Development and Evaluation of Chromium-Modified Aluminide Diffusion Coating

Ahmed Ali Moosa

University of Technology

Jaleel Kareem Ahmed

Ali Hubi Haleem.

University of Babylon, College of Engineering

Abstract

In this work nickel superalloy Inconel 600 was coated with two different types of coatings Cr modified aluminide coating this is called(Aluminizing- Chromizing) and Y-doped Cr modified aluminide coating this is called (Aluminizing- Chromizing- Yttriumizing) using pack cementation method. Diffusion coating was carried out in one step process by simultaneous aluminizing – chromizing process and by simultaneous aluminizing- chromizing- yttriumizing process using the conversion reaction of 10 wt.% Cr_2O_3 , 10 wt.% Al, 2 wt.% NH_4Cl , 4 wt% NaCl and 72 wt.% Al_2O_3 and 2% Y_2O_3 when required. The diffusion coating processes were carried out at 1050 °C for 8hrs. After diffusion coating the coating layer was found to be consisted of: β -NiAl.

Cyclic oxidation test in air was conducted on the Inconel 600 coated with Cr-modified aluminide coating and on the Inconel 600 alloy coated with Y-doped Cr modified aluminide coating in the temperature range 700- 1100 °C. The oxidation cycles include 10 hrs. at furnace temperature. The results indicate that Y-doped Cr modified aluminide coating is more effective in increasing the oxidation resistance of Inconel 600 alloy. The role of yttrium can be attributed to an improvement of the adherence of the oxide scales.

Keywords: Cyclic oxidation, diffusion coating, Inconel 600 alloy, nickel aluminide.

Introduction

Coatings have over the last 50 years been developed to meet the increasing demands in power generation applications. The request for higher performance and more efficient power units have been met with more advanced materials. For components operating at high temperatures in such units, coating play an important role in this development. High temperature coatings are used to restrict surface degradation or to thermally insulate the material against the hot environment. As the lifetime of structural components is frequently controlled by surface degradation, significant cost and performance improvements are obtained by application of coating. Surface degradation takes place through corrosion, oxidation and solid particle erosion [Eskner,2004].

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Among the various coating methods, pack cementation has been applied for many years to enrich chromium or aluminum at the alloy surfaces. However, in aluminizing an Inconel 600 alloy substrate, the resulting depletion of chromium from the surface of Inconel 600 alloy is problematic, especially for such materials used in hot – corrosion environments. In addition, chromized alloys have poor oxidation resistance at high temperature [Sidhu et al., 2006]. Therefore, to alleviate this difficulty, simultaneous deposition of two elements by pack cementations is very effective in protecting substrates from hot corrosion and oxidizing environments at high temperature.

Various codeposition processes of aluminum and chromium have been suggested: a two-step pack- cementation process in which a substrate is first pack-chromized and then pack- aluminized [Berthod *et al.*, 2006]; a pack process using a Cr-Al masteralloy powder as a pack component [Bianco and Rapp, 1993]; and a pack cementation process using a mixture of pure elemental powder [Zheng and Rapp, 2000].

In this study, aluminum, chromium, and yttrium were simultaneously deposited by diffusion into austenitic Inconel 600 substrates using a single- step pack process with a conversion reaction of Cr_2O_3 to chloride.

Experimental procedures

The substrate alloy used in this study was Inconel alloy 600, which is Nibased superalloy. The nominal composition of this alloy as chemical limits is shown in Table 1[Spetail Metals, 1996] and (ARL Spectrometer 3460) carried out the spectrochemical analysis at Nasir industry as shown in Table 2.

The Inconel 600 alloy samples were, cut into squares with dimensions (20 mm×20mm ×4mm) and a total surface area of 1120 mm². These samples were cut from a sheet perpendicular to the rolling direction. Small hole 2 mm diameter was drilled in each sample for holding. All the surfaces, including the edges were wet ground using 120, 220,320,600,800, and 1200 grit silicon carbide papers. These samples were then cleaned with water, degreased with acetone and then ultrasonically cleaned for 30 minutes using aceton as a medium. After drying, the samples were stored in polyethylene zip-lock bags. As samples were taken for use, their dimensions were measured.

