

Oxidation of Ni – Al Electrodeposited Composite Coating of Inconel 600 Alloys

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Abstract :

The oxidation behavior of nickel matrix/ aluminum particle composite coatings in air at (600-800°C) for 25 hrs. was studied. The composite coatings were applied to Inconel 600 alloy by the electrodeposition technique and heat treated under argon atmosphere for three hours at 800 °C prior to oxidation testing. During heat treatment, coating morphological development was found to occur by the exothermic reaction $Ni + Al = NiAl$. For all samples, weight gain kinetics was obtained from furnace exposure and the composition and morphology of the oxidation products were examined using XRD analysis and light optical microscopy. An outer Al_2O_3 layer was formed on the composite coating surface. At test temperatures such as 800 °C, the coatings benefits from a small grain size that enhances Al diffusion to the surface to form the protective alumina layer.

Keywords: Composite Coating, Ni-Al Phase, EMMC, Inconel 600 Alloy, Nickel Aluminide Electroplating.

الخلاصة :

تم دراسة آلية التأكسد لسبيكة الانكونل 600 المكتسبة بالطلاء المركب النيكل-ألومنيوم عند درجات حرارة (600-800 °C) ولفترة (25 hrs.). تم إجراء عملية الطلاء بتقنية الترسيب الكهربائي والمتبوعة بمعالجة حرارية في جو من الأركون لفترة (3 hrs.) عند (800 °C). حصل أثناء المعاملة الحرارية تفاعل باعث للحرارة ($Ni + Al = NiAl$). جرت اختبارات التأكسد لجميع النماذج وكذلك تم تحديد صفات ومكونات طبقة الأوكسيد باستخدام المجهر الضوئي وحيود الأشعة السينية، لوحظ عند درجة حرارة الاختبار (800 °C) إن الحجم الحبيبي الناعم للطلاء يعزز من انتشار الألومنيوم إلى السطح مكونا طبقة الألومينا الواقية.

الكلمات الرئيسية : الطلاء المركب ، أطوار Ni-Al ، EMMC ، سبيكة الانكونل 600 ، الطلاء الكهربائي بالنيكل - ألومنيوم .

1. Introduction

Conventional electrolytic plating, in which the component is used as the cathode in an electrolytic cell, has the advantage that coating of any thickness can be applied to selected areas of the component by using masking techniques. The coatings have excellent adhesion, forming a strong bond at the interface between the deposited and base metals. The process has the added benefit that it is carried out at low temperatures (below 90 °C), so that there is no risk of distortion of the component. The drawbacks of electrolytic plating are that some base materials require low temperature de-embitterment (at 150 to 400 °C) after plating, and that deep holes, recesses and similar shapes are more difficult to plate and may require specially shaped anodes [1]. Electrodeposited composite coatings (ECCs) maintain all the features described above, although a minimum coating thickness is required to trap the particles of the codeposited material. Successful ECCs formed by electrolytic methods require clean particles, typically 2 to 8 μm in size, to be dispersed uniformly throughout the electrolyte during the plating cycle. The particles must be processed so that the wet out and remain suspended in the solution, and it's important that the purity of the electrolyte is maintained [2]. Particles that have been successfully used in commercial ECCs to date include hard ceramic particles, such as Al_2O_3 or TiO_2 , in an electrodeposited matrix such as nickel [2]. Another type of composite coating can be produced by codeposition of aluminum particles into an electrodeposited nickel matrix to produce electrodeposited metal matrix / metal particle composite coatings (EMMC). Subsequent diffusion heat treatment produces a Ni-Al alloy coating. Electrodeposited composite coatings rely on small particles as the source of protective

scale- forming elements, i.e., Al, in the present case. In this sense, composite coatings can be considered an extension of the "dispersed reservoir effect", first studied by Nagarajan and Wright [3] and Wright et al. [4]. In electrodeposited-composite coatings, metallic particles (Al) are used, which diffuse into the matrix (Ni) to form a uniform alloy coating. After heat treatment, the initially distinct particles are no longer present, but Al is now distributed throughout the microstructure. The result is a uniform, oxidation resistant alloy coating. Although porosity is found within Ni-Al composite coatings, few other coating defects are observed. Nickel- aluminum intermetallic alloys based on NiAl or Ni₃Al have shown good resistance to high temperature oxidation [5]. Application of nickel aluminides as coatings provides the benefit of corrosion protection while maintaining the mechanical properties of the substrate. Conventional techniques for applying nickel aluminide coatings include thermal spray, weld overlay, and pack cementation (aluminizing) among others. The EMMC technique offers an alternative method for applying nickel aluminide coatings with few defects, good coating / substrate bonding, and low deposition and heat treatment temperatures [1]. The morphological development of EMMC coatings during heat treatment is similar to that found in synthesis of bulk nickel aluminides by powder metallurgy [6]. However, in EMMC coatings, only one component is in particulate form (Al) while the other component is present as a surrounding matrix (Ni). In EMMC coatings, the particles are effectively isolated from each other by the surrounding electrodeposited matrix. At elevated temperature, phase formation takes place locally around the individual particles. While the as- deposited coatings are essentially fully dense, depending on the particle/ matrix system, porosity can develop during inter diffusion and intermetallic formation between the particles and matrix [7]. This work describes coating processing and examines intermetallic phase development during heat treatment of Ni-Al coatings. Further this work aims to gain an understanding of the oxidation behavior of Ni-Al electrodeposited composite coatings as a function of exposure temperature and time.

