

INVESTIGATION OF OXIDATION RESISTANCE OF NI- Ti ELECTROCHEMICAL CODEPOSITION COATING OF AUSTENITIC STAINLESS STEEL (316 L)

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ABSTRACT

The effects of Ti particle loading in a plating bath on the morphology and hardness of Ni-Ti composite coatings via an electrochemical codeposition coating were investigated. The Ti-reinforced Ni-matrix composite coatings were codeposited on Austenitic Stainless Steel (316 L) substrates using a Ni-ion electrolytic solution stably suspended with -20 μm max. diameter Ti particles and heat treated under vacuum atmosphere for six hours at 950 °C prior to oxidation testing. During heat treatment, coating morphological development was found to occur by the exothermic reaction $\text{Ni} + \text{Ti} = \text{NiTi}$.

Further, the oxidation behavior of nickel matrix/ titanium particle composite coatings in air at (700-900°C) for 50 hrs. was studied. The prepared coatings were characterized by X-ray diffraction (XRD), optical microscope, scanning electron microscopy (SEM), Vickers hardness, and atomic absorption. 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 optical microscopy.

An outer TiO_2 layer was formed on the composite coating surface. At test temperatures 700, 800, and 900 °C, the coatings benefits from a small grain size that enhances Ti diffusion to the surface to form the protective TiO_2 layer.

Key words: Composite Coating, Ni-Ti Phase, Austenitic Stainless Steel (316 L) Alloy, Nickel Titanium Electroplating.

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1. INTRODUCTION

Electrochemical codeposition is a cost effective method of producing metal matrix composite coatings. Numerous particle-dispersed metallic-matrix coatings have been successfully produced through electrochemical codeposition by electroplating the coatings from a conventional plating bath suspended with particles. By combining the electrodeposited metallic matrix such as Ni with a range of dispersed particles such as SiC, WC, ZrO₂, or Al₂O₃, composite coatings of improved mechanical properties have been produced [1].

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 [2].

Electrochemical codeposition maintain all the features described above, although a minimum coating thickness is required to trap the particles of the codeposited material. Successful Electrochemical codeposition 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 [3].

Furthermore, the technique can also be employed to fabricate an intermetallic-contained layer which generally requires two processing steps including (i) codeposition of metals and active particles and (ii) heat treatment for a formation of intermetallic phases or compounds. For example, a film of Ni matrix and Ni₃Al intermetallic alloys was successfully fabricated by codepositing a Ni matrix layer containing Al particles and subsequently annealing under a vacuum at (550 °C to 800 °C) for 2 hours [4].

Another important intermetallic alloy system with interesting functional properties in which the codeposition could be utilized as its alternative processing technique is nickel-titanium (NiTi). Due to their relatively high mechanical strain outputs under thermal stimulus as well as their superelasticity within room temperature proximity [4].

Electrochemical codeposition of Ni-Ti in composite coatings has been shown to impart many useful properties. Serek and Bundniok [5] reported improved mechanical properties of a steel substrate by Ni-Ti composite coating. The results showed rough metallic coating with very good adhesion to the substrate. Panek et al. [6] found that Ni-Ti composite layers exhibited greater hydrogen-evolution activities when compared to that of pure nickel coating. Both studies reported that the changes in the mechanical and chemical properties of Ni-Ti composite coatings depended on the amount of Ti particles embedded in the coatings. i.e. 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 titanium formation is possible (Fig .1) [7].

