

# CORROSION STUDIES OF HVOF COATED BOILER TUBE MATERIALS

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**Abstract:** The high velocity oxy-fuel (HVOF) process belongs to the family of thermal spray processes and has grown into a well-accepted industrial technology. This process has been shown to produce coatings with better density, coating cohesive and bond strength than other thermal spray processes. The hot corrosion behavior of high velocity oxy-fuel sprayed (WCNi 65%+NiCr35%) coating on substrate alloy T22 was investigated. Thermocyclic hot corrosion studies were performed in a molten salt environment of Na<sub>2</sub>SO<sub>4</sub>-60% and V<sub>2</sub>O<sub>5</sub>-40% for 50 cycles at 700°C. It was observed that the coated steel showed better hot corrosion resistance than the uncoated steels. The coated steels showed slow oxidation kinetics which indicated that the reaction rate is diffusion limited. The oxides of Cr<sub>2</sub>O<sub>3</sub> and NiO formed on the outermost surface of the coatings are found to be passive in the molten NaVO<sub>3</sub> melt. It is seen from the results that the cumulative weight gain for all the HVOF coated steels are significantly lower than that of uncoated steels subjected to molten salt hot corrosion.

**Index Terms** – HVOF, Coatings, SA213-T22 Alloy, SEM/EDAX analysis.

## 1. INTRODUCTION

A single material will not possess different properties to meet the demand of today's industry, hence a composite system of base material providing necessary mechanical strength is resorted to. The process of corroding or materials being corroded and phenomena called corrosion is encountered. Some applications demand the materials to possess adequate corrosion resistance properties. To improve the performance of the components in such applications a composite made out of base material and a protective surface layer different in structure and/or chemical composition can be thought of as a substitute in combining material properties. Coatings play a major role in the prevention of corrosion of materials. They form a scale deposition and protect the surface against corrosion. Chemical vapor deposition technique, Physical vapor deposition technique, Thermal spray technique are the main coating techniques [1]. Flame spraying with a powder, Plasma spraying, High Velocity Oxy Fuel spraying are gaining importance. Improvement in coating technology has paralleled coating material developments with the aim of providing protection to superalloy components which are subjected to high temperature, increased oxidation and corrosion rates in service environments as in the case of turbine blades, turbine section of aeroplane, marine, utility turbines and in power plant industries [2].

Degradation by high-temperature oxidation, hot corrosion and erosion are the main failure modes of components in the hot sections of gas turbines, boilers, industrial waste incinerators, metallurgical furnaces, petrochemical installations, etc. Superalloys have been developed for high-temperature applications, but they are not able to meet the requirements of both the high-temperature strength and the high-temperature erosion-corrosion resistance simultaneously [3,4]. One possible way to overcome these problems is the use of thin high-temperature erosion-corrosion- and wear-resistant coatings. An ever-increasing demand on high performance of superalloys makes the surface coatings more favorable than ever before as they offer cost-effective ways to combat degradation resulting from mechanisms such as wear, oxidation, corrosion, or failure under an excessive head load without sacrificing the bulk properties of the component materials. Many components operating at high temperature are coated or surface treated to achieve the required properties [5,6].

Hard chromium coatings, with relatively high corrosion resistance, have been widely used in the engineering industry to protect the components whose surfaces are subjected to corrosion conditions such as valves, propellers, piston rods and hydraulic machine parts in the past decades [7]. Nevertheless, industrial applications of this coating have been limited by the presence of carcinogenic and environmentally problematic hexavalent chromium in the plating bath. To avoid these problems, great attention has been paid to the thermally sprayed coatings, such as WC-based cermet coatings. The reason is that, with their good corrosion resistance and high hardness with satisfactory toughness, WC-based cermet coatings seem to be an attractive alternative to hard chromium coating [8,9].

HVOF process has the advantage of being a continuous and most convenient process for applying coatings to industrial installations at site. Several HVOF sprayed coatings have been subjected to corrosion testing in seawater, including cermet and anti-corrosion alloy [10]. These studies concluded that the HVOF method produced coatings with higher corrosion resistance when compared with other spraying technologies such as flame spraying, arc spraying, and plasma spraying. Therefore, in this study, the HVOF process is used to deposit the coatings [11,12].