The samples dimensions were measured in three places along the length to a precision of \pm 0.01 mm with a calibrated micrometer. The measured dimensions were averaged to provide the sample length, width, height to be used in surface area calculation. The weight of each sample was measured using a Mettler 200 microbalance with an accuracy of \pm 0.1 mg. The balance was calibrated frequently using both its internal 100 g standard weight and an external 50 g standard weight. Prior to weighing, all samples were held overnight in glass desiccators to eliminate any effect of humidity on the sample weight determination.

The pack mixture used for chromium-modified aluminide diffusion coating consist of 10 wt.% Al powder ($50\text{-}150~\mu m$ in particle size) as an aluminum source (Riedel-de Haen AG-Germany), 10 wt.% Cr_2O_3 powder ($38\text{-}170~\mu m$ in particle size)as chromium source (BDH Chemicals LTD Poole England), 2wt.% NH₄Cl and 4 wt.% NaCl as activators (Chem.-Supply, LTD, Australia) and the balance was α -alumina powder($70\text{-}210~\mu m$ in particle size)(BDH Chemicals LTD Poole England) as the inert filler. The NH₄Cl was dried in an oven at 90° C for 24 hours before mixing with other powders. The pack powder was mixed in a ball mill for 180~m in. with the addition of n-hexane ($n\text{-}C_6H_{14}$) in

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order to prevent powders oxidation due to frictional heat. The pack was then dried at 75 $^{\circ}$ C for 10 hours [Zubedy,2006].While yttrium doped chromium-modified aluminide diffusion coating pack mixture used is the same as in chromium modified aluminide diffusion coating pack except 2 wt.% of the pack alumina filler was replaced with yttria (Y_2O_3) as reactive element oxide. Assay 99.9% (Fluka, Switzerland).

Inconel 600 alloy sample was placed in a sealed stainless steel cylindrical retort of 50 mm in a diameter and of 80 mm in a height in contact with the pack mixture. The retort was then put in another stainless steel cylindrical retort of 80 mm in a diameter and 140 mm in a height. The outer retort has a side tube through which argon gas pass and second hole in the top cover for argon gas outlet. Type – K thermocouple was inserted through the cover of the outer retort for recording real temperature near inner retort. Figure 1 shows the apparatus used for pack cementation. This combined system was then put in an electrical holding furnace under an argon atmosphere with a flow rate of 1.5 L/min. Once the inert atmosphere had been established, heating cycle started for diffusion coating as shown in Figure 2.

After coating, the samples were cooled to room temperature under argon atmosphere. The samples were then removed from the pack and ultrasonically cleaned for 30 minutes to remove any loosely entrapped pack material on the surface, and weighed in order to determine the Al, Cr pickup. No further heat treatment was given to the specimens after coating.

Each sample was cut using a low-speed diamond saw, and the cross section was mounted in a cold-setting epoxy. Grinding was conducted with successively finer silicon carbide papers from 220 to 1200 grit. The samples were then polished using 5.0 µm and 0.3 µm alumina suspension sequentially. These samples were then cleaned and the microstructures of the coated layers were observed after being etched for approximately 10 seconds in an etchant containing 3 parts hydrochloric acid, 2 parts nitric acid, 2 parts acetic acid, and 1 part glycerol by volume [Park et al., 1998]. Cross sections and coating morphology were examined using light optical microscope (L.O.M) type (CARLZEISS JENA, DDR) operated at 60-500X magnification. For this purpose optical microscopy fitted by digital camera type (Smartic with 5 Mega pixels resolution).

Several experiments were conducted to obtain the best coating thickness. It was found that diffusion coating time of eight hours at 1050 $^{\rm o}C$ produce coating thickness of 65-80 μm .

Cyclic oxidation at extended range of temperature 700-1100 °C in air were conducted to study the thermal shock and oxidation resistance of Inconel 600 alloy with and without chromium-modified aluminide diffusion coating, and with yttrium doped chromium-modified aluminide diffusion coating.

During cyclic oxidation the furnace temperature was controlled within ± 3 °C by using Ni-chrome thermocouple type K. The thermocouple was inserted into the furnace chamber through an access hole on the top of the furnace and positioned in the proximity of the samples. The thermocouple was calibrated at three standard temperatures boiling water, tin melting point, and aluminum melting point.

The evaluation of the oxidation resistance of the coatings has been carried out by heating the samples in a furnace at test temperature and weighing them every 10h. The samples were extracted from the furnace, allowed to cool,

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ultrasonically cleaned to detach the spalled oxide and the weight change per unit surface area was determined [Paul et al., 2007].