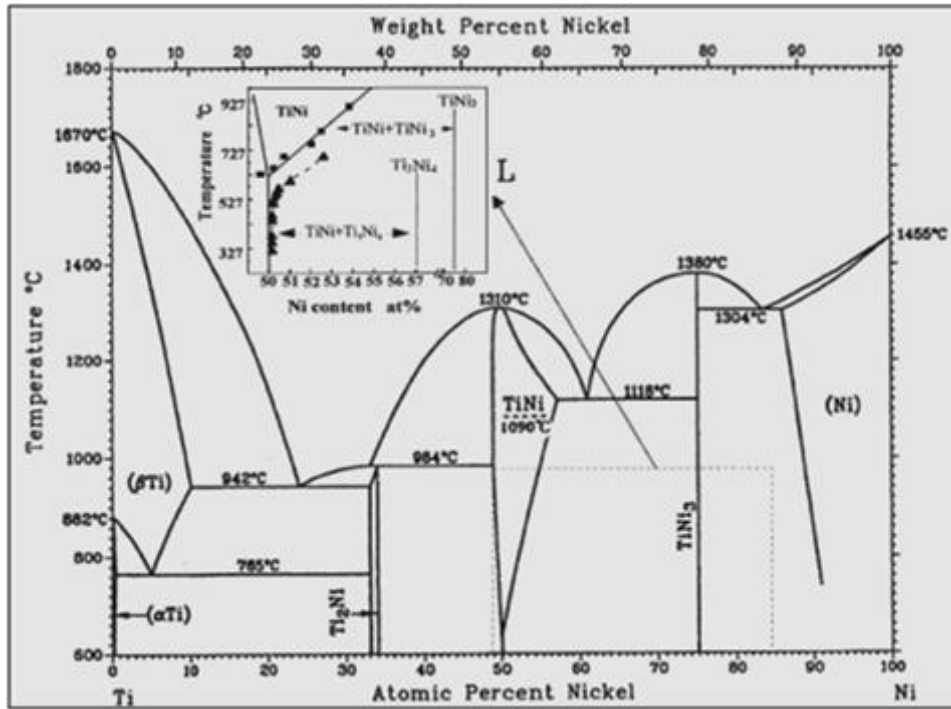


Figure 1 Equilibrium phase diagram for Ni-Ti [7].

In this study, we examine the influence of codeposition on the resulting composite morphology and the hardness of the coating and examines intermetallic phase development during heat treatment of Ni-Ti coatings. Further this work aims to gain an understanding of the oxidation behavior of Ni-Ti electrodeposited composite coatings as a function of exposure temperature and time.

2. EXPERIMENTAL METHODS

2.1. Substrate Alloy

The substrate alloy used in this study was Austenitic Stainless Steel (316 L). The nominal composition of austenitic stainless steel 316L alloy is shown in Table 1 [8]. Whereas Table 2 illustrates Spectrochemical analysis of austenitic stainless steel in wt. %.

Table 1 Limiting chemical composition of austenitic stainless steel in wt. % according to [8]

EL	Fe	C	Mn	Si	Cr	Ni	P	S	Mo	V	Cu
Wt. %	bal.	<0.02- <0.03	2.0	1.0	16-18	10-14	—	—	2-3	—	—

Table 2 Spectrochemical analysis of austenitic stainless steel in wt. %.

EL	Fe	C	Mn	Si	Cr	Ni	P	S	Mo	V	Cu
Wt. %	bal.	0.041	1.42	0.27	19.38	10.24	0.025	0.003	0.283	0.080	0.218

The Austenitic Stainless Steel (316 L) 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 hole 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. Electrochemical Codeposition Coating

Before Ni-Ti Electrochemical codeposition Coating begins, thin layer of Ni deposits as a preparation step were done according to the following steps [9]:

- Cold rinse.
- Dip in 20% hydrochloric acid (1.098 sp. gr.) at 21-27 °C for 1 min.
- Cold rinse.
- 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.
- Plate in wood's bath at 43 °C, using deposition current density of 0.0538 amps / cm² for 2 min.
- Cold rinse.

The aim of this step is to get a bonding layer (Ni) between Austenitic Stainless Steel (316 L) surface and the electrodeposited composite coating (Ni-Ti). Electrodeposition of Ni-Ti composites was performed in a typical watts bath on the Austenitic Stainless Steel (316 L). A nickel sulfamate electrolyte was used with the following composition: 300 g/l nickel sulfamate Ni (NH₂SO₃)₂, 30 g/l boric acid, 5 g/l nickel chloride (NiCl₂), 0.2 g/l sodium laurel 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 bath [11]. It was found that excessive amounts of the surfactant (coumarin) could cause "foaming" of the plating bath when Ti particles are added and, therefore, minimum levels of this additive were used.

After the plating bath was prepared, the Ti powder was added. The powders are codeposited within the nickel matrix to provide for nickel titanium formation. The titanium powder obtained from Changxing Galaxy International Trabe CO., LTD., high purity (99.65%), 20 μm max. diameter. Powder additions of 250 g/l were used in experiments.

The deposition was carried out in a plating bath containing 300 cm³ of the electrolyte solution. The substrate was placed horizontally inside the cell at 1 cm below the solution surface and held vertically in the stirred stream of the electrodeposition bath.

The bath was maintained at 50 °C and pH ≅ 4.0. It was found that deposition current density of about 4.5 amps / dm² produced the best results, and this current density was fixed for all coatings in the present research. To summarize Table 3 shows the major processing parameters for typical coating deposition

Table 3 Major Processing Parameters for a typical electrochemical codeposition coating.

Plating Bath	Nickel Sulfamate
The particle size~	20 μm max.
Particle loading (Ti)	250 g/l
Temp.	50 $^{\circ}\text{C}$
pH	4.0 – 4.1
Current Density	4.5 A/dm^2
Stirring	400 RPM
Plating Time	3 hrs.

After plating, the Ni-Ti composite coating was rinsed, ultrasonically cleaned with distilled water, and dried. Surface and the cross-sectional microstructures and chemical compositions of the coatings were analyzed using a scanning electron microscope (Olympus BX51M- Japan made), light optical microscope (LOM) (type (Tescan Vega III China made). For this purpose optical microscopy fitted by digital camera type (Smartic with 5 Mega pixels resolution) was used.

