to the external surface leads to the precipitation of alloy elements at the interface between the  $\beta$ -NiAl layer and the substrate, with the formation of an interdiffusion layer (IDL) containing secondary phases (e.g. carbides). In this case, the thickness of the surface modified layer remains almost constant upon further exposure to high temperatures; therefore, these coatings are more stable.

In addition to these two cases (pure high and low activity) there are also many intermediate situations in which the coating formation is caused by hybrid growth mechanisms that involve the diffusion of both Al and Ni [19,20].

Although pack cementation processes have already been in industrial use for a long time, systematic investigations dealing with the

**Table 3**  
Process conditions and corresponding specimen labels.

Substrate	Thermal treatment in vacuum atmosphere at 1120 °C	Pack-aluminizing temperature			CVD (1040 °C)
		950 °C	1000 °C	1040 °C	
Inconel 738	No	950-INC	1000-INC	1040-INC	CVD-INC
	Yes	950-INC-HT	1000-INC-HT	1040-INC-HT	
DSCM247LC	No	950-DSC	1000-DSC	1040-DSC	CVD-DSC
	Yes	950-DSC-HT	1000-DSC-HT	1040-DSC-HT	
Ni20Cr	No	950-NiCr	1000-NiCr	1040-NiCr	CVD-NiCr
	Yes	950-NiCr-HT	1000-NiCr-HT	1040-NiCr-HT	
Ni	No	950-Ni	1000-Ni	1040-Ni	CVD-Ni
	Yes	950-Ni-HT	1000-Ni-HT	1040-Ni-HT	

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