Hot corrosion is the accelerated oxidation when the surfaces covered with a thin film of few salts are exposed to elevated temperature conditions. This form of corrosion is commonly seen in gas turbines, boilers, internal combustion engines etc. To minimize such corrosion, coating materials have been developed and different coating techniques have also been investigated [12,13]. In this

investigation, an attempt has been made to study the hot corrosion behaviour of the high velocity oxy fuel sprayed coating on boiler tube steel. The feed stock powders namely WCNi+NiCr has been sprayed on the boiler tubes and thermogravimetric studies have been carried out to determine corrosion behavior of the HVOF sprayed sample[14,15].

## 2. EXPERIMENTAL DETAILS

### 2.0 Substrate materials

The substrate boiler tube steel for the study was procured from M/S Mishra Dhatu Nigam Limited (MIDHANI), Hyderabad. The chrome moly steel (designated as ASTM-SA213-T22) which is being used as material for water wall, super heater and reheater tubes in coal fired thermal power plants in northern part of India has been used as a substrate material in the present study. The nominal composition of the boiler tube steels is given in Table 2.1.

Sl. No.	Alloy Grade (ASTM code)	Chemical Composition (wt. %)								
		Fe	Ni	Cr	Ti	Al	Mo	Mn	Si	C
1	SA213-T22	Bal.	-	2.5	-	-	1.1	0.5	0.4	0.1

Table 2.1 Chemical composition (Wt %) for substrate alloy.

Sl No	Material	Designation	Dimensions (L B×T) (mm <sup>3</sup> )
1	Chrome moly steel (Substrate)	ASTM-SA213-T22	25×25×4.85
2	CNi+NiCr Coated sample	ASTM-SA213-T22	15×20×5.47

Table 2.2 Dimensional details of specimens

The dimensional details of substrate and the coated material is given below in Table 2.2. These specimen were cut from the tubes for corrosion studies.

### 2.1 Coating materials

Two types of commercially available feedstock materials has been used in the powder form. This has been used to as a spray coating material on three different types of substrate materials using High velocity oxy fuel coatings (HVOF). The details of the chemical composition and particle size of powder is reported in Table 2.3.

Sl No	Coating powder	Chemical Composition (Wt %)	Particle size
1	WCNi+NiCr	WCNi(65%),NiCr(35%)	-45 + 15 μm

Table 2.3 Chemical composition and particle size of coating powders used.

### 2.2 Deposition of the coatings

HVOF spraying technique has been carried out using a HIPOJET 2700 equipment (M/S Spraymet Coating Industry, Peenya, Bangalore, India) has been used in the present investigation. This equipment utilizes supersonic jet generated by the combustion of liquid petroleum gas and oxygen mixture. The spraying parameters selected during the HVOF deposition are listed in Table 2.4. The process parameters namely spray distance and all other process parameters were kept constant throughout coating process. The substrate materials were grit-blasted using Al<sub>2</sub>O<sub>3</sub> (Grit 45) before the spray coating to develop better adhesion between the substrate and the coating.

HVOF process parameter	Quantity
Oxygen flow rate	250 l/min
Fuel (LPG) flow rate	65-70 l/min
Air-flow rate	550 l/min
Spray distance	178 mm
Powder feed rate WCNi(35%)+NiCr(65%)	28 g/min
Fuel(LPG) pressure	681 kPa
Oxygen pressure	981 kPa
Air pressure	588 kPa

Table 2.4 Spray parameters employed for HVOF spray process

### 2.3 Experimental setup and procedure details

Hot corrosion studies were conducted using silicon carbide tube furnace (Make Digitech, India). The studies were carried out at a temperature of 700°C. Figure 2.1 shows the hot corrosion study experiment setup which has been used in the present investigation.