2. Experimental Procedure:

2.1. Materials

The substrate alloy used in this study was Inconel alloy 600, which is Ni- based super alloy. The nominal composition of this alloy as chemical limits is shown in Table 1 [8] 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 shape with dimensions (20 x 20 x 4 mm) and a total surface 1120 mm². These samples were cut from a sheet perpendicular to the rolling direction. Small whole of 2 mm diameter was drilled in each sample for holding. All 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 distilled water, degreased with acetone and then ultrasonically cleaned for 30 minutes using ethanol as a medium. After drying, the samples were stored in polyethylene zip-lock bags.

2.2 Coating Deposition:

Before Ni-Al electrodeposited composite coating begins, thin layer of Ni deposits as a preparation step were done according to the following steps [9]:

- 1.Cold rinse.
- 2.Dip in 20% hydrochloric acid (1.098 sp. gr.) at 21-27 °C for 1 min.
- 3.Cold rinse.
- 4.Anodic etch in wood's nickel- strike bath which consisted of: 240 g/ liter NiCl₂ and 86 ml / liter HCl (concentration 37%) at 43 °C as solution temperature using deposition current density of 0.0538 amp / cm² for 20 sec.
- 5.Plate in wood's bath at 43 °C, using deposition current density of 0.0538 amps / cm² for 2 min.
- 6.Cold rinse.

The aim of this step is to get a bonding layer (Ni) between Inconel 600 alloy surface and the electrodeposited composite coating (Ni-Al).

Electrodeposition of Ni-Al composites was performed in a typical Watts bath on the Inconel 600 alloy. This work was performed in the University of Technology- Department of Production and Metallurgy-Corrosion Lab. A nickel sulfamate electrolyte was used with the following composition: 400 g/l nickel sulfamate $\text{Ni}(\text{NH}_2\text{SO}_3)_2$, 30 g/l boric acid, 5 g/l nickel chloride (NiCl_2), 0.2 g/l sodium lauryl sulfate (wetting agent), and 0.1 g/l coumarin (leveling agent). All of the bath compositions were mixed in 1 liter of deionized water. The bath composition is similar to that of Guglielmi [10] and other commercial plating baths [11]. It was found that excessive amounts of the surfactant (coumarin) can cause "foaming" of the plating bath when Al particles are added and, therefore, minimum levels of this additive were used. After the plating bath was prepared, the Al powder was added. The powders are codeposited within the nickel matrix to provide for nickel aluminide formation. The aluminum powder obtained from BDH Chemicals Ltd., Poole England, high purity (99% Al), 8 μm max. diameter. Powder additions of 75, 150, 225, 300, 400 and 500 g/l were used in experiments. During deposition, the Inconel 600 substrate was held vertically in the stirred stream of the electrodeposition bath. The bath was maintained at 50 °C and $\text{pH} \cong 4.0$. It was found that deposition current density of about 4.5 $\text{amps} / \text{dm}^2$ produced the best results, and this current density was fixed for all coatings in the present research. Plating time of three hours was found to produce coatings of 125-140 μm thickness. To summarize Table 3 shows the major processing parameters for typical coating deposition. After deposition the samples were mounted for metallographic and / or used in heat treatment process

2.3 Heat Treatment and X-ray Diffraction Analysis

To investigate nickel aluminide coating formation, samples were heat treated in tube furnace type Carbolite programmable furnace, manufactured by Sheffield, England that was kept under argon atmosphere with a flow rate of 1.5 L /min. to avoid the oxidation of the underlying materials during the process (Fig. 1 shows the apparatus used for heat treatment). Once the inert atmosphere had been established, heating cycle is started. Typical annealing conditions were three hours at 550 °C followed by three hours at 800 °C. The argon atmosphere was maintained during all the annealing process as well as during cooling. Light optical microscopy observations showed that all the electrodeposits contained Al particles became included in the surface involves the formation of weak chemical bonds between the surface and Al particles suspended in the electrolyte. The particles are thus at first chemically bonded to the surface, becoming built into the coating as the plating process proceeds. This random process results in a uniform dispersion of Al particles throughout Ni matrix (Fig. 2). It can be shown that the black regions observed in the nickel coating are Al particles. Further, Fig. 3 shows light optical microscopy image of the cross section of Ni- Al composite layer which has thickness of about 140 μm , with no cracks or other defects, and good bonding with the substrate. In general, the Al particles are approximately spherical in shape and distributed uniformly throughout the coating. The coatings are not etched to show the Al particles distribution but identified using X- ray with general electric diffractometer, operating at scanning speed of 2° (2 θ) per minute with $\text{CuK}\alpha$ ($\lambda = 1.54 \text{ \AA}$). However, the major phase on the surface is NiAl, as in Fig. 4. On other hand the results of atomic absorption test indicated that the average aluminum concentration is $\cong 32 \%$. This test was performed in Bin Sina Company.