The coating cross-sectional areas were first mechanically polished with abrasive papers (240, 400, 800, 1200, 2500 SiC) and then polished using 5.0 μm and 0.3 μm alumina suspension sequentially, before ultrasonically cleaned with distilled water.

The thicknesses of the coatings were measured directly from the cross sectional microstructures; the thickness value is a mean value of 5 random thickness measurements along the coating's cross section. The phase composition of the coating was analyzed by X-ray diffractometry type (Minifcex II, Rgaku-200g-Japan made) with $\text{CuK}\alpha$ radiation ($U = 40 \text{ kV}$; $I = 20 \text{ mA}$).

2.3. Heat Treatment Process

To investigate nickel titanium coating formation, samples were heat treated in vacuum furnace (GSL-1600x). The pressure of the vacuum furnace was 10^{-4} torr. to avoid the oxidation of the underlying materials during the process

A two-stage heat treatment profile was conducted on the specimens for the formation of intermetallic alloys. The samples were heated from room temperature to (700 $^{\circ}\text{C}$) with a constant heating rate of (10 $^{\circ}\text{C}/\text{min}$) and subsequently heated to (950 $^{\circ}\text{C}$, 5 $^{\circ}\text{C}/\text{min}$) for 6 hours. at 950 $^{\circ}\text{C}$. A slow heating rate was chosen to avoid a severe reaction of Ni and Ti. After heat treatment, the Ni-Ti samples were furnace-cooled to room temperature.

2.4. Micro Vickers Hardness Tests

Micro Vickers hardness tests were carried out to evaluate the hardness of composite coatings using Micro Hardness tester (Tester TH 717). Coating surfaces were mechanically polished first with abrasive papers and then with diamond pastes, before rinsing them in water, and indenting them with the 10 g load for 10 seconds. The final hardness value is the average value of 5 random test measurements made on each coating surface.

2.5. Isothermal Oxidation in Dry Air

The prepared samples were suspended with platinum wire and inserted into a tube furnace held at temperature (700, 800, and 900 ± 3 $^{\circ}\text{C}$) where they were oxidized in dry air at 1 atmospheric pressure for up to 50 hrs. duration (Fig. 2).

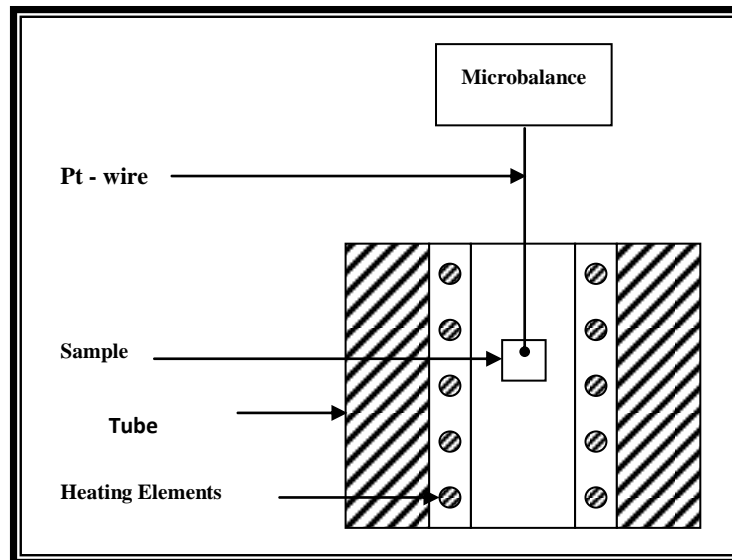


Figure 2 Apparatus used for isothermal oxidation tests in dry air.

The Ni-Ti composite coated samples were oxidized isothermally and the weight gain was monitored and recorded at suitable intervals. Test temperature was controlled within ± 3 °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.

3. RESULTS AND DISCUSSION

3.1. Phase analysis of the codeposited Ni-Ti coatings

The Ti-dispersed Ni-matrix composite coatings were successfully produced through codeposition. The obtained coatings have shown a uniform gray rough metallic surface with grains of incorporated Ti particles.

The representative XRD results are shown in Fig. 3. It is found that all the as-deposited samples are characterized by a similar XRD pattern showing Ni and Ti peaks with no sign of any other oxide or metallic compound phases.