Figure 2.1 Hot corrosion Experimental setup

### 2.4 PROCEDURE

- Furnace was calibrated Platinum/Platinum-13% Rhodium thermocouple (fitted with a temperature indicator of Electromek Model- 1551P, India) to an accuracy of  $\pm 5^\circ\text{C}$ .
- The coated specimens and uncoated specimens were polished using polishing machining ( $1\mu\text{m}$  accuracy).
- Dimension of the specimens were noted down using digital vernier caliper
- The specimens were thoroughly cleaned, washed with acetone and dried in hot air to remove moisture.
- The specimens were then heated in an oven up to  $250^\circ\text{C}$ , which helps in uniform application of salt mixture.
- Salt mixture sample containing  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  dissolved in distilled water was coated on the warm polished specimen using a brush (thickness between  $3.0$  -  $5.0$   $\text{mg}/\text{cm}^2$ ).
- The Alumina boats and the salt coated specimen were dried in the oven at  $150^\circ\text{C}$  for 30 minute duration-weighed precisely.
- These specimens kept in aluminum boat were preheated to maintain the weight constant for high temperature cyclic corrosion studies (preheated at constant temperature of  $1200^\circ\text{C}$  for 10hr duration).
- The boat containing the specimen was introduced into hot zone in furnace whose temperature was set at  $700^\circ\text{C}$ .
- Holding time inside the furnace was maintained for 1 hour duration and then afterwards the boat with the specimen was taken out and cooled to room temperature in still air.
- At the end of each cycle, Visual observations were made to study the color change, the luster, formation of oxide scale...etc.
- The above study (cyclic hot corrosion studies) was carried out for 50 cycles.

### 3 RESULTS AND DISCUSSION

#### 3.1 Microstructure of substrate steels

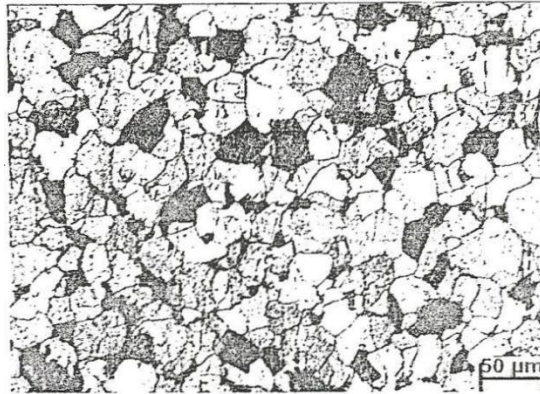


Fig 3.1 Optical micrograph of substrate T22 steels at 500X magnification

The optical microstructures of the substrate steels are shown in Fig 3.1. The microstructure of T22 steel consist of polygonal ferrite (white constituent and bainite (dark constituent)).

#### 3.2 Morphology of coating powder

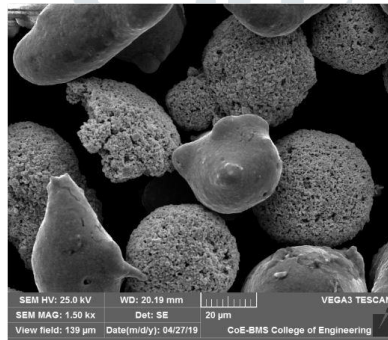


Fig 3.2 Scanning electron micrograph of coating powder WCNi+NiCr

The morphology of coating powders has been evaluated using the scanning electron microscopy which is shown in Fig 3.2. It is found from this figure that the WC-Ni+NiCr have irregular shapes. The particle size distribution of powder as determined by the image analysis of the secondary electron micrographs are found to be consistent with nominal size distribution as provided by the manufacturer. The micrograph of sample before hot corrosion is shown in Fig. 3.3.



Fig 3.3 Macrograph of coated sample WCNi+NiCr

### 3.3 Uncoated steels

#### 3.3.1 Thermo gravimetric studies

The macrographs of T22 steel subjected to hot corrosion in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  molten salt for 50 cycles at  $700^\circ\text{C}$  is shown in Fig.3.4. The color of the as- sprayed coating was grey which turned into black during the first cycle of exposure to salt environment. It can be observed that there is intense spalling of oxide scale for the T22 steel and after eighth cycle, the scale starts separating from the substrate. For the T22 materials during 37<sup>th</sup> cycle brown powder appears on the surface.

The plots of cumulative weight gain ( $\text{mg}/\text{cm}^2$ ) as a function of time expressed in number of cycles are shown in Fig 3.5. The weight gain for the T22 at the end of 50 cycles is found to be 24.37. Evidently, The T22 steel showed a maximum weight gain during the hot corrosion studies in molten salt environment. Further the weight gain square ( $\text{mg}^2/\text{cm}^4$ ) data is plotted as a function of time as shown in Fig 3.6. The plot shows that the material T22 follows parabolic behaviour. The parabolic rate constants,  $K_p$  ( $\text{g}^2 \text{cm}^{-4} \text{S}^{-1}$ ) for T22 is  $0.3313 \times 10^{-8}$ .