Inconel 600 alloy with and without chromium-modified aluminide diffusion coating, and with yttrium doped chromium-modified aluminide diffusion coating samples were accurately weighed and then placed into ceramic crucibles. Cyclic oxidation tests were carried out in a Carbolite programmable furnace (manufactured by Sheffield, England) in the temperature range 700-1100 °C in air at one atmospheric pressure. Each heating cycle includes heating in the furnace for 10 hours and cooling in still air.

Samples weight changes before and after each oxidation cycle were measured. Normally; at least 3 weight measurements were taken.

To aid in the identification of coating and oxide phases, X-ray diffraction was performed. A Riga Ku X-ray generator with Cu K α radiation at 40 kV and 20 mA was used. The X-ray generated by general electric diffractometer, type Philips (pw 1840), operating at scanning speed of 6° (20) per minute. The detector was moved through angle of $2\theta = 10$ to 90 degrees. In addition, X-ray Fluorescence (XRF) type (MiniPal 2) as an analytical method was used to determine the concentrations of Cr, Al, and Y, of Inconel 600 alloy with and without coated system.

Result and discussion

Xiang, and Datta [Xiang, and Datta, 2003] reported an outer layer composed of brittle and continuous β -NiAl intermetallic phase, while the interior of the polycrystalline interdiffusion zone was BCC ferrite. Both layers had better corrosion resistance than the underlying austenitic substrate material.

As mentioned previously, simultaneous chromizing and aluminizing of Inconel 600 alloy was carried out using a single – step pack process with a conversion reaction of Cr_2O_3 to chloride. The pack consisted of 10: 10: 2: 4: 74 of (Al: Cr_2O_3 : NH₄Cl: NaCl: Al₂O₃) respectively.

During pack processing at 1050 °C for 8 hr, aluminum and chromium diffuses into the substrate and results in a phase transformation, while nickel at the surface is pushed inward the substrate. It was expected that coating layer is composed of three zones: an outer layer of β -NiAl, a nickel-rich nickel aluminide band, and an alpha ferrite containing Ni₃Al precipitates.

It has been reported [Nychk and Clarke, 2005] that a displacement reaction involving a reactive element (RE) oxide source as Y_2O_3 in the high chlorine activity pack may also generate additional metal chloride species as follows:

$$MCl_x(v) + REO_y(s) \longrightarrow RECl_x(v) + MO_y(s)$$
 (1)

Where M represents Cr and/or Al and RE is Y. In our present study, M corresponds to Al and RE to Cr or Y. The reduction reaction of Cr₂O₃ or Y₂O₃ by Al itself can, of course, be included in Eq. (1), which is followed by a halide reaction of Cr.

Figure 3a illustrates the homogenous topography of coated surface while Figure 3b indicated that coated layer was 63 μ m thick. The point to be noted from Figure 4 is that, peaks of XRD analysis were present due to the range of 20 is closed to 10-90 degree. These peaks are related mainly to NiAl phase.

On other hand depending upon the results of XRF tests as in Figure 5 which determined the aluminum concentration in the outer coating layer as 20.15 wt.%, 17.91 wt.%, and 12.54 wt.% for a number of Inconel 600 substrates with chromium-modified aluminide diffusion coating. Whereas, XRF results referred to

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chromium concentration in the coated layer which increased from about 15.5 wt.% for bare Inconel 600 alloy substrate (Figure 6) to 24.08 wt.% for chromium-modified aluminide diffusion coated Inconel 600 substrate as in Figure 7. From these comparisons among the concentration profiles shown in the previous Figure 5, Figure 6 and Figure 7, the aluminum and chromium concentration at the outer coating layer is higher after codeposition process than in the substrate.

Yttrium – doping of a Cr – Al coating was achieved for the Inconel 600 alloy substrate. Yttrium was introduced by replaced a small amount (2 wt. %) of the previous pack alumina filler with yttria. The concentration of Y introduced into the Inconel 600 alloy substrate would be limited by its low solubility and low activity in the pack. The XRF of the coated layer indicated that only 0.1% was introduced. This trace amount is expected to be segregated at the metal – scale interface and can affect significant benefits.