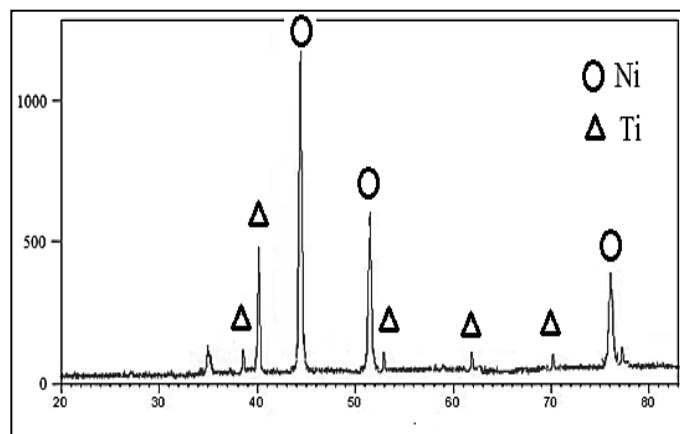


Figure 3 The XRD patterns of the Ni-Ti composite coating deposited at 4.5 A/dm² from the plating baths loaded with Ti particles loading at 250g/L

The XRD results of these samples following the heat treatment protocol, holding at 950 °C for 5 hours presented in Fig. 4, show that the post annealed Ni-Ti specimen consists of two major phases including NiTi and Ni₃Ti. Evidently, the Ni and Ti phases present in the as-deposited samples completely transform into Ni-Ti intermetallic compounds.

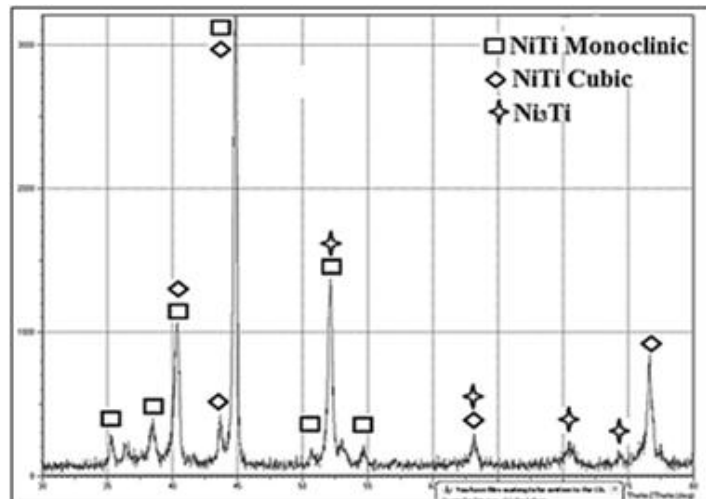


Figure 4 The XRD patterns of the heat-treated Ni-Ti composite coating sample

On other hand, the results of atomic absorption spectroscopy test (type Phoenix-986s Japan made) indicated that the average titanium concentration is $\cong 35\%$.

3.2. Hardness of the codeposited Ni-Ti coatings

Without Ti particle loading, the hardness of the electrodeposited Ni is about 120 HV. With a presence of Ti loading in the bath (250 g/L, the hardness of the Ti-reinforced coatings was significantly higher than that of pure Ni coating (175 HV). The presence of dispersed Ti particles in coatings, which has enhanced the hardness of the coatings has also been observed in composite Ni-matrix coatings with different reinforced particles such as WC, SiC, and TiO₂ [12, 13, and 14].

Increased composite coating hardness obtained in this study is possibly related to the particle strengthening mechanism since the particle size used in this study was relatively large (with a particle size distribution of $\sim 20\ \mu\text{m}$ max.) and the reinforcement volume fraction used $\sim 35\%$. Thus, the hardness is enhanced with the reinforcement of the incorporated Ti particles.

3.3. Morphology of the codeposited Ni-Ti coatings

When Ti particles are dispersed into bath, they develop a positive charge due to a layer of Ni⁺² ions on their surface. Bath stirring supplies particles at a constant rate to the substrate surface where they adhere to the relatively charged substrate [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-Ti composite coatings.

The Scanning electron microscopy observations showed that all the electrodeposits contained Ti particles became included in the surface involves the

formation of weak chemical bonds between the surface and Ti 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 Ti particles throughout Ni matrix (Fig. 5).