2.4 Isothermal Oxidation in Dry Air

Sample mass determinations were made using a (Mettler 200) microbalance capable of weighing to a precision of $\pm 0.1 \text{ mg}$. The balance calibration was frequently checked using 10 and 5 g standard mass. The dimensional and mass specifications for each sample were recorded on the run sheet for the sample throughout its testing. The prepared samples were suspended with platinum wire and inserted into a tube furnace held at temperature (600, 700, and $800 \pm 3 \text{ }^\circ\text{C}$) where they were oxidized in dry air at 1 atmospheric pressure for up to 25 hrs. duration (Fig. 5). The Ni-Al composite coated samples were oxidized isothermally and the weight gain was monitored and recorded at suitable intervals. Test temperature was controlled within $\pm 3 \text{ }^\circ\text{C}$. Temperature

measurements of the furnace chamber were made with Nichrome thermocouple mounted in a high – purity alumina protection tube. The Nichrome thermocouple was inserted in to the furnace chamber through an access hole on the top of the furnace. The surfaces of oxidized samples in air were examined using (L.O.M), whereas the phases present on the surfaces were identified with XRD analysis.

3. Results and Discussion

3.1 Coating Deposition

To determine the effect of Al particles bath loading on the volume fraction of particles in the coatings, deposition experiments were performed with 75, 150, 225, 300, 400 and 500 g/l of powder in the bath. The relative volume of particles in the as- plated coatings determines the resultant coating composition and dictates, through the phase diagram, whether more stable nickel aluminide formation is possible (Fig.6) [12]. In fact, the amount of particles in the coating increases as the bath loading increases. Therefore, the coatings deposited with 500 g/l particles bath loading were used in the heat treatment and XRD experiments presented during this research. For a given particle content in the bath, a higher resultant volume present is found in the coatings. When particles are dispersed into bath, they develop a positive charge due to a layer of Ni²⁺ ions on their surface. Bath stirring supplies particles at a constant rate to the substrate surface where they adhere to the relatively charged substrate (cathode) [2]. During deposition, a steady state is thereby established in which the coating grows around the particles as new particles and fresh electrolyte are continuously supplied at the cathode surface. However, because of the electrostatic attraction, the steady state particles loading local to the surface is higher than the bulk bath loading. In summary, the results show that the composite electrodeposition techniques can be used to successfully produce Ni-Al composite coatings. The deposition follows the general trend of increasing coating volume percent with increased bath loading, up to approximately 500 g/l.

3.2 Isothermal Oxidation in Dry Air

Studies of oxidation kinetics provide valuable information about the oxidation mechanism and the rate – limiting step of the total reaction. Oxidation rate measurements also commonly serve as a basis for a quantitative numerical description of the oxidation behavior.

Weights changing were recorded for kinetics identification in dry air in the temperatures range 600 – 800 °C for up to 25 hrs. The specific weight change data of the Ni-Al electrodeposited coated Inconel 600 alloy for each temperature test is plotted in Fig. 7 as a function of time. The initial kinetic is rapid, but the rate of specific weight change gradually decreases at longer times. The kinetics can be described by examining the growth rate time constant or n value, which is found as the exponent in the following rate equation [13]:

$$\Delta W/A = k t^n \quad \dots\dots\dots (1)$$

Where ΔW is the weight change, A is the sample surface area, k is the rate constant, n is the growth- rate time constant, and t is the time of oxidation.

At 600°C, the n-value is about 0.53, i.e., parabolic behavior is found (A computer program is used to calculate n values according to the best fit to Equation 1). Another n values are shown in Table 4. It is found that the relationship is parabolic where n = 0.5. The exponential constant n characterizes the oxidation rate as follows: if n = 1, the oxidation rate is linear, n = 0.5 the oxidation rate is theoretically parabolic, if n = 0.33, then the oxidation rate is cubic [14]. When the value of n is greater or lower than 0.5 then oxidation kinetic does not fall in the simple parabolic behavior and this implies a faster or slower oxidation rate. For example, for n > 0.5 it is an over- parabolic, while for n < 0.5 the rate is under parabolic (sub- parabolic). The results show that sub- parabolic may be found because of grain boundary (short circuits) mechanisms. Deviation from theoretical value of n = 0.5 can be explained by an oxide layer cracking, leading to a sudden increase of the surface area in contact with oxygen and then accelerating the oxidation kinetics. The weight gain during the

transient stage corresponds to above interpretation ,experimental conditions and the many variables like area and weight measurement, and others may play an important role in this case [15].

These results show that the parabolic kinetics at this temperature range can be quantified on the modified parabolic rate law with the assumption that oxidation is controlled by diffusion mechanism and the grain boundaries are the only effective short circuit diffusion paths. The grain boundary diffusion mechanism provides an initially high oxidation rate. As time passes, oxide grain growth occurs, which decreases the number of easy diffusion paths and slows the oxidation rate. Therefore, the easy pathways cut off, and the oxidation rate is decreased beyond that for parabolic kinetics [16].

For the parabolic kinetics, the rate equation takes the form:

$$\Delta W/A = k t^{0.5} \dots\dots\dots (2)$$

Where k now refers to the parabolic rate constant. A plot of specific weight change vs. square root of time gives a line as in Fig. 8; the slop is the parabolic rate constant in units of (mg/cm²)/hrs.^{1/2}. The k_p value is then squared to give k_p in units of (mg²/cm⁴)/hrs. as in the following expression:

$$(\Delta W/A)^2 = k_p t \dots\dots\dots (3)$$

The parabolic oxidation rate constants for three series of experiments are calculated and the linear lines represent the least squares curve fits to the data in Fig. 8, the parabolic oxidation rate constants (k_p) for the set of experiments are listed in Table 4.