(a)

Fig 3.4 Macrograph of uncoated steel subjected to hot corrosion in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  at  $700^\circ\text{C}$  for 50 cycles. (a) T22 steel

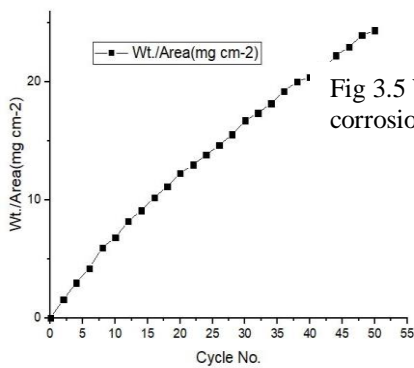


Fig 3.5 Weight change/area versus number of cycles plot for uncoated T22 steel subjected to hot corrosion

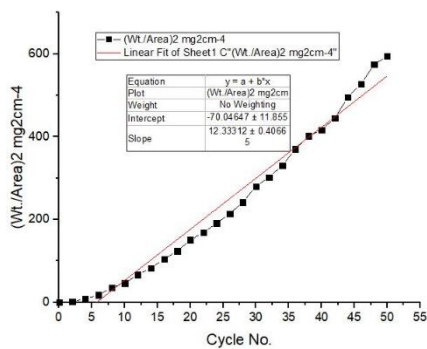


Fig 3.6  $(\text{Weight change/area})^2$  versus number of cycles plot for uncoated T22 steel subjected to hot corrosion

### 3.3.2 SEM/EDX analysis

The SEM micrograph showing the scale morphology along with the EDAX analysis is illustrated in Fig.3.7. Iron oxide was the main constituent of the outer layer of all the steels along with a small amount of  $\text{Cr}_2\text{O}_3$ . The surface scale in the case of T22 steels show intergranular cracks and spalled regions are clearly visible on the surface scale. The presence of oxides of Fe, C, V, O, Mo, Cr and Si in the surface oxide layers reveal that these elements have diffused from the substrate to the uppermost part of the scale during hot corrosion of the specimen.

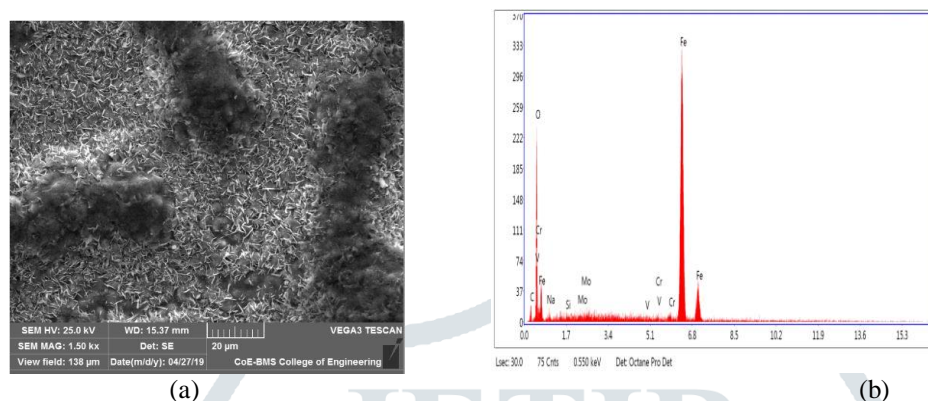


Fig. 3.7 (a) Surface scale morphology and (b) EDAX point analysis for uncoated T22 steel subjected to hot corrosion for 50 cycles in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  environment at  $700^\circ\text{C}$ .

### 3.3.3 Uncoated steel discussion

The results of thermogravimetry data demonstrates the accelerated kinetics induced due to  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  eutectic mixture. Thick oxide scale formed on T22 steel mainly consists of iron oxide. T22 steel shows a higher corrosion rate and intense spalling of oxide scale.