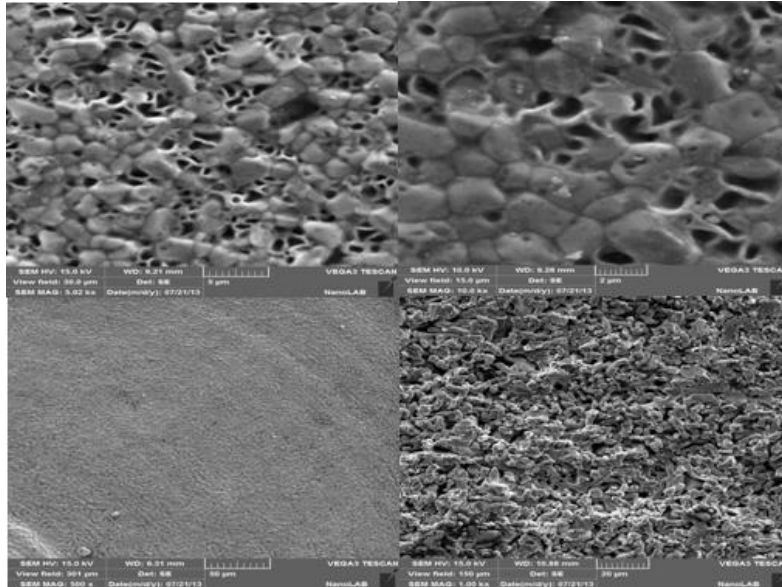


Figure 5 Surface morphology of the Ti-dispersed Ni-matrix coating

Further, Fig. 6 shows light optical microscopy image of the cross section of heat treated Ni- Ti composite layer, which has thickness of about 115 μm , with no cracks or other defects, and good bonding with the substrate.

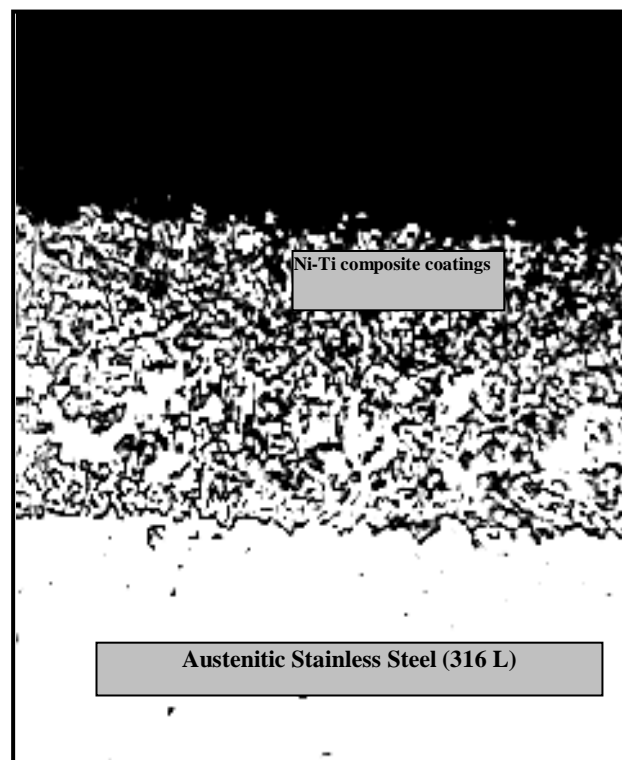


Figure 6 Cross-sectional images of the Ni-Ti composite coatings (Magnification 300X).

3.4. Isothermal Oxidation in Dry Air

Weights changing were recorded for kinetics identification in dry air in the temperatures range 700 – 900 °C for up to 50 hrs. The specific weight gain data of the Ni-Ti electrodeposited coated Austenitic Stainless Steel (316 L) alloy for each temperature test is plotted in Figures 7 and 8 as a function of time. The initial kinetic is rapid, but the rate of specific weight gain 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 [15]:

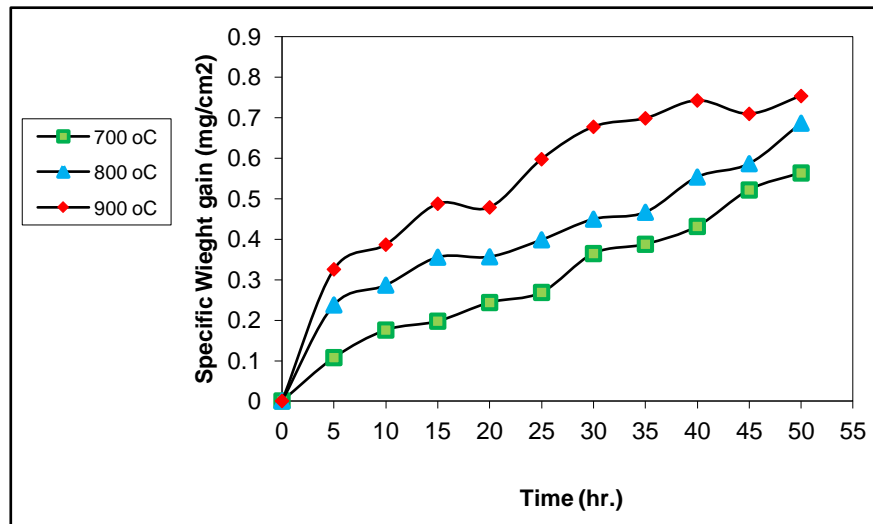


Figure 7 Specific weight gain vs. time plot for Ni-Ti electrodeposited composite coating of Austenitic Stainless Steel (316 L) alloy oxidized in air at temperatures between 700 and 900 °C for 50 hrs.