Over the temperatures range from 600 to 800 °C, the parabolic oxidation rate coefficients and thus the oxidation rates of nickel aluminide coated Inconel 600 alloy in air, vary in magnitude from a low value of 2.14x10⁻⁷ (mg²/cm⁴)/s. at 600 °C to a high value of 3.06 x10⁻⁶ (mg²/cm⁴)/s. at 800 °C. The point to be noted is that the weight gain calculated by application of the parabolic oxidation rate coefficient in Table 4 with Equation 3 results in the weight of oxygen gained by the sample under isothermal oxidation.

From the experimental work, data revealed that a parabolic oxidation rate (k_p) obeys an Arrhenius- type equation of the form:

$$k_p = k_o \exp (-Q/RT) \dots\dots\dots (4)$$

Where k_p is the parabolic oxidation rate, k_o is the pre-exponential factor, Q is the activation energy, T is the temperature, and R is the universal gas constant (8.33 J/K) [17]. Plot of log (k_p) vs. (1/T), the effective energy is calculated from the least square fitting (R² = 0.94) of the observed data in the temperature range from 600 to 800 °C is to be ~ 103 KJ/mol. as shown in Fig. 9. The obtained value is in a good agreement with data from Salmon et al. [18] where the value of activation energy for the parabolic growth of Cr₂O₃ is (100-170) KJ/mol., on Ni- 20Cr.

Nickel-aluminum electrodeposited composite coating appears quite adequate for high temperature oxidation for two reasons. First, the most desirable alumina protective scale layer forms on the sample surface during oxidation, and this scale layer has sufficient adherent with the coated substrate to withstand the imposed isothermal oxidation. This is clearly evident from the continuous weight gain noted during oxidation. Second, the NiAl phase of the coating remaining untransformed totally 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. When composite coating containing Al is exposed to a high- temperature oxidation environment, Al₂O₃ scale is formed on the surface of the alloy. This scale prevents the rapid oxidation of nickel and iron in the alloy, thereby eliminating the dissolution and inward diffusion of oxygen. In general, a slow rate of scale growth is not the only objective for better resistance to high temperature degradation. Once scales formed on the alloy surface are damaged, a new scale has to grow during continuous application at high temperature. Maintaining scale integrity and adherence to the alloy substrate during service is another important requirement for high temperature applications. As long as the scale maintains integrity and adherence during isothermal oxidation, it can accommodate large stresses generated and resist scale spalling for longer periods of time [19]. The phase constitution of the coatings was determined using XRD

analysis. The major phase in the as – called nickel aluminide composite coating was found to be NiAl phase. Isothermal oxidation at oxidized test temperature (600 - 800°C) resulted in the formation of Al₂O₃ on the sample surface and, as anticipated, its amount increased with exposure duration (Fig. 10), which shows the X – ray diffractograms of the composite coating corresponding to isothermal oxidation test temperatures. Also, no excessive scale spalling is noticed during the entire oxidation period. It is, however, interesting to note that the bulk coating phase NiAl does not transform to lower aluminum – containing phases, such as γ' - Ni₃Al and γ -Ni phases, even after 25 hrs. of exposure. This is a clear indication of the lower rate of aluminum loss from the coating due to the lack of spalling of the protective alumina layer. Thus, the composite coating in this study appears to be fairly adequate in providing isothermal oxidation protections to the Inconel 600 alloy up to 800°C. Furthermore, Fig. 11 show top view image of LOM of the nickel aluminide composite coating after isothermal oxidation in air at 800 °C for 25 hrs. From the surface appearance of the sample, it does not show severe scale spalling and appeared uniform and smooth after isothermal oxidation.

4. Conclusions:

1. The amount of Al particles codeposited into a nickel matrix increase as the amount of particles in the bath is increased. A maximum amount of codeposited particles is obtained at bath loading of 500 g/l and deposition current density of 4.5 A/ dm². Codeposition of Al particles into a nickel matrix produces coatings with a uniform distribution of particles.
2. Heat treatment at 800 °C for 3 hrs. under argon atmosphere results in diffusion reaction between the Al particles and nickel matrix to produce a range of Ni-Al Intermetallic compounds like NiAl and Ni₃Al phases.
3. Electrodeposition of Al followed by diffusion annealing could produce NiAl phase.
4. Over the temperatures rang between 600-800 °C, the parabolic rate constant (k_p) varies from a ($k_p = 2.14 \times 10^{-7}$ (mg²/cm⁴)/s.) at 600 °C to a value of ($k_p = 3.06 \times 10^{-6}$ (mg²/cm⁴)/s.) at 800 °C.
5. Nickel aluminide composite coating exhibited parabolic oxidation rate dependence in air over the temperatures between 600- 800 °C, and the activation energy is ~ 103 KJ/mol.
6. Alumina is the major phase that exists in the outer layer under oxidation conditions.

4. References

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Table 1: Limiting chemical composition of Inconel 600 alloy in wt. % [8].

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

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

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			

Table 2: Major Processing Parameters for a Typical Ni-Al EMMC Coating.