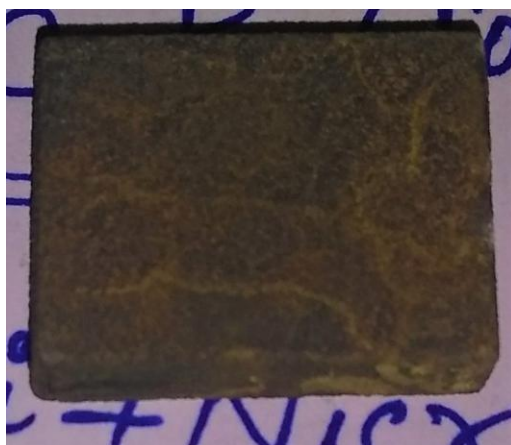
The uncoated T22 steel showed intense spalling, peeling of scale and enormous weight gain. The higher corrosion rate during initial hours of study, which might be attributed to the rapid oxygen pick up by diffusion of oxygen through the molten salt layer, is identical to the results reported by investigators during their hot corrosion studies. The stress developed due to higher volume of the oxide scale leads to cracking and severe spalling of oxide scale during hot corrosion of T22 steel in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  molten salt environment.

## 3.4 WCNi(65%)+NiCr(35%) coating

### 3.4.1 Thermo gravimetric studies

Macrographs of HVOF sprayed WCNi+NiCr coated steels subjected to hot corrosion in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  environment for 50 cycles at  $700^\circ\text{C}$  are shown in Fig 3.8. The color of the as-sprayed coating was grey which turned into black during the first cycle of exposure to salt environment. Green colored oxide scale has been observed on the corroded surface of the coated steels. Contrast grey color was observed on corroded surface of the coated steel. In case of T22 steel during 5<sup>th</sup> cycle onwards slight brown color has been observed and 7<sup>th</sup> cycle onwards light green color was identified throughout the 50 cycle.

The plots of cumulative weight gain ( $\text{mg}/\text{cm}^2$ ) as a function of time are shown in Fig 3.9. It can be seen from the thermo gravimetric data that the necessary protection against hot corrosion has been provided by the WCNi+NiCr coating, as the weight gain values for the coated steel are smaller than respective uncoated steel. The total weight gain value for the coated T22 specimen at the end of 50 cycles of hot corrosion studies are found to be  $1.8936 \text{ mg}/\text{cm}^2$ . Further the weight gain square ( $\text{mg}^2/\text{cm}^4$ ) data were plotted as a function of time shown in the Fig 3.10. The T22 steel followed parabolic behaviour and the parabolic rate constants  $k_p$  calculated is  $0.0015 \times 10^{-8} \text{ g}^2 \text{ cm}^{-4} \text{ S}^{-1}$ .



(a)

Fig 3.8 Macrograph of WCNi+NiCr coated steel subjected to hot corrosion in Na<sub>2</sub>SO<sub>4</sub>- 60% V<sub>2</sub>O<sub>5</sub> environment at 700°C for 50 cycles.  
(a)T22 steel

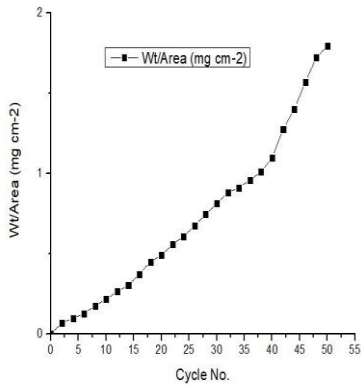


Fig 3.9 Weight change/area versus number of cycles plot for WCNi+NiCr coated steel subjected to hot corrosion

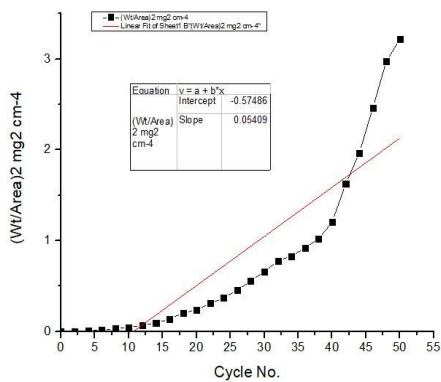


Fig 3.10 (Weight change/area)<sup>2</sup> versus number of cycles plot for WCNi+NiCr coated steel subjected to hot corrosion

### 3.4.2 X-ray Diffraction Analysis

The X-ray diffraction patterns for WCNi+NiCr coated specimens after exposure to molten salt environment for 50 cycles are shown in Fig 3.11. The XRD result reveals presence of Cr<sub>23</sub>C<sub>6</sub>, NiO<sub>2</sub> and Cr<sub>2</sub>O<sub>3</sub> as major phases along with minor phases of Ni<sub>3</sub>V<sub>2</sub>O<sub>8</sub> and Fe.

