

# High Effect of 22Cr10AlY Coating Thickness on Air Hot Corrosion of Ni Based Alloy

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**Abstract** - Oxidation due to hot corrosion is a serious problem for the components and the structures exposed to elevated temperature specifically in case of aircraft, marine, power plants and land-based gas turbines. This is because of the usage of wide range of fuels coupled with higher operating temperatures. The higher operating temperature of these systems leads to the degradation of material properties and increase the chances of failure of the components or the system as whole. In past there are several occasions where the failure has occurred due to hot corrosion, which leads to heavy losses in monetary term as well as the loss of human life. Many materialists' come up with several solutions like new material (alloy) that has ability to with stands high temperature for long time without degradation. In such case the cost of the setup also increases. The other economical alternative solution of the above problem is to have a thermal barrier coating on the surface of the component exposed to higher temperature. To obviate these problems, Ni alloy and coated Ni alloys, are the prominent materials for the high temperature applications. It is very essential to investigate the degradation mechanism of alloys due to oxidation and hot corrosion and substantiate the role of alloying elements for the formation of protective oxide films over the surface of the Ni alloys.

The present work investigates the effect of coating thickness on oxidation and hot corrosion behavior of Ni alloys exposed to air at 850°C and 900°C under cyclic conditions. The weight change measurements made on the alloys during the experiments are used to determine the kinetics of oxidation and hot corrosion. Scanning electron microscope (SEM) is used to observe the corroded products of the Ni alloys. It is observed that coated Ni alloy 100 micron, contributed for the better resistance to oxidation and hot corrosion of coated Ni alloy 50 micron 900°C. Whereas lesser hot corrosion resistance of Ni alloys is due to the formation of non-protective oxides. The formation of a dense oxide scale formed on the coated alloys and hot corrosion resistance of coating might be due to the formation of protective phases like NiO, Cr<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, NiCr<sub>2</sub>O<sub>4</sub>, and NiAl<sub>2</sub>O<sub>4</sub>.

**Key Words:** High effect of 22Cr10AlY coating thickness on air hot corrosion of NI based alloy.

## 1. INTRODUCTION

Gas turbines have progressed greatly over the last 70 years due in part to academic and industrial research efforts focused on increasing the operating temperature of the engine, thereby increasing the efficiency of the engine. Advancements such as single-crystal turbine blades and thermal barrier coatings (TBCs) have aided significantly in the increase in temperature for aero turbines and industrial gas turbines (IGTs), The development of thermal barrier coating (TBC) system coupled with modern cooling systems has allowed for an almost 150° C reduction at super alloy blade surface temperature thereby allowing the gas turbine combustion temperature to be above the melting temperatures of super alloys. The technological developments in super alloys over the time providing an increase in the operational temperature .The expanding market of IGTs for power generation has influenced research as well. The ever increasing temperature requirements for turbines result in oxidation becoming a primary degradation mode. In addition, as the overall operating temperature in the turbine continue to rise; certain components are becoming susceptible to degradation via hot corrosion [1].

The simple model of the gas turbine is broken into two sections, the cold section and the hot section, the cold section is comprised of the intake and the compression segments of the engine. The intake draws air into the engine using larger vanes connected to the driveshaft of the turbine. The incoming air is then compressed with a different set of vanes that are connected to the drive shaft. The compressed air then enters the hot section of the engine.

The hot section consists of the combustor, turbine, and exhaust segments. The compressed air then enters the combustor segment where fuel is injected and combustion occurs. The rapidly expanding gas exits the combustor and enters the turbine segment of the engine. Here the expanding gas travels between alternating sets of stator and rotor turbine blades. The gas forces the turbine section to rotate, in turn powering the cold section of the turbine as well any equipment connected to the shaft. In IGTs the hot exhaust gases are used to power secondary steam turbines (combined cycle configuration) or to heat buildings or water (cogeneration configuration).