Figure 8a shows the coating microstructure of Y- doped chromium modified aluminide diffusion coating while Figure 8b illustrated that coated layer was 60 μ m thick. On other hand Figure 9, determine the phases present at the coated layer and it was consist mainly of NiAl phase, while yttrium or yttrium compounds phases that probably exist in the outer layer in insufficient amount is difficult to identify or is undetectable by X – ray diffraction analysis because of the lower Y content in the coated layer.

Studies of oxidation kinetics provide valuable information about the oxidation mechanism and the rate – limiting step of the total reaction.

Cyclic oxidation test in air was conducted on the Inconel 600 alloy in the temperature range 700-1100 °C. The oxidation cycles include 10 hrs at furnace temperature. The obvious fluctuated specific weight observed in the bare Inconel 600 alloy from the beginning of exposure especially at 1000 °C and 1100 °C (Figure 10) indicates that the alloy possesses insufficient resistance to cyclic oxidation at these higher temperatures. The high rate of weight change at these temperatures also confirms the insufficient resistance of this alloy at these temperatures. Obvious spalling of oxides is observed during the oxidation of Inconel 600 alloy at these temperatures, and this is reflected in the weight change plots, although it appears to be reasonably resistant to oxidation at 900 °C.

As observed in the cyclic oxidized Inconel 600 alloy sample shown in Figure 11, large numbers of voids exist at the scale / alloy interface and in the alloy. It is believed that these voids reduce the scale / alloy contact and hence facilitate spalling. These voids may act as concentration sites of thermal stresses induced during cooling, leading to crack formation in and spalling of the surface oxide scale.

The surface of oxide scales formed on Inconel 600 alloy after 100 hours exposure time of oxidation in air at temperatures range 700–1100°C; was examined by X – ray diffraction analysis (XRD). At 700 °C, only minor quantities of nickel and iron oxides are observed on the surface and a significant amount of unoxidized chromium metal are detected within the oxidized area, even close to the outer surface. While the major phases occurred on Inconel 600 alloy surfaces at oxidized temperatures 800-1100°C in air up to 100 hr are NiO, (Cr, Fe)₂O₃, and spinal phases of Ni(Cr₂O₄, Fe₂O₄).

Oxidation of Coated System

Both chromium-modified aluminide and yttrium-doped chromium modified aluminide coated Inconel 600 substrates were subjected to cyclic oxidation test in air in the temperature range 700-1100 °C. The oxidation cycles

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include 10 hrs at furnace temperature. The specific weight change of the samples with both types of coated system indicated above during oxidation is plotted as a function of time as shown in Figure 12 and Figure 13 respectively.

Based on the positive weight – change data the coated system of Inconel 600 alloy appears to be fairly resistance to oxide spallation even at 1100°C despite the fact that spinel forms on the surface with alumina throughout the exposure. Both types of coated system appear quite adequate for this purpose for two reasons. First, the most desirable alumina protective oxide layer forms on the sample surface during oxidation, and this oxide layer has sufficient adherent with the coated substrate to withstand the imposed cyclic heating and cooling environment. This is clearly evident from the continuous weight gain noted during oxidation. Second, the NiAl phase of the coating remaining untransformed over the entire exposure period indicates that the aluminum loss from the coating during oxidation is very slow because of the formation of a spall – resistant alumina layer especially at higher temperatures. However, the yttrium doped-chromium modified aluminide coating showed less weight gain especially at 900 - 1100°C in comparison with chromium-modified aluminide coating due to the effect of yttrium. By this time, as the severity of the cyclic oxidation conditions increases with increasing oxidation temperature, the superiority of the both types of coated system in providing protection becomes more prominent.

Inconel 600 alloy underwent obvious spalling when subjected to thermal cycling. The surface layer of Inconel 600 alloy spalled over a significant portion of the sample surface after only two—three of heating and cooling cycles. Whereas the coated system; showed spalling of the surface oxides that occurs in the advanced stages of oxidation which takes place uniformly as a fine powder over the entire surface of the sample.

The phase constitution of the coatings was determined using XRD analysis. The major phase in the as–formed coated system (yttrium doped and un doped–chromium modified aluminide) was found to be NiAl phase. Cyclic oxidation at oxidized test temperature (700 -1100°C) resulted in the formation of Al₂O₃ on the sample surface and, as anticipated, its amount increased with exposure duration.