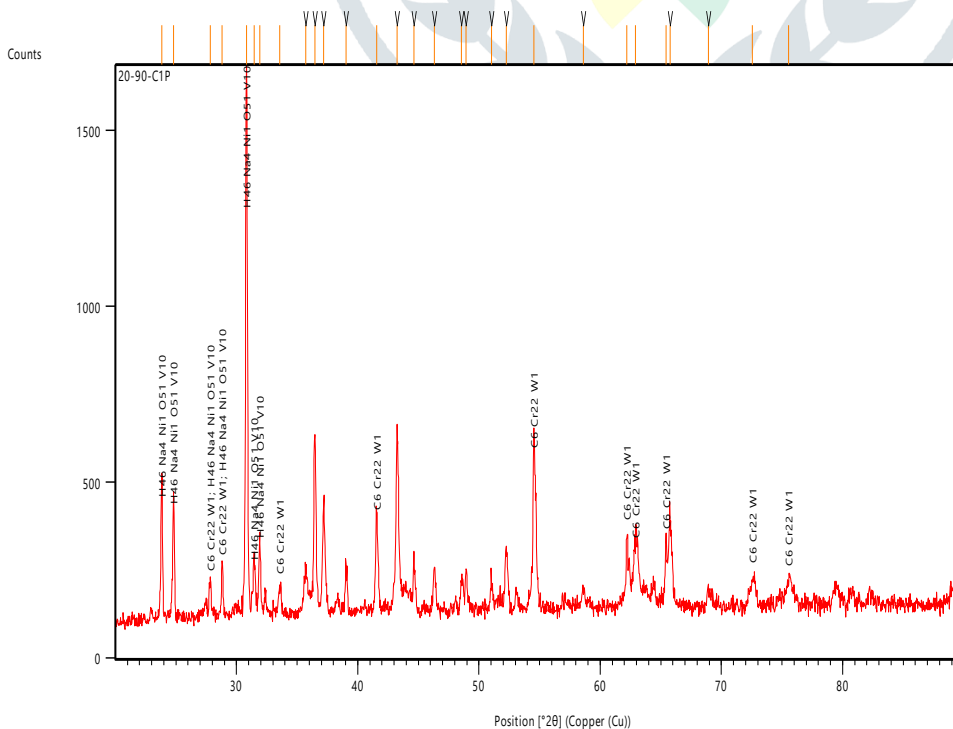


Fig 3.11 X-ray diffraction for WCNi+NiCr coated T22 steel subjected to hot corrosion for 50 cycles in Na<sub>2</sub>SO<sub>4</sub>-60% V<sub>2</sub>O<sub>5</sub> environment at 700°C

### 3.4.3 SEM/EDX analysis

The surface morphology of the corroded WCNi+NiCr coated T22 steel shows the formation of the loosely held microstructure of hot corroded coatings exemplifies the formation of porous bulky oxide scale. The surface morphology shows the protrusions rich in oxides of tungsten and nickel oxides. Fig. 3.12 also corroborates about the formation of non-uniform loosely held oxide scale with voids. The EDAX analysis on the surface oxide scale shows oxides of W (12.78%), Ni (27.81%), C(4.97%), O(21.62%), Na(20.07%) and Cr(3.55%). Minor amount of oxides of vanadium and sulphur have also been detected.