Plating Bath	Nickel Sulfamate
Particle loading (Al)	500 g/l
Temp.	50 °C
pH	4.0 – 4.1
Current Density	4.5 A/dm ²
Stirring	400 RPM
Plating Time	3 hrs.

Table 4: n values and parabolic oxidation rate constants k_P for isothermal oxidation for 25 hrs. of Inconel 600 alloy with Ni-Al electrodeposited composite coating

Temperature ($^{\circ}\text{C}$)	n values	k_P (mg^2/cm^4)/s.
600	0.53	2.14×10^{-7}
700	0.57	1.25×10^{-6}
800	0.62	3.06×10^{-6}

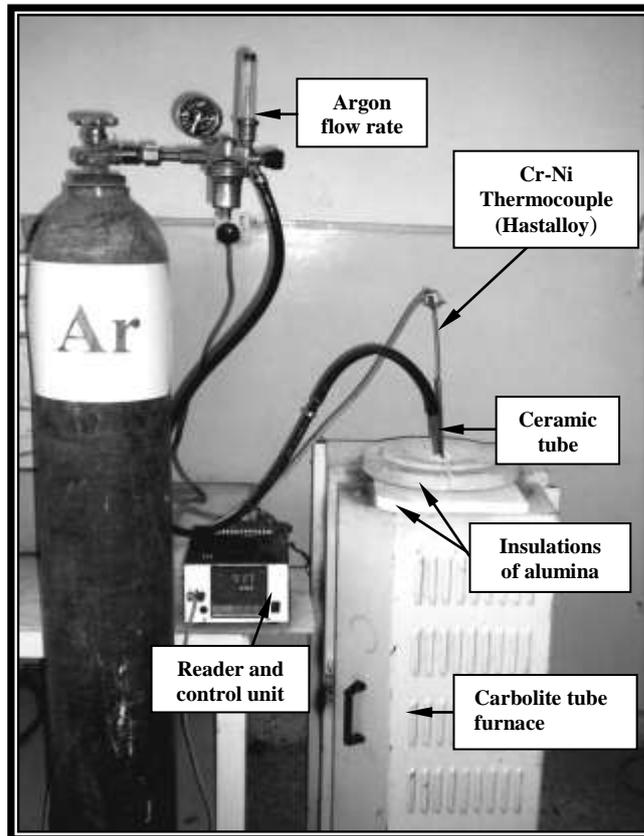


Fig. 1: Experimental set up used for heat treatment.

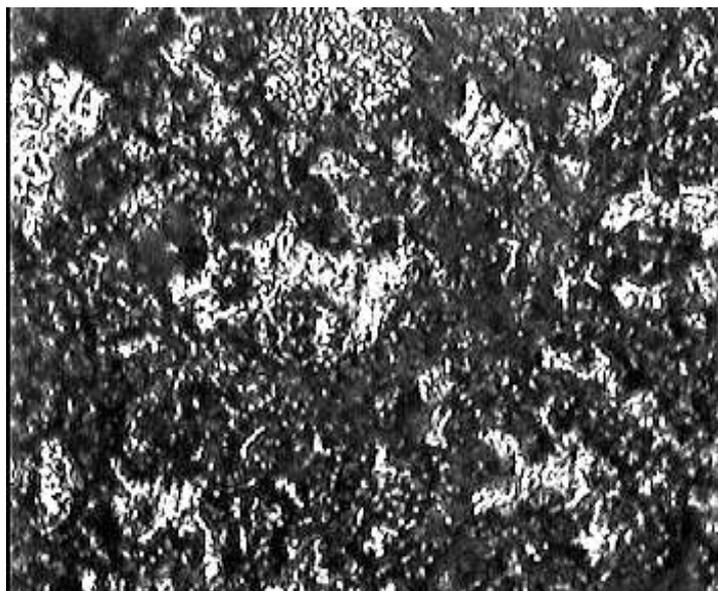


Fig. 2: LOM image of the surface of Ni-Al composite layer (300X).

Fig. 3: Cross section image of Inconel 600alloy with Ni-Al electrodeposited composite coating layer (300X).