Cyclic oxidation performed at 900-1100 °C, however, resulted not only in the transformation of the bulk coating phases, but also in the formation of spinel as the additional oxide phase in coated system. For example, when the chromium—modified aluminide coating is thermally cycled at 1100 °C, the bulk coating phase transforms partially to alumina, with the accompanying formation of spinel like NiCrO₄ in addition to alumina.

Rapp et al. [Rapp *et al*, 1991] performed cyclic oxidation tests at 1100°C in air of coated Rene 80 alloy substrate prepared by the above – pack arrangements. Two of the Cr / RE–modified coatings produced acceptable protective scales of α – Al₂O₃ and NiAl₂O₄ spinel for 500 one–hour cycles.

Figure 14 revealed cross section images of LOM of chromium – modified aluminide diffusion coated Inconel 600 substrates after cyclic oxidation in air at 800 and 1000 °C for up to 100 hr.

Further, Figure 15 showed cross section images of LOM of yttrium – doped chromium modified aluminide diffusion coated Inconel 600 substrates after cyclic oxidation in air at 800 °C and 1000 °C respectively for up to 100 hr, under same oxidation conditions indicated previously

Conclusions

In this study, two different types of coatings were performed, the first was aluminum and chromium were simultaneously deposited and, the second was aluminum, chromium, and yttrium were simultaneously deposited by diffusion in to Inconel 600 substrates through pack cementation using the conversion reaction of Cr_2O_3 and Y_2O_3 to chloride, one- step coating process and their results were compared. Increasing concentration of Cr and Al were obtained from the coating layer. A codeposited coating layer, which shows high oxidation resistance, is mainly characterized by three zones: an outer layer of β -NiAl, a nickel- rich nickel aluminide, and an interdiffusion zone consisting of alpha ferrite and nickel aluminide precipitates.

In summary, the results observed in this study have confirmed that:

- 1. The Y- doped chromium modified aluminide diffusion coating displayed better cyclic oxidation resistance than the Cr-modified aluminide diffusion coating without Y. This means that Y-doped chromium modified aluminide diffusion coating had service lives longer than Cr- modified aluminide diffusion coating without yttrium.
- 2. During oxidation tests, both of coatings kept their adhesion to the substrate.
- 3. The addition of Y as a reactive element resulted in a reduction of void formation at the oxide / substrate interface and improve oxide adhesion, attributed this to Y consisted particles acting as vacancy sinks.
- 4. The phases formed during the oxidation of Inconel 600 alloy (uncoated) are: NiO, (Cr, Fe)₂O₃, and spinel phases of Ni (Cr₂O₄, Fe₂O₄).
- 5. Cyclic oxidation at oxidized test temperature 700-1100 $^{\circ}$ C resulted in the formation of Al_2O_3 on the both types of coated system surface and, its amount increased with exposure duration.

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Table 1: Limiting chemical composition of Inconel 600 alloy in wt. %, according to [Spetail Metals, 1996].

Ni	Cr	Fe	Mn	Cu	Si	S	P	C
72.0	14.0-	6.0-	1.0	0.5	0.5	0.015	0.015	0.15
Min.	17.0	10.0	Max.	Max.	Max.	Max.	Max.	Max.

Table 2: Spectrochemical analysis of Inconel 600 alloy in wt. %

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Ni	Cr	Fe	Mn	Cu	Si	S	P	C
Rem.	15.902	8.453	0.102	0.099	0.223	0.013	0.012	0.153
		Zn	Mo	Ti	Mg	Al		
		0.009	0.507	0.003	0.143	0.399		

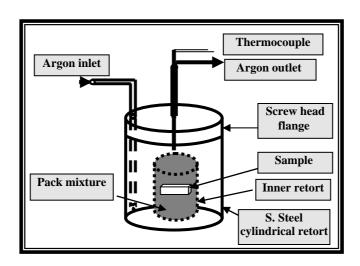


Figure 1: Schematic diagram illustrates apparatus used for pack cementation process.

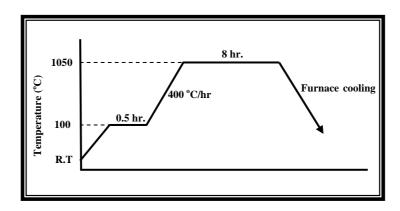
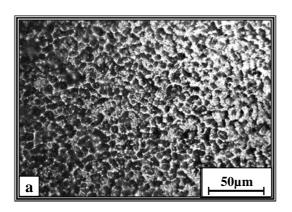


Figure 2: Heating cycle for one-step pack cementation process.



