

# Effect of TiO<sub>2</sub> on Plasma Sprayed Al<sub>2</sub>O<sub>3</sub> Based Composite Coatings at 900°C in Molten Salt Environment

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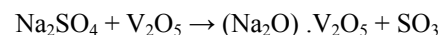
**Abstract** The present work investigates the effect of hot corrosion on ASTM-SA213-T-22 Steel with different coatings of Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> at 900°C in molten salt environment (40wt%Na<sub>2</sub>SO<sub>4</sub>+60wt%V<sub>2</sub>O<sub>5</sub>). The experimentation consisted of 50 cycles of heating coated and uncoated specimens for 1 hour at 900°C in muffle furnace and cooling for 20 minutes at ambient temperature. The corroded specimens were analyzed using visual examination, weight change measurement, X-ray diffraction technique and Scanning electron microscopy/Energy-disperse X-ray analysis. The results showed that the uncoated substrate had been more affected with corrosion and gained more mass due to formation of iron oxide (Fe<sub>2</sub>O<sub>3</sub>) as compared to coated substrate because of better adhesion of Al<sub>2</sub>O<sub>3</sub> coating with base metal SA213-T22 due to presence of TiO<sub>2</sub>. The presence of TiO<sub>2</sub> increased the strength and durability of Al<sub>2</sub>O<sub>3</sub> coating to withstand with high temperatures.

**Keywords** Hot Corrosion, Boiler Steel, Composites, Thermal Spray, High Temperature

## 1. Introduction

The corrosion occurs at elevated temperatures due to exposure of steels and alloys in atmosphere, which causes formation of oxide layer, when the environmental oxygen reacts with ferrous metal, and is called hot corrosion [1-4]. The failure occurs in boiler tubes is due to the corrosion of base metal at elevated temperature in the molten salt environment which results in damage to the physical and chemical properties of the substrate [5-7]. This can be prevented by applying thin layer of coating on the surface of base metal by the various coating processes like plasma spray coating, high velocity oxy fuel (HVOF) and powder coating etc. As analyzed by literature survey, the alumina

based coatings provide higher corrosion and wear resistance to steel alloys up to 900°C [8, 9]. The rate of corrosion is very fast in metals and alloys; this results in the formation of porous non-protective oxide layer on the surface of material [7, 10-12]. The corrosion rate is highly dependent on the presence of corrosive elements, such as Na<sub>2</sub>SO<sub>4</sub> and V<sub>2</sub>O<sub>5</sub> in atmosphere with elevated temperature.



In this reaction, the sodium sulphate and vanadium pentoxide results in formation of Sodium oxide and sulfur tri-oxide which is known as corrosive agent [4, 13]. To coat the substrate, various coating process are used such as Plasma spray coating, High Velocity Oxy Fuel (HVOF) coating and D-gun spray coating, to decrease the rate of corrosion. Goya et al [14] all stated that all coatings are porous, due to the porosity at microscopic level in coating, allows oxygen to react with the base metal through pores and cause formation of oxide layer under the coating region in molten salt environment. Due to oxide layer formation between the coating and base metal boundaries spallation takes place which results in erosion in between the coating and the surface of substrate [15-20]. Hot corrosion is of two types; high temperature hot corrosion (HTHC) – above 750°C and low temperature hot corrosion (LTHC) – 550-750°C. The high rate corrosion occurs with the reaction of sulfur, sodium, potassium, lead and vanadium, usually having low melting points. The rate of corrosion is highly dependent on partial pressure of oxygen present in atmosphere [21-24]. Various coatings are used to prevent the rate of corrosion, as per analysis, the Al<sub>2</sub>O<sub>3</sub> is more protective in prevention of oxidation rate, formation of cracks and spallation caused by oxides of molybdenum after repetitive cycle at elevated temperature in molten salt environment. It is observed from literature study that various types of coatings are used on the substrates with different deposition method. To reduce this effect of