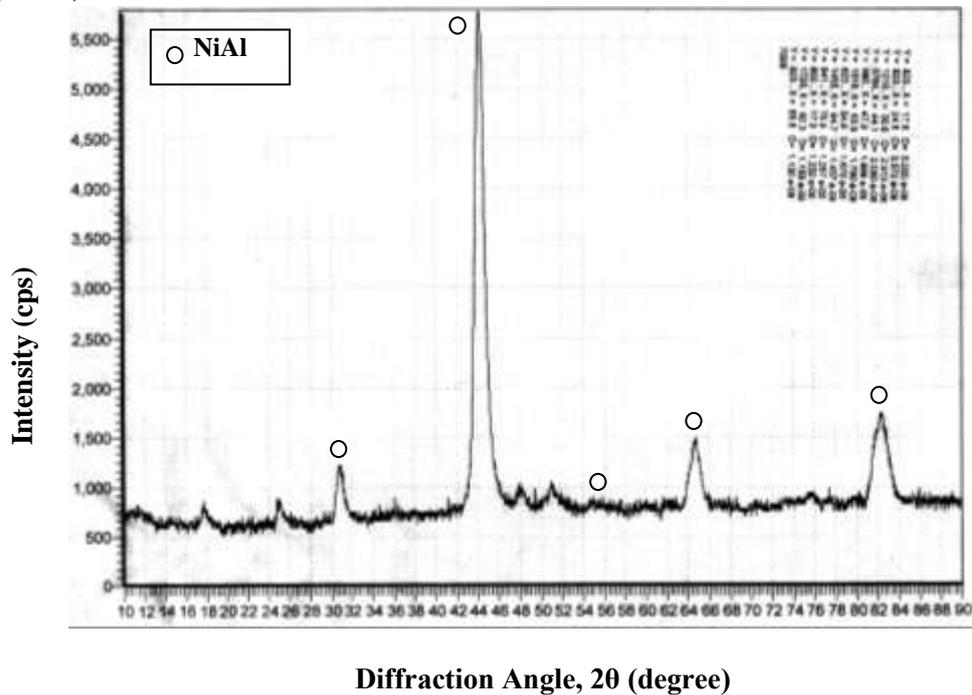


Fig. 4: Diffractograms from the surface of Ni-Al electrodeposited composite coating of Inconel 600alloy.

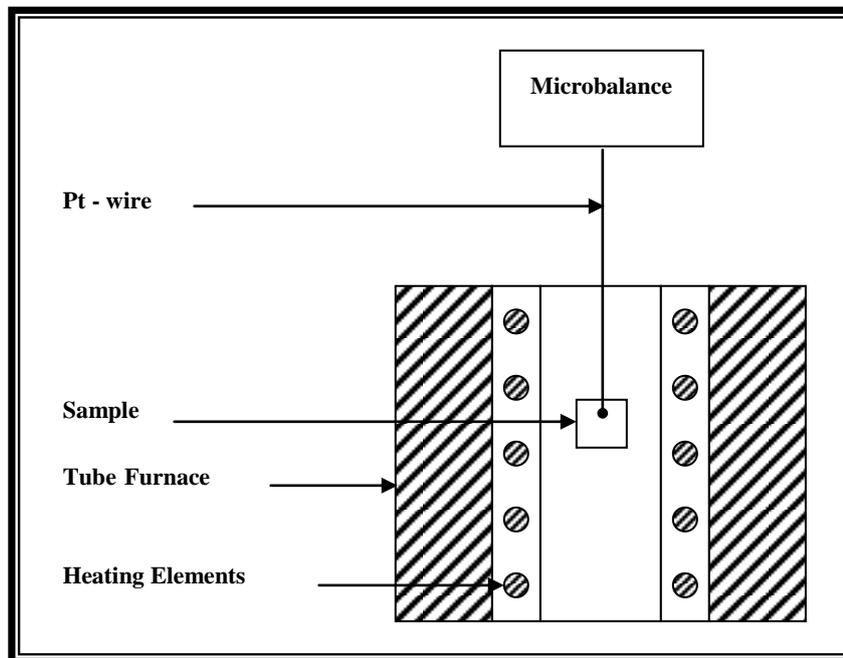


Fig. 5: Apparatus used for isothermal oxidation tests in dry air.