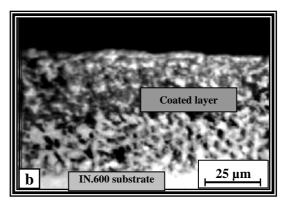


Figure 3: LOM images of Inconel 600 alloy with chromium-modified aluminide diffusion coating
(a) Top view, and (b) Cross section view

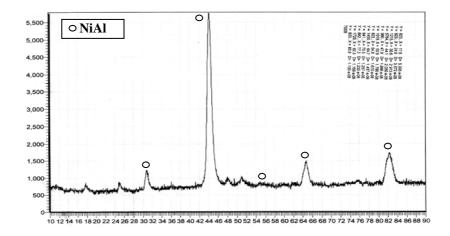


Figure 4: Diffractograms from the surface of Inconel 600 with chromium-modified aluminide diffusion coated sample.

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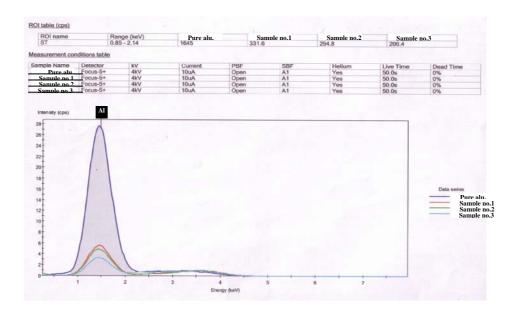


Figure 5: X-ray fluorescence pattern illustrated aluminum percentage for pure aluminum sample (99.8%) in comparison with Inconel 600 alloy substrate no. (1, 2 and 3) with chromium- modified aluminide diffusion coating.

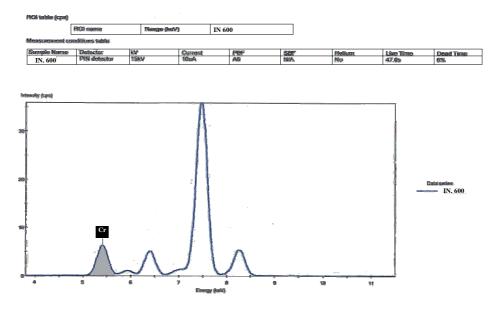


Figure 6: X-ray fluorescence pattern illustrated chromium percentage for Inconel 600 alloy.

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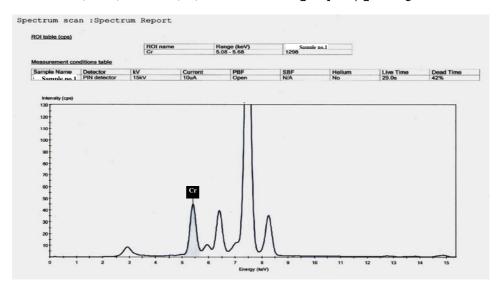


Figure 7: X-ray fluorescence pattern illustrated chromium percentage for Inconel 600 alloy with chromium-modified aluminide diffusion coating.

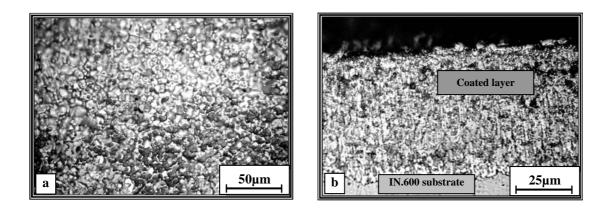


Figure 8: LOM images of yttrium-doped chromium modified aluminide diffusion coating (a) Top view, and (b) Cross section view

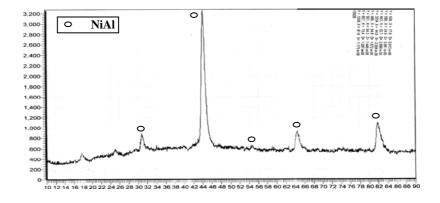


Figure 9: Diffractograms from the surface of Inconel 600 with yttrium-doped chromium modified aluminide diffusion coated sample.