coating, bi-layered coating of Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> is used [8-10]. It is analyzed that the use of Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> coating shows better results. The bilayer of TiO<sub>2</sub> with Al<sub>2</sub>O<sub>3</sub> sprayed with plasma coating helps to reduce pores so that the electrolyte and other corrosive elements could not react with the base metal of substrate. It also increases the properties of substrate to withstand with high temperature, wear and corrosion. TiO<sub>2</sub> is used along with fabrication of Al<sub>2</sub>O<sub>3</sub> coating, TiO<sub>2</sub> helps in adhesion of coating with base metal [3]. It has been observed with analysis of various research papers that Al<sub>2</sub>O<sub>3</sub> enhance the properties of material by both physically or chemically to prevent the rate of corrosion at 900°C [17, 18]. The aim of this experiment is to slow down the rate of corrosion at elevated temperatures in molten salt environment by using the reinforced coating of Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> with different weight percentage composition, exposed to molten salt environment at 900°C. This experiment defines the behavior of hot corrosion on SA213-T22 boiler steels coated with Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>, with different weight-percent combinations, in silicon tube furnace at 900°C for 50 cycles consisting of one hour heating followed by 20 minutes of cooling in molten salt

(40wt%Na<sub>2</sub>SO<sub>4</sub>+60wt%V<sub>2</sub>O<sub>5</sub>). XRD, SEM/EDS is undertaken to obtain various results.

## 2. Experimental Procedure

### 2.1. Basic Composition of SA213-T22 Material

ASTM-SA213-T-22 Steel is used as base material, obtained from Guru Gobind Singh Thermal Power station, Rupnagar, Punjab. Detailed specifications are given in Table 1.

### 2.2. Preparation of Coatings

The coating was sprayed on steel specimens at Metallizing Equipment Corporation Private Ltd. Jodhpur, Rajasthan. The size of each specimen was 20 mm x 15 mm x 5 mm. Table 2 shows the composition of coatings obtained, and Table 3 shows the parameters employed in plasma spraying process.

**Table 1.** Composition of SA213-T22 steel boiler tubes

Type of steel	ASME code	Composition	C	Mn	Si	S	P	Cr	Mo	K	Fe
T-22	SA213 T-22	Actual	0.15	0.60	0.48	0.01	0.02	2.6	1.13	0.02	Bal.

**Table 2.** Composition of SA213-T22 material

Coating No.	Coating composition	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Particle size
1	Al <sub>2</sub> O <sub>3</sub>	100	0	40±10µm
2	Al <sub>2</sub> O <sub>3</sub> - 3 wt.% TiO <sub>2</sub>	97	3	40±10µm
3	Al <sub>2</sub> O <sub>3</sub> - 13 wt.% TiO <sub>2</sub>	87	13	40±10µm
4	Al <sub>2</sub> O <sub>3</sub> - 20 wt.% TiO <sub>2</sub>	80	20	40±10µm

**Table 3.** Parameters used for plasma spray coating

Parameters	Value
Plasma Current – A	600
Plasma Voltage - V	50
Carrier gas flow (g-min)	50
H <sub>2</sub> Gas flow pressure- psi	62
Argon Gas flow pressure - psi	50
Spray distance – mm	50
Spray passes	5

### 2.3. Formulation of Coating

#### Experimental studies are undertaken in molten salt environment (40wt%Na<sub>2</sub>SO<sub>4</sub>+60wt% V<sub>2</sub>O<sub>5</sub>).