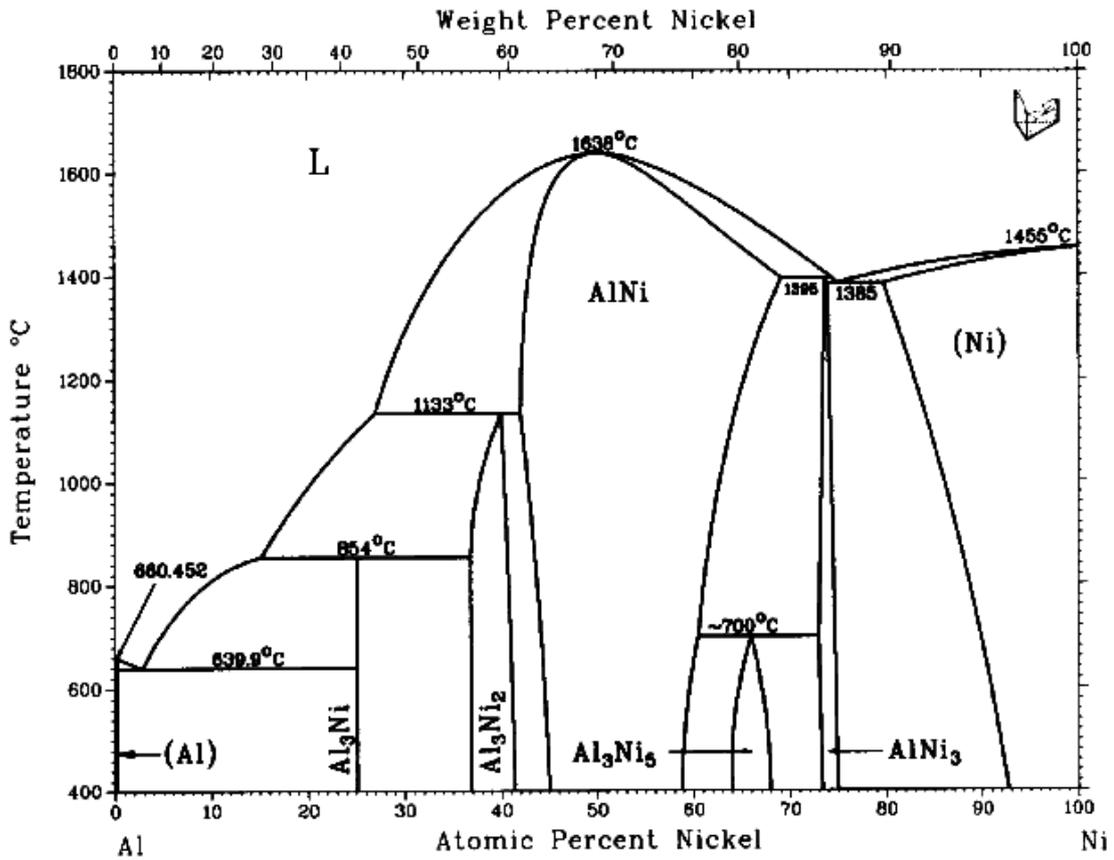


Fig. 6: Equilibrium phase diagram for Ni-Al [13].

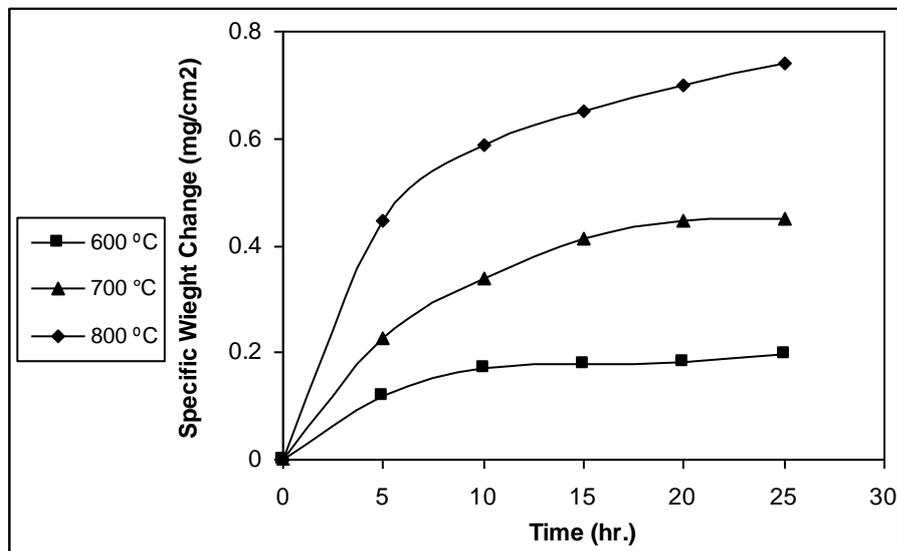


Fig. 7: Specific weight change vs. time plot for Ni-Al electrodeposited composite coating of Inconel 600 alloy oxidized in air at temperatures between 600 and 800 °C for 25 hrs.

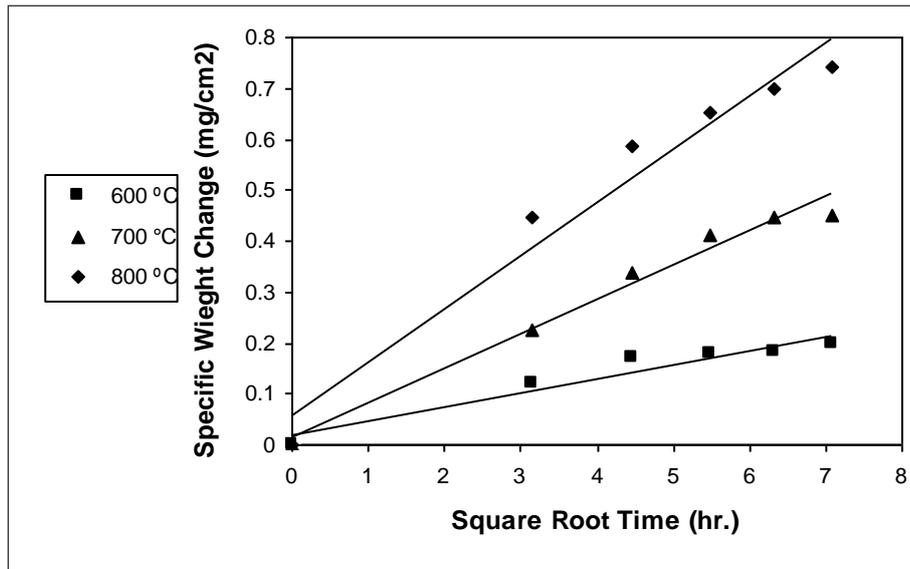


Fig. 8: Linear fitted results of specific weight change vs. $t^{0.5}$ plot for Ni-Al electrodeposited composite coating of Inconel 600 alloy oxidized in air at temperatures between 600 and 800 °C for 25 hrs.