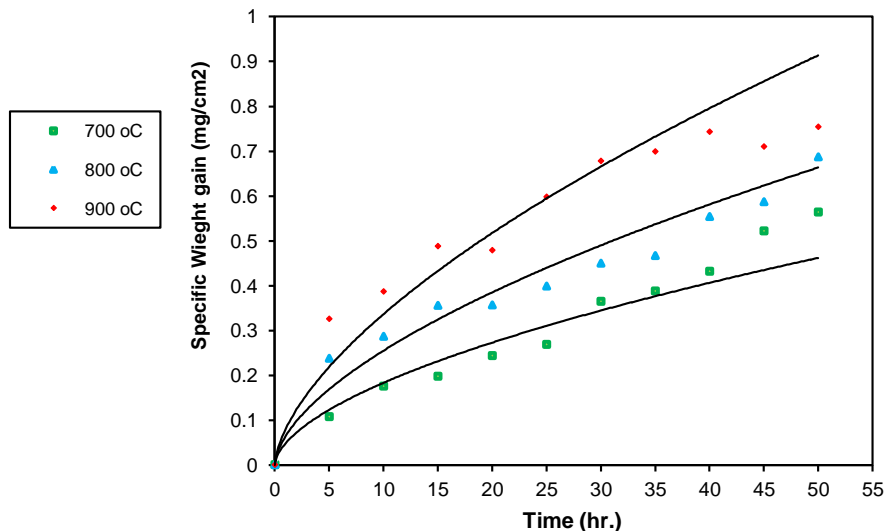


Figure 8 Parabolic fitted results of Specific weight gain vs. time plot for Ni-Ti electrodeposited composite coating of Austenitic Stainless Steel (316 L) alloy oxidized in air at temperatures between 700 and 900 °C for 50 hrs

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

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 700°C, the n-value is about 0.57, 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 [15].

Table 4 n values and parabolic oxidation rate constants k_p for isothermal oxidation for 50 hrs. of Austenitic Stainless Steel (316 L) alloy with Ni-Ti electrodeposited composite coating

Temperature (°C)	n values	k_p (mg ² /cm ⁴)/s.
700	0.57	6.696×10^{-7}
800	0.59	1.177×10^{-6}
900	0.62	1.813×10^{-6}

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 [15]:

$$\Delta W/A = k t^{0.5} \tag{2}$$

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. 9; 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:

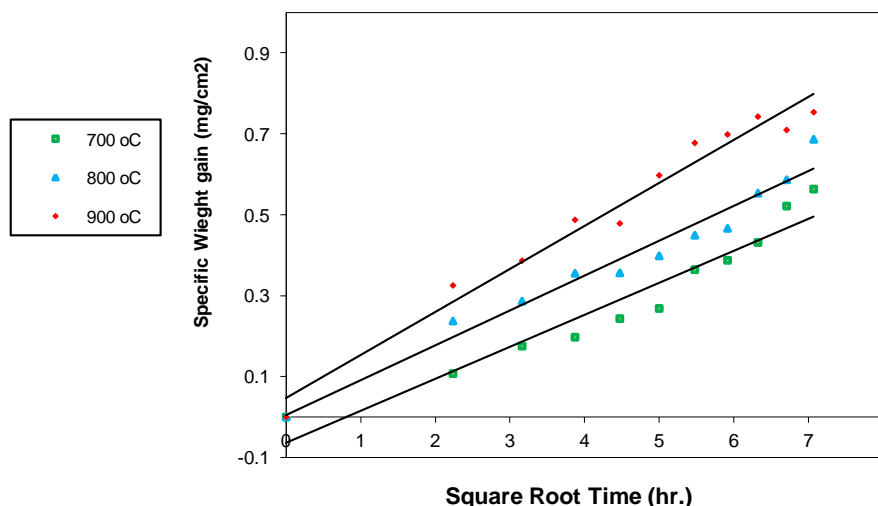


Figure 9 Linear fitted results of specific weight change vs. $t^{0.5}$ plot for Ni-Ti electrodeposited composite coating of Austenitic Stainless Steel (316 L) alloy oxidized in air at temperatures between 700 and 900 °C for 50hrs.

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

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.

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

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

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 ($R^2 = 0.93$) of the observed data in the temperature range from 700 to 900 °C is to be ~ 55 KJ/mol. as shown in Fig. 10.