There are two distinct modes of attack that occur in gas turbine engines. Oxidation occurs at elevated temperatures and is the primary mode of attack above 1000°C. Below 1000°C, hot corrosion is the main form of degradation. These two modes of attack are discussed. The coatings currently used in the turbine industry vary greatly in composition and application method. The coatings used ultimately reflect the main type of attack associated with the service environment (e.g., aluminides for oxidation protection and MCrAlX coatings for hot corrosion environments).

**1.1. Objectives of the present work**

1. Investigate the hot corrosion of coatings deposited on the selected Ni alloy.
2. Weight change technique has been used to establish the kinetics of hot corrosion of the coated Ni alloys and Ni alloys exposed environment, under cyclic conditions at 850°C and 900°C.
3. The micro structural characterizations of the coated samples and corrosion products were carried out by SEM mechanisms.

**2. Methodology**

**2.1. Problem statement**

The materials used for high temperature strength are highly susceptible to hot corrosion and the surface engineering plays a key role in effectively combating the hot corrosion and oxidation problems. The literature reviewed on the topic revealed that almost less work has been conducted in the past with a view to improve the coatings 22Cr10AlY coating thickness on air hot corrosion of NI based alloy.

**2.2. Material**

Nickel- based alloy were used as the substrate, which were procured from Mishra Dhatu Nigam Limited, Delhi (India) in the rolled sheet form. Nominal compositions of the Ni alloy substrate materials are shown in Table 2.1. Rectangular 6 specimens of dimensions (in mm) 10\*10\*5 were cut from the alloy sheets, polished using emery papers of 220, 400, and 600 grit sizes and subsequently on 1/0, 2/0, 3/0, and 4/0 grades. Samples were degreased with acetone and subsequently grit blasted with alumina powders (Grit 45) (36 mesh size) before deposition of the coating by the D-gun process.

Table 2.1 - Chemical composition of the substrate material-

Alloy Midhani grade (similar grade)	Chemical composition (wt.%)										
	Fe	Ni	Cr	Ti	Al	Mo	Mn	Si	Cu	Ta	C
Ni base alloy	3.0	77.1	19.5	0.3			0.1				

**2.3 Development of Coating**

A commercially available NiCrAlY (particle size: 5/45 micron, H.C. Starck-413.3) powder with the chemical composition of Ni-22Cr-10Al-1Y was chosen for the deposition of coatings. Figure (3.1) show that the particles are of spherical shape with wide particle size distribution, ranging from 5 to 35 micron, which are nearly consistent with the nominal size range provided by the manufacturer. The particle size less than 5 micron depicts the presence of cerium oxide. A mixture of CeO<sub>2</sub> (0.4 wt.%) with 99.99% purity and NiCrAlY powder were dry-ball milled in a conventional rotating ball mill with stainless steel balls as a milling/grinding medium for 8 h to obtain the homogenous powder for the deposition of coatings.

**2.4 Characterization of oxidation**

To observe the cyclic oxidation of the Ni alloy and coated Ni alloy samples they are heated in Muffle furnace as shown in Fig 2.1



**Fig 2.1 Muffle furnace**

### 2.5 Characterization of weight

The samples weight either increased or decreased are measured with help of digital weight machine as shown in fig(2.2).



**Figure 2.2 Digital weighing balances**

### 2.6 Characterization of Surface

To observe the surface of hot corrosion samples Scanning Electron Microscopy (SEM) to be done, Figure (2.3) shows the machine used for doing SEM, available at IIIT Kanpur.

#### Essential specifications of SEM

<b>Electron Gun</b>	<b>Tungsten heated filament/optionally <math>L_aB_6</math></b>
<b>Chamber size</b>	<b>300 mm (width) × 310 mm (height)</b>
<b>Accelerating Voltage</b>	<b>200V to 30 KV</b>
<b>Probe current</b>	<b>1 Pa to 2<math>\mu</math>A</b>
<b>Resolution</b>	<b>3 nm at 30 KV, 8 nm at 3 KV</b>
<b>Magnification</b>	<b>20 to 10000 x</b>