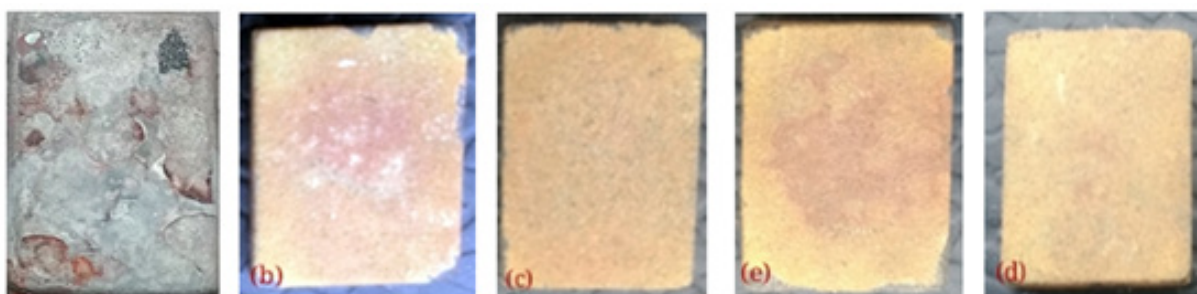
The behaviour of TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> coatings on T-22 boiler steel at 900°C in the molten salt environment (40wt% Na<sub>2</sub>SO<sub>4</sub>-60wt% V<sub>2</sub>O<sub>5</sub>) was examined. The experiment was consisted of 50 cycles. Each cycle consisted of 1 hour heating in a silicon tube muffle furnace followed by 20 minutes of cooling in room temperature. Before conducting the experiment, the physical dimensions of each specimen was measured with the help of vernier caliper. The de-burring operation was performed on each surface to remove any foreign particles at the microscopic level. The plasma spray coating was performed before the application of molten salt on the surface of specimens. The (40wt% Na<sub>2</sub>SO<sub>4</sub>-60wt% V<sub>2</sub>O<sub>5</sub>) salt was mixed with distilled water and was applied on each face of the specimen after washing all specimens with acetone. The layer of molten salt was applied gently on each face of specimen with the help of camel hair brush. To make the proper adhesion of molten salt on each surface of the specimen, all specimens were heated up to 250°C in the oven. The molten salt environment becomes more aggressive to react with metals with the increase in temperature and the rate of reaction also increases. The increase in temperature causes the faster rate of corrosion on the surface of the metal. The samples were kept in alumina boats in a muffle furnace at 900°C with 50 repetitive cycles of 1-hour heating followed

by 20 minutes of cooling at room temperature. The aggressiveness of corrosion was examined with a number of repetitions in cycles. After the experimentation, all the specimens were analyzed by the X-ray diffraction, SEM and EDS. The experiment was performed in metallurgy laboratory of Chandigarh Engineering College, Landran (Mohali), Punjab.

## 3. Results

### 3.1. Visual Examination

Before conducting the experiment, all samples were in physically perfect condition. The experiment was conducted in a molten salt environment (40wt% Na<sub>2</sub>SO<sub>4</sub>-60wt% V<sub>2</sub>O<sub>5</sub>) kept in silicon tube muffle furnace at 900°C. During the experiment, after the 4<sup>th</sup> cycle, the uncoated T-22 specimen started to change in brownish colour and after 15<sup>th</sup> cycles formation of cracks were started to develop near corners of the specimen as shown in (Fig. 1(a)). In the case of Al<sub>2</sub>O<sub>3</sub> coated T-22 specimen, cracks were started to appear on the coated surface after 30<sup>th</sup> cycle. Minor spallation of scale was seen on the surface after 50<sup>th</sup> cycle as shown in (Fig. 1(b)). But in the case of other specimens coated with TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> composite coatings, no change in colour was observed and till 30<sup>th</sup> cycle, there was no development of cracks found even after 50 cycles as shown in images (Fig. 1(c-e)).



**Figure 1.** Visual inspected images of (a) uncoated, (b) Al<sub>2</sub>O<sub>3</sub> (c) Al<sub>2</sub>O<sub>3</sub> - 3 wt.% TiO<sub>2</sub>, (d) Al<sub>2</sub>O<sub>3</sub> - 13 wt.% TiO<sub>2</sub>, and (e) Al<sub>2</sub>O<sub>3</sub> - 20 wt.% TiO<sub>2</sub> coated substrates

### 3.2. Mass Change Observations

The thermo gravimetric study was done to find the mass change characteristics showed by coated and uncoated T-22 boiler steel. The change in mass of uncoated and coated T-22 steels were analyzed after exposing each specimen in the molten salt environment (40wt%Na<sub>2</sub>SO<sub>4</sub>-60wt%V<sub>2</sub>O<sub>5</sub>) for 50 cycles at 900°C. The change in mass showed the kinetics of corrosion. The high mass gain is identified as a high rate of corrosion. The graph shows that uncoated specimen showed the higher

mass gain, the specimen coated with Al<sub>2</sub>O<sub>3</sub> shows a little less mass gain than uncoated specimen.

The mass change is observed after each complete cycle shown in. (Fig 2)

### 3.3. XRD Analysis

X-Ray diffraction tests are conducted to figure out the nature of metallurgy of material affected due to high temperature and corrosive environment.