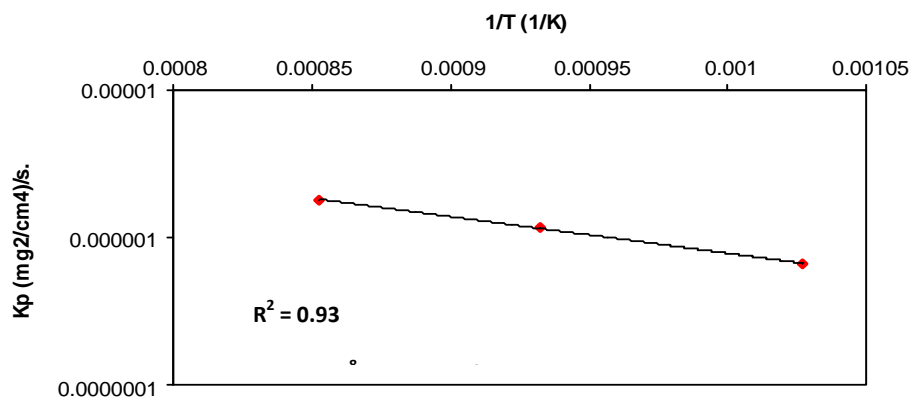


Figure 10 Plot of k_p vs. $1/T$ for Ni-Ti electrodeposited composite coating of Austenitic Stainless Steel (316 L) alloy oxidized in air at temperatures between 700 and 900 °C for 50 hrs.

When composite coating containing Ti is exposed to a high- temperature oxidation environment, TiO_2 scale is formed on the surface of the alloy. This scale prevents the rapid oxidation of nickel, Cr, 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 titanium composite coating was found to be NiTi phase. Isothermal oxidation at oxidized test temperature (700 - 900°C) resulted in the formation of TiO_2 on the sample surface and, as anticipated, its amount increased with

exposure duration (Figures 11, 12 and 13), which show 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, even after 50 hrs. of exposure. Thus, the composite coating in this study appears to be fairly adequate in providing isothermal oxidation protections to the Austenitic Stainless Steel (316 L) alloy up to 900°C.

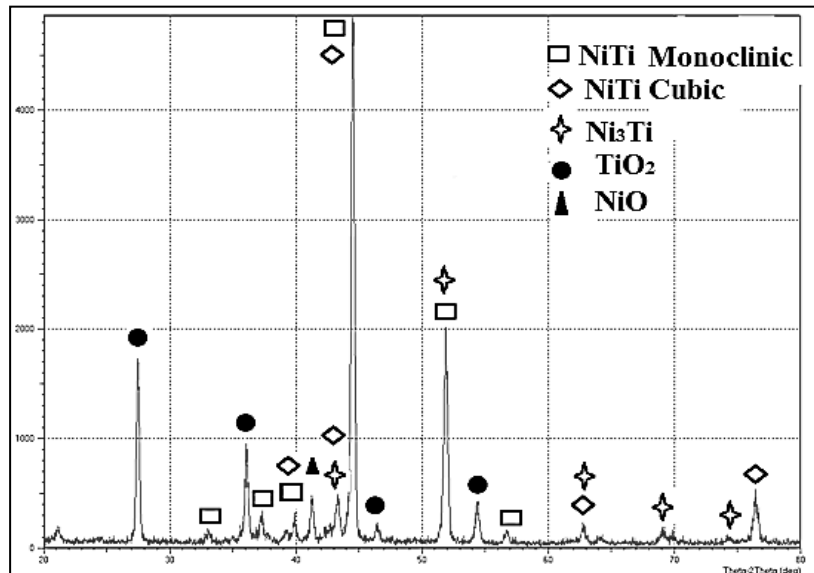


Figure 11 Diffractograms from the surface of with NiTi electrodeposited composite coating after isothermal oxidation in dry air at 700 °C for 50 hrs.

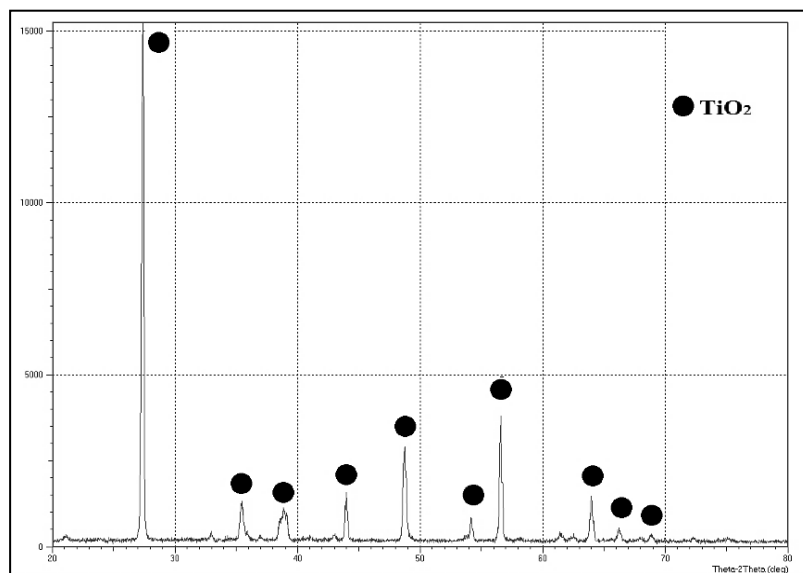


Figure 12 Diffractograms from the surface of with NiTi electrodeposited composite coating after isothermal oxidation in dry air at 800 °C for 50 hrs.