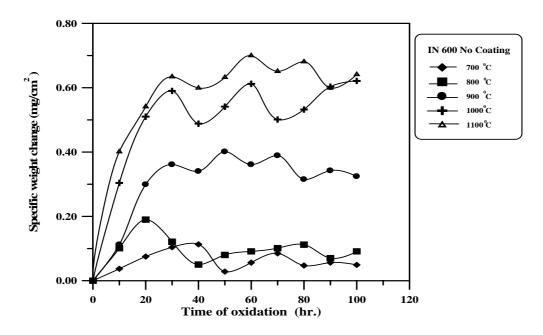


Figure 10: Specific weight change vs. time plot for Inconel 600 alloy samples cyclic oxidized in air at temperatures between 700 and 1100 $^{\rm O}{\rm C}$ for up to 100 hr, using 10 hr for each cycle.

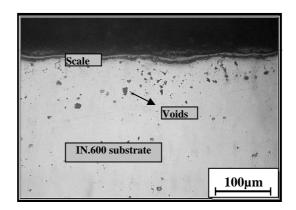


Figure 11:Cross section image of LOM of Inconel 600 alloy after 100 hr of cyclic oxidation in air at 1000 °C, using 10 hr for each cycle.

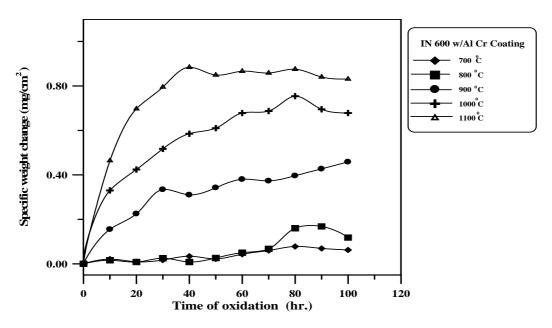


Figure 12: Specific weight change vs. time plot for chromium-modified aluminide diffusion coated Inconel 600 substrates cyclic oxidized in air at temperatures between 700 $^{\circ}$ C and 1100 $^{\circ}$ C for up to 100 hr, using 10 hr for each cycle.

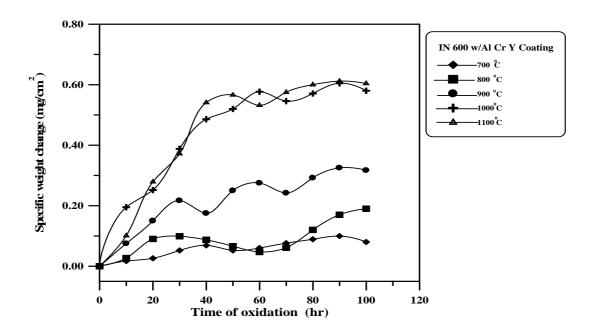
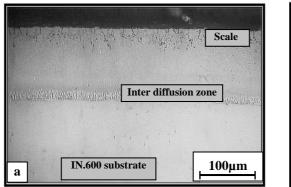


Figure 13: Specific weight change vs. time plot for yttrium-doped chromium modified aluminide diffusion coated Inconel 600 substrates cyclic oxidized in air at temperatures between 700°C and 1100 °C for up to 100 hr, using 10 hr for each cycle.

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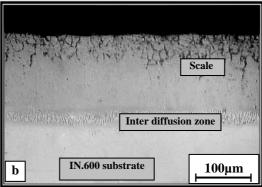
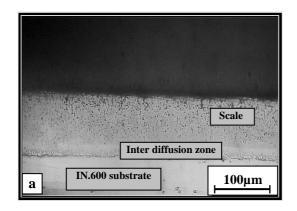


Figure 14: Cross section images of LOM of chromium-modified aluminide diffusion coated Inconel 600 substrates after cyclic oxidation in air at (a) 800 $^{\circ}$ C, and (b) 1000 $^{\circ}$ C using 10 hr for each cycle up to 100 hr.



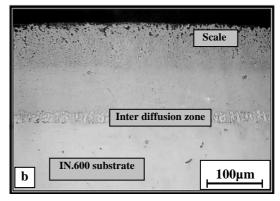


Figure 15: Cross section images of LOM of yttrium-doped chromium modified aluminide diffusion coated Inconel 600 substrates after cyclic oxidation in air at (a) 800 $^{\circ}$ C, and (b)1000 $^{\circ}$ C, using 10 hr for each cycle up to 100 hr.