**Fig 2.3. TECSAN VEGA Scanning Electron Microscopy (SEM)**

## 2.7 Detonation-Gun Coating for specimen preparation

D-gun process was used to apply coatings on rectangular 4 specimens of dimensions (in mm) 10\*10\*5 at SVX Powder M Surface Engineering Pvt. Ltd, New Delhi (India). Standard spray parameters were used for depositing the NiCrAlY + 0.4 wt.% CeO<sub>2</sub> coatings. All the process parameters, including the spray distance, were kept constant throughout the coating process. The spraying parameters were oxygen/acetylene flow rate (ratio) 1:1.21, carrier gas flow rate (N<sub>2</sub>) 0.82(m<sup>3</sup>/h), frequency 3 shots/s, spot size diameter 20 mm, and spraying distance from nozzle to 165 mm. The thickness of the coatings was measured from the optical microscope images, taken along the cross section of the mounted samples. The average thicknesses of the rectangular 2 specimen's coatings range were 50 micron and other 2specimen'scoatings range were 100 micron.

## 2.8 Hot Corrosion Oxidation Testing

The cyclic oxidation studies were performed in air environment for 10 cycles. Each cycle consisted of 5 hr heating at 850°C and 900°C for 3 specimens each (Ni alloy in Figure [2.4], coated Ni alloy 50 micron in Figure[2.5] and coated Ni alloy 100 in Figure[2.6] in a Muffle furnace in Figure(2.1) ,followed by 20 min cooling at room temperature. The specimens were kept in alumina boat. Weight of alumina boat with samples was taken after every cycle using digital weighing balance in Figure (2.2) of 1mg accuracy. Alumina boats were preheated for 8 hr at 850°C and 900°C so as to remove manufacturing defect present in it. It was assumed that the weight of the alumina boats will remain constant during experiment. The weight change measurements were also included to determine total rate of corrosion. The kinetics of corrosion was determined from weight gain plot of weight change/unit area with time. SEM techniques were used to analyze the corrosion products.



**Figure 2.4 Ni alloy**



**Figure 2.5 Coated Ni alloy 50 micron**



**Figure 2.6 Coated Ni alloy 100 micron**

### 3 RESULTS AND DISCUSSION

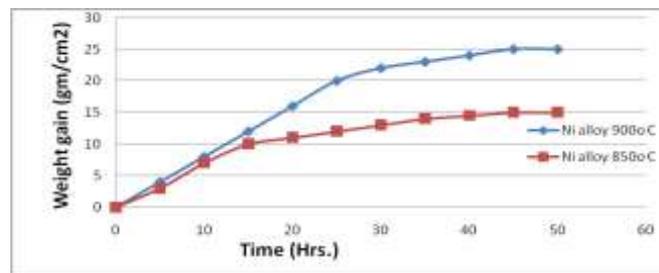
#### 3.1 Weight Change Measurements

The kinetics of hot corrosion were determined from the weight change (mg/cm<sup>2</sup>) versus time plots for the Ni alloy and coated Ni alloy subjected to hot corrosion in environment at 850°C and 900°C up to 10 cycles as given below by tables and graphs.

**Table (3.1) Reading for Ni alloy at 900°C and 850°C**

Time (hour)	Reading at 900°C for Ni	Reading at 850°C for Ni
	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )
5	4	3
10	8	7
15	12	10
20	16	11
25	20	12
30	22	13
35	23	14
40	24	14.5
45	25	15
50	25	15

i) Ni alloy 850°C shows lower weight gain than Ni alloy 900°C in Fig (3.1)



**Fig 3.1 Weight gain/Surface area (mg/cm<sup>2</sup>) versus plot for Ni alloy 900°C and Ni alloy 850°C subjected to cyclic oxidation.**

**Table (3.2) Reading for Coated Ni alloy at 900°C and 850°C for 50 micron.**

Time(hour)	Coated Ni alloy(50microns) at 850°C	Coated Ni alloy(50microns) at 900°C
	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )
5	0.5	1
10	1	2
15	2	4
20	2.5	5
25	3.5	7
30	5	9
35	7	11
40	9	12
45	10	13
50	11.5	14

ii) Coated Ni alloy 50 micron 850°C shows lower weight gain than Coated Ni alloy 50 micron 900°C in Fig (3.2)

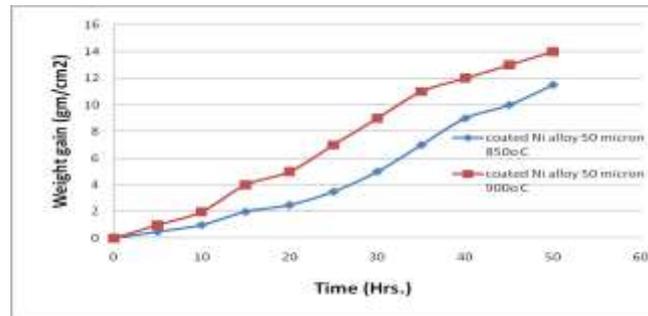


Fig 3.2 shows the variation of Weight gain/Surface area(mg/cm<sup>2</sup>) versus time for Ni alloy having the coating of 50microns hot corroded at 900°C and 850°C.

Table (3.3) Reading for Coated Ni alloy at 900°C and 850°C for 100 micron

Time(hour)	Coated Ni alloy(100microns) at 850°C	Coated Ni alloy(100microns) at 900°C
	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )
5	0.1	0.5
10	0.3	1
15	0.5	2
20	0.8	3
25	1	4
30	1.2	5
35	1.5	5.5
40	1.8	6
45	2	6.5
50	2.2	7

iii) Coated Ni alloy 100 micron 850°C shows lower weight gain than Coated Ni alloy 100 micron 900°C in Fig (3.3)

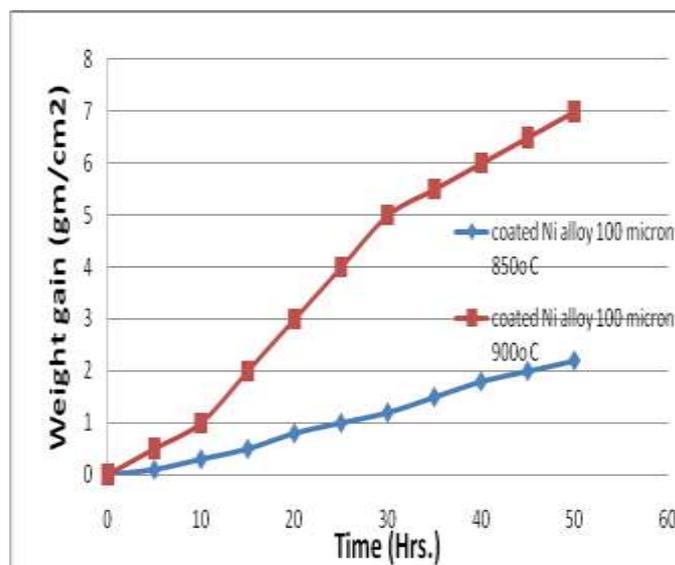
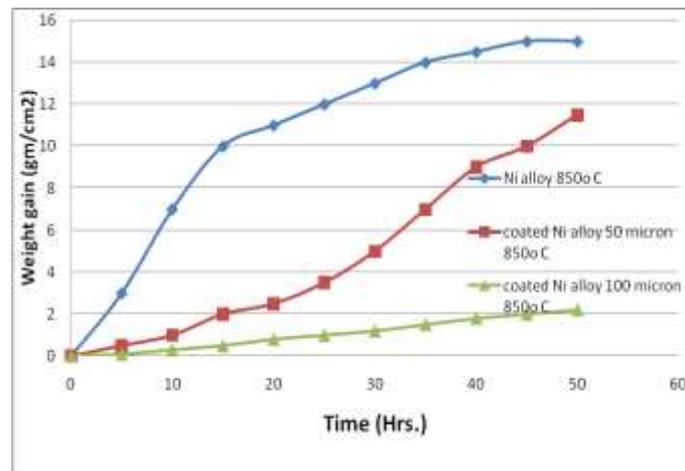


Fig 3.3 Weight gain/Surface area (mg/cm<sup>2</sup>) versus number of cycles plot for coated Ni alloy 100micron 900°C and coated Ni alloy 100 micron 850°C subjected to cyclic oxidation.

**Table (3.4) Reading for Ni alloy, Coated Ni alloy at 50 micron and Coated Ni alloy at 100 micron for 850°C**

Time (hour)	Reading at 850°C for Ni	Coated Ni alloy(50microns) at 850°C	Coated Ni alloy(100microns) at 850°C
	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )
5	3	0.5	0.1
10	7	1	0.3
15	10	2	0.5
20	11	2.5	0.8
25	12	3.5	1
30	13	5	1.2
35	14	7	1.5
40	14.5	9	1.8
45	15	10	2
50	15	11.5	2.2

iv) Coated Ni alloy 100 micron 850°C shows lowest weight gain than Coated Ni alloy 50 micron 850°C, and Coated Ni alloy 50 micron 850°C shows lower weight gain than Ni alloy 850°C in fig(3.4).

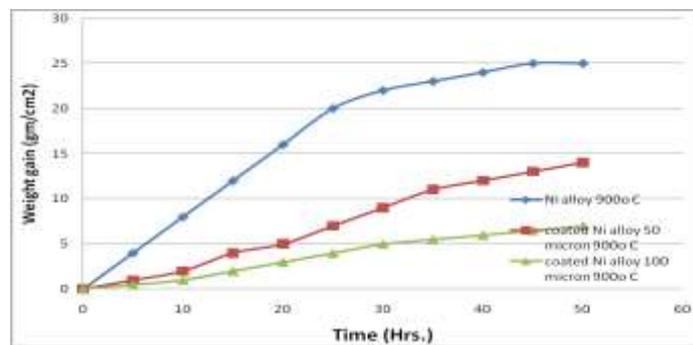


**Fig 3.4 Weight gain/Surface area (mg/cm<sup>2</sup>) versus number of cycles plot for coated Ni alloy 850°C, Ni alloy 50micron 850°C and coated Ni alloy 100 micron 850°C subjected to cyclic oxidation.**

**Table (3.5) Reading for Ni alloy, Coated Ni alloy at 50 micron and Coated Ni alloy at 100 micron for 900°C**

Time(hour)	Reading at 900°C for Ni	Coated Ni alloy(50microns) at 900°C	Coated Ni alloy(100microns) at 900°C
	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )	weight gain(mg/cm <sup>2</sup> )
5	4	1	0.5
10	8	2	1
15	12	4	2
20	16	5	3
25	20	7	4
30	22	9	5
35	23	11	5.5
40	24	12	6
45	25	13	6.5
50	25	14	7

v) Coated Ni alloy 100 micron 900°C shows lowest weight gain than Coated Ni alloy 50 micron 900°C, and Coated Ni alloy 50 micron 900°C shows lower weight gain than Ni alloy 900°C. in fig (3.5).



**Fig 3.5 Weight gain/Surface area (mg/cm<sup>2</sup>) versus number of cycles plot for coated Ni alloy 900°C, Ni alloy 50micron 900°C and coated Ni alloy 100 micron 900°C subjected to cyclic oxidation.**

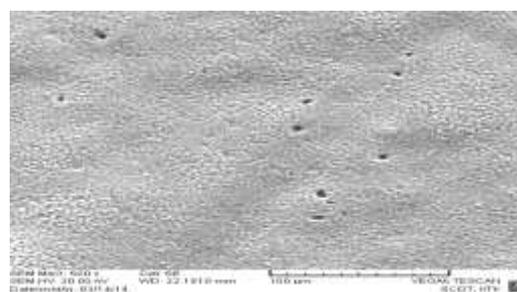
In general, the hot corrosion behavior of both Ni alloy and coated samples follow a parabolic rate law, except for Coated Ni alloy 100 micron 850°C, as it slightly deviates from parabolic rate, as can be inferred from the square of weight change (mg<sup>2</sup>/cm<sup>4</sup>) versus number of cycle plots.

The weight gain graph shows that the weight gained by Ni alloys increases continuously due to accelerated oxidation in the environment, where as the coated Ni alloys in all cases showed better hot Corrosion resistance. The Coated Ni alloy 100 micron 850°C and Coated Ni alloy 100 micron 900°C shows lowest weight gain and the hot corrosion, respectively.

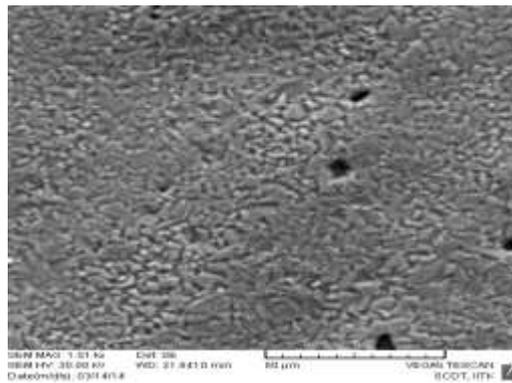
The weight gain of the coated and uncoated specimens is relatively high during the early cycles of hot corrosion, but subsequently increase in weight gain is found to be gradual. Protective oxide of Al has not formed, because Ni and Cr on the surface were oxidized at a faster rate. As the oxidation cycle proceeds, the air entrapped during D-gun deposition and sheltered in the porosities, since the cooling of coating was rapid. There is shortage of time for the residual air to react with the surrounding coating alloys. However, the coating took in reaction during high temperature at 850°C and 900°C oxidation and formed alumina (Al<sub>2</sub>O<sub>3</sub>) at the splat boundaries and with in open pores. During subsequent cycles the formation of oxides has blocked the pores, splat boundaries and acted as diffusion barrier to the inward diffusion of corrosive species. This leads to slow oxide scale growth. All the Ni alloys followed parabolic behavior where as coated Ni alloys deviates from the parabolic rate law except for Coated Ni alloy 100 micron 850°C. Coated Ni alloy 100 micron 900°C and Coated Ni alloy 100 micron 850°C coating provides maximum hot corrosion resistance in their in their temperature range.

### 3.2 Surface micrograph of the Ni alloy and Coated Ni alloys

From SEM micrographs (Fig. 3.5, 3.6 and 3.7), after cyclic oxidation test performed on the alloys at 900°C, dull dark grey oxide layer was formed on the surfaces of the alloys. Little pinch of green color was also observed on the surface which may be due to the presence of Ni O. Initial weight gain is little more because of diffusion of chromium and oxygen which become less with the passage of time. From SEM micrographs (Fig. 3.5, 3.6 and 3.7), the oxide layer was found to be compacting dense and more adherent to alloys.



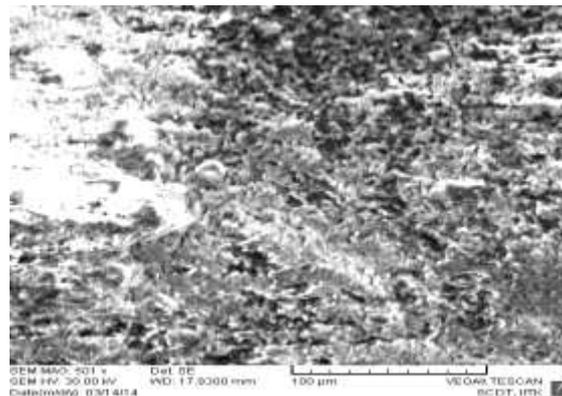
(a)



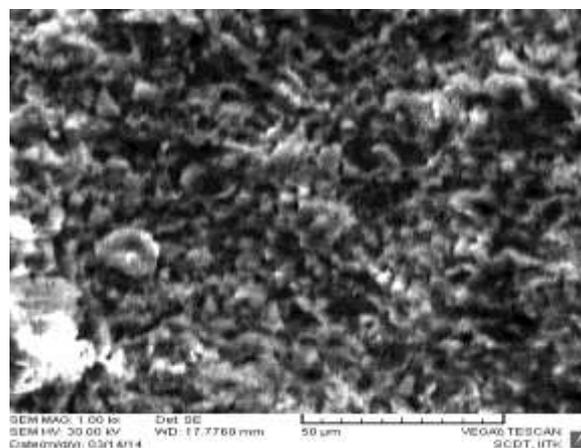
(b)

**Fig. 3.5 SEM micrographs for Ni alloy subjected to cyclic oxidation in air at 900°C after 10 cycles (a) at magnification of 500x and (b) at magnification of 1000X**

In SEM micrographs (Fig. 3.2.2 and 3.2.3) it was also observed that chromium and oxygen are present in major amount in scale of the alloys which means oxide of chromium is present inside the scale which is responsible for the protection of the alloy. Formation of chromium oxide may be because of the affinity of chromium towards oxygen at high temperature in SEM micrographs (Fig. 3.2.2 and 3.2.3). SEM micrographs (Fig. 3.2.2 and 3.2.3), which allows the penetration of corrosive species through the scale to the coating. The NiO formed in the surface scale is porous due to re-precipitation by fluxing action; due to which the oxygen has penetrated little deep into the coating as attributed.

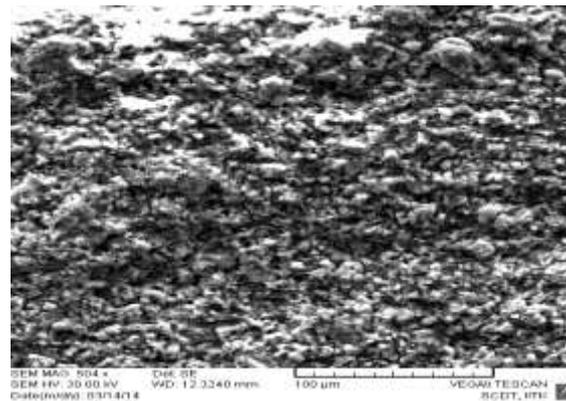
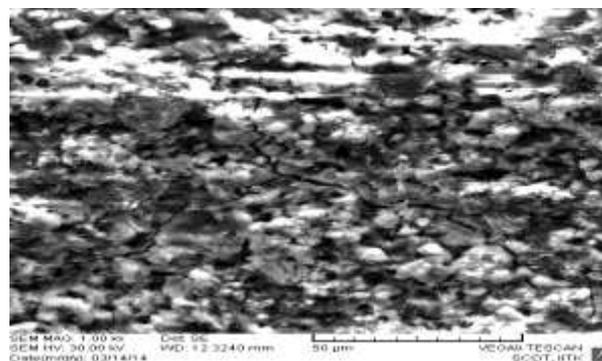


(a)



(b)

**Fig. 3.6 SEM micrographs for Coated Ni alloy 50 micron subjected to cyclic oxidation in air at 900°C after 10 cycles (a) at magnification of 500x and (b) at magnification of 1000X**


**(a)**

**(b)**

**Fig. 3.7 SEM micrographs for Coated Ni alloy 100 micron subjected to cyclic oxidation in air at 900°C after 10 cycles (a) at magnification of 500x and (b) at magnification of 1000X**

#### 4. CONCLUSIONS

On the basis of present research, following conclusions can be drawn

- All the Coated Ni alloy are resistant to oxidation and hot corrosion at 850°C and 900°C for 10 cycles.
- The Ni alloy show least and coated Ni alloy 900° C show highest resistance to the hot corrosion, respectively.
- D-gun-sprayed NiCrAlY + 0.4 wt. % CeO<sub>2</sub> coating found to be effective in imparting hot corrosion resistance to all Coated Ni alloy in cyclic oxidation in air.
- The formation of oxides along the splat boundaries and within open pores of the coatings might have acted as diffusion barrier to the inward diffusion of molten salt.
- A dense oxide scale formed on the coated alloys and hot corrosion resistance of coating might be due to the formation of protective phases like NiO, Cr<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, NiCr<sub>2</sub>O<sub>4</sub>, and NiAl<sub>2</sub>O<sub>4</sub>.

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