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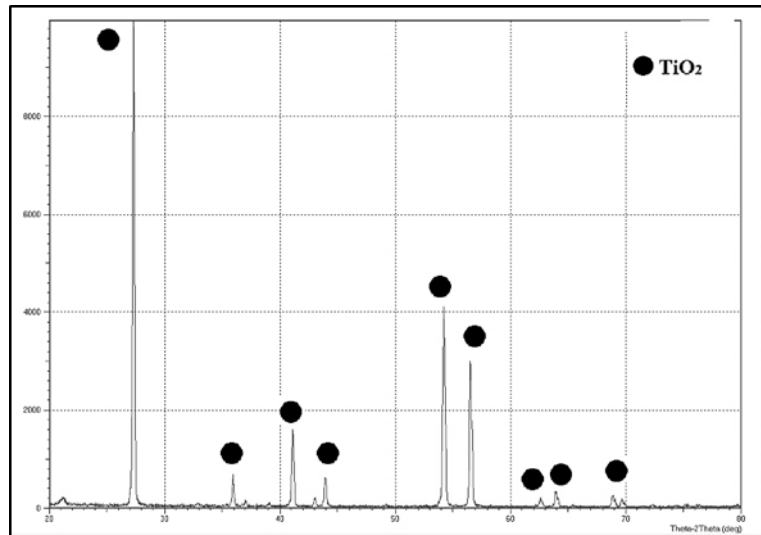


Figure 13 Diffractograms from the surface of with NiTi electrodeposited composite coating after isothermal oxidation in dry air at 900 °C for 50 hrs.

Furthermore, Fig. 14 show cross-sectional view of LOM of the nickel titanium composite coating after isothermal oxidation in air at 900 °C for 50 hrs. From the surface appearance of the sample, it does not show severe scale spalling and appeared uniform and smooth after isothermal oxidation.

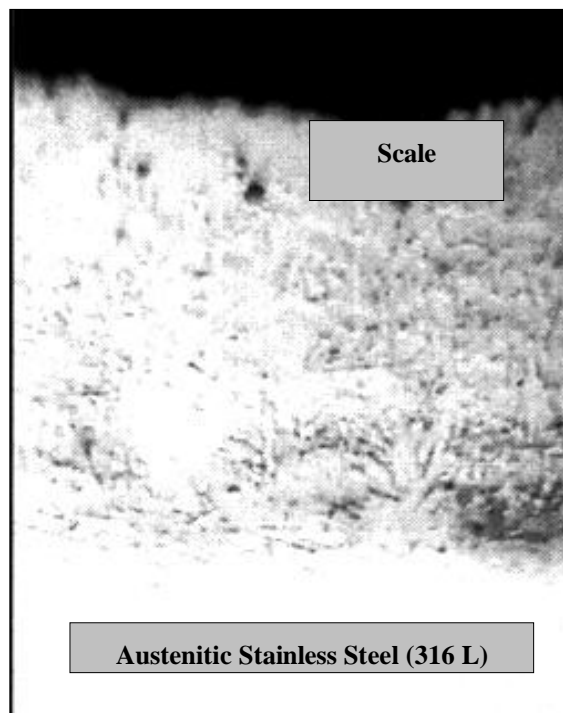


Figure 14 Cross-sectional images Ni-Ti composite coatings after isothermal oxidation in air at 900 °C for 50 hrs (300X)

4. CONCLUSIONS

1. The Ni-Ti composite coating was successfully produced through the electrochemical codeposition route. The composite coating produced a gray, rough metallic surface with thickness ranging up to 115 μm and Ti content up to 35%.
2. Heat treatment at 950 $^{\circ}\text{C}$ for 3 hrs. under vacuum atmosphere results in diffusion reaction between the Ti particles and nickel matrix to produce a range of Ni-Ti Intermetallic compounds like NiTi and Ni₃Ti phases.
3. Over the temperatures range between 700-900 $^{\circ}\text{C}$, the parabolic rate constant (k_p) varies from a ($k_p = 6.696 \times 10^{-7} \text{ (mg}^2/\text{cm}^4\text{)}/\text{s.}$) at 700 $^{\circ}\text{C}$ to a value of ($k_p = 1.813 \times 10^{-6} \text{ (mg}^2/\text{cm}^4\text{)}/\text{s.}$) at 900 $^{\circ}\text{C}$.
4. Titanium dioxide is the major phase that exists in the outer layer under oxidation conditions.

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