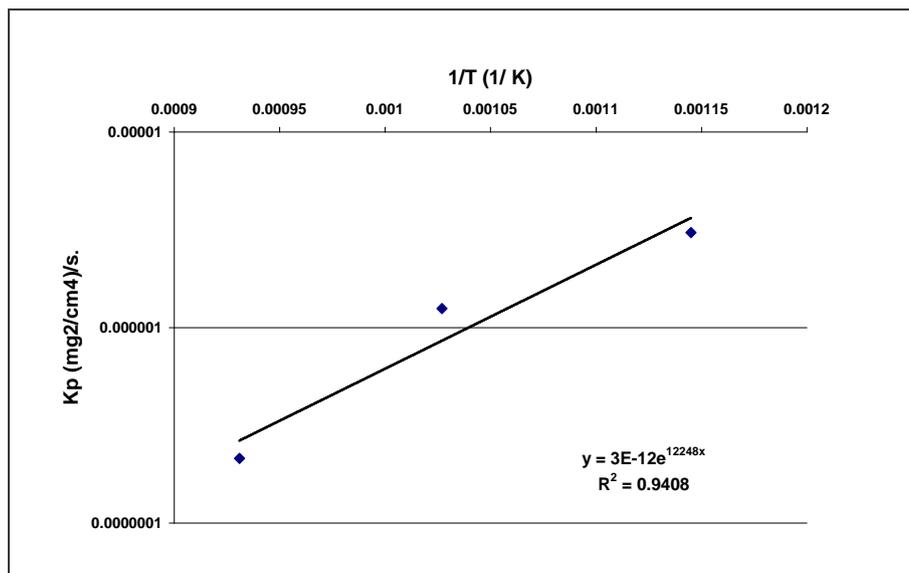


Fig. 9: Plot of k_p vs. $1/T$ for Ni-Al electrodeposited composite coating of Inconel 600 alloy oxidized in air at temperatures between 600 and 800 °C for 25 hrs.

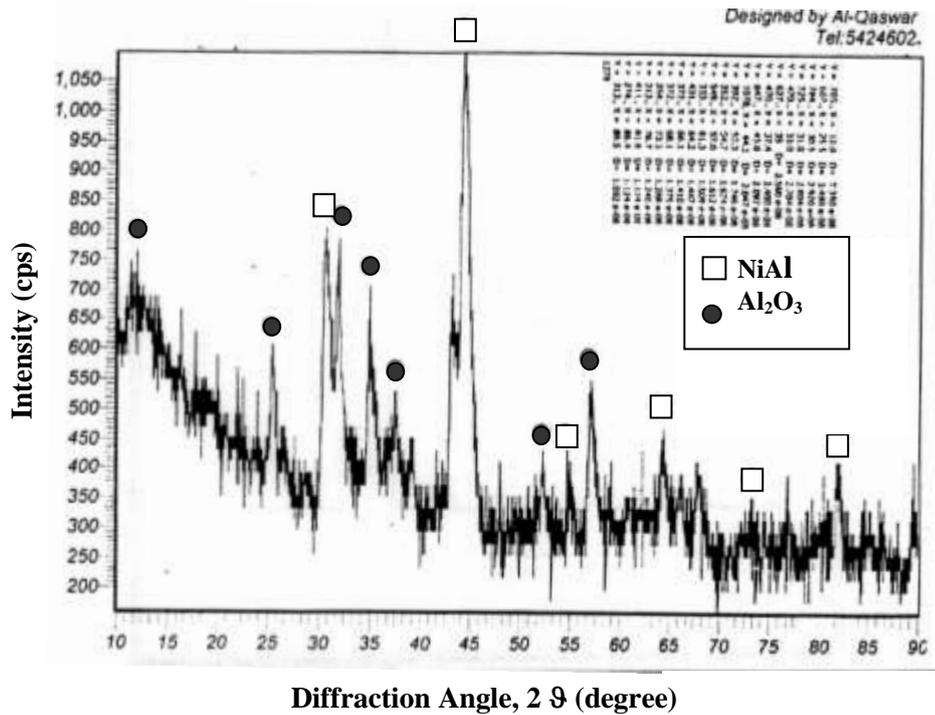


Fig. 10: Diffractograms from the surface of Inconel 600 alloy with Ni-Al electrodeposited composite coating after isothermal oxidation in dry air at 800 °C for 25 hrs.

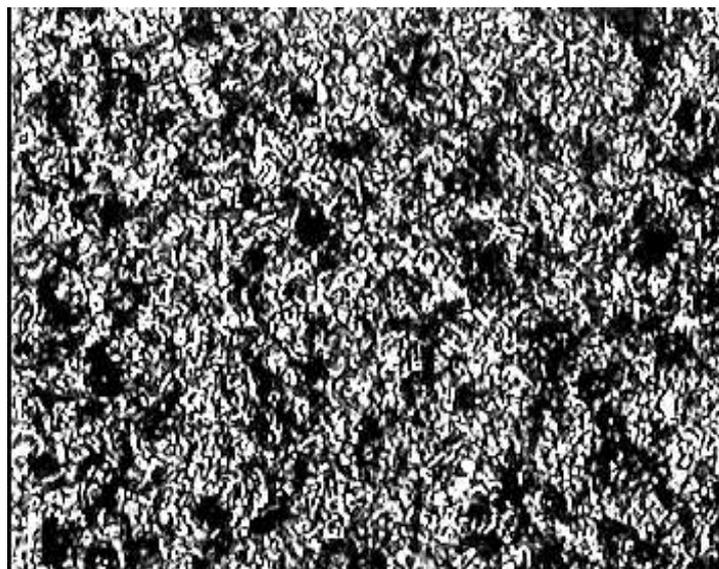


Fig.11: Surface morphology of Ni-Al composite layer after 25 hrs. isothermal oxidation at 800 °C for 25 hrs.