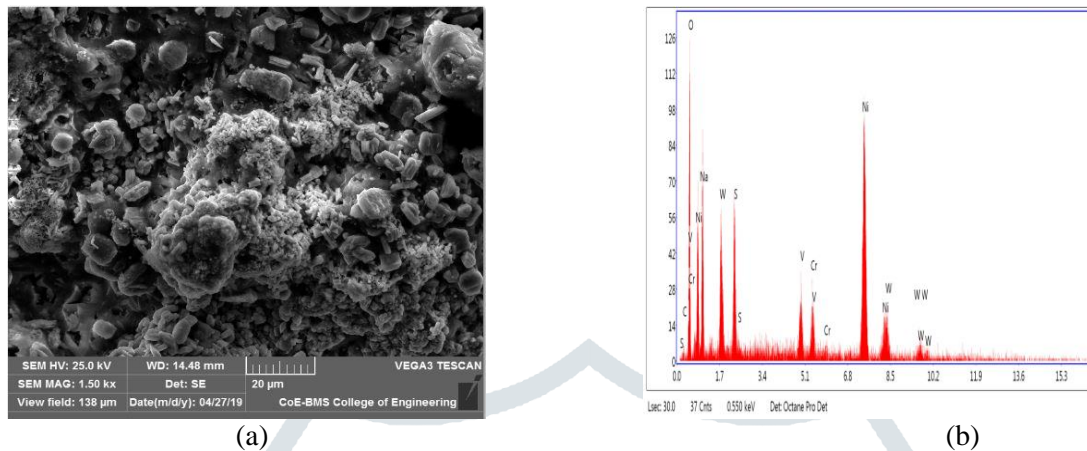


Fig. 3.12 Surface scale morphology(a) and EDAX point analysis(b) for WCNi+NiCr coated T22 steel subjected to hot corrosion for 50 cycles in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  at  $700^\circ\text{C}$

### 3.4.4 WCNi+NiCr coating discussion

The WCNi+NiCr coated specimen shows lower weight gain in comparison to the uncoated specimen, when exposed to  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  molten salt environment. The presence of minor phase such as  $\text{Fe}_2\text{O}_3$  on the surface of hot corroded WCNi+NiCr indicates the diffusion of Fe from the substrate during hot corrosion of the specimens at temperature about  $700^\circ\text{C}$ . The formation of  $\text{Fe}_2\text{O}_3$  in the spalled scale has also been reported to be non-protective by investigator.

The initial high oxidation rate of the coated specimen might be ascribed to the rapid formation of oxides at the splat boundaries and within open pores due to the penetration of the oxidizing species. During hot corrosion, initially the corroding species reacts with the top surface of the coating and starts migrating through the inter splat interface and diffusion of elements of substrate for example iron moves upward along this inter splat space at the coating substrate interface, as the oxidation proceeds elements basically chromium get oxidised and forms a continuous  $\text{Fe}_2\text{O}_3$  layer below the top oxide layer. The continuous band of  $\text{Fe}_2\text{O}_3$  in the subscale and  $\text{Fe}_2\text{O}_3$  along the splat boundaries will not allow any further transport of the oxidizing species and the metallic ions. The presence of these elements at the coating surface will decrease oxygen availability in the underlying alloy and favors the formation of most thermodynamically stable oxide, i.e.  $\text{Fe}_2\text{O}_3$ . With the passage of time coating get partially oxidised along the splat boundaries with this further oxidation become negligible. This partially oxidised coating provides protection to the substrate.

$\text{Cr}_2\text{O}_3$  and  $\text{NiO}_2$  nuclei at the coating surface react to form  $\text{NiCr}_2\text{O}_4$  spinel, as it is evident from XRD analysis. So the reaction is confined mainly to the top of the coating. Some minor spalling of the oxide scale of coated specimens especially on the edges and corners during cooling periods of the thermal cycles may be due to different values of thermal expansion coefficients of the coatings, substrate and oxides. Initial spallation and sputtering might be due to the different values of thermal expansion coefficients of the coatings, substrate and oxides.

The NiCr coating has provided the best protection to the substrate steel, which may be due to the formation of  $\text{NiO}_2$ ,  $\text{NiCr}_2\text{O}_4$  and  $\text{Cr}_2\text{O}_3$  as confirmed by XRD analysis, which are reported to be the protective oxides by researchers.

## 4. CONCLUSIONS

High velocity oxy-fuel thermal spraying with liquid petroleum gas as the fuel gas has been used successfully used to deposit WC-Ni+NiCr alloy coatings on boiler tube materials.

Under the given spray parameters, seemingly dense laminar structured coating with thickness in the desired range of 130 to 150 µm and porosity less than 3.45% has been achieved.

Uncoated specimen suffered a higher corrosion rate and intense spalling of oxide scale was observed. The main constituent of oxide scale formed on specimen is iron oxide. The acidic fluxing of the oxides by the molten salt mixture resulted in massive, porous oxide scale.

The cumulative weight gain for all the HVOF coated T22 boiler materials are significantly lower than that of uncoated specimen subjected to hot corrosion in  $\text{Na}_2\text{SO}_4$ -60%  $\text{V}_2\text{O}_5$  molten salt environment for 50 cycles at  $700^\circ\text{C}$ . All the coated specimen exhibit characteristic thick protective oxide scale, composed of oxides and spinel oxide of the active elements of the coating and imparted resistance to the hot corrosion in the given salt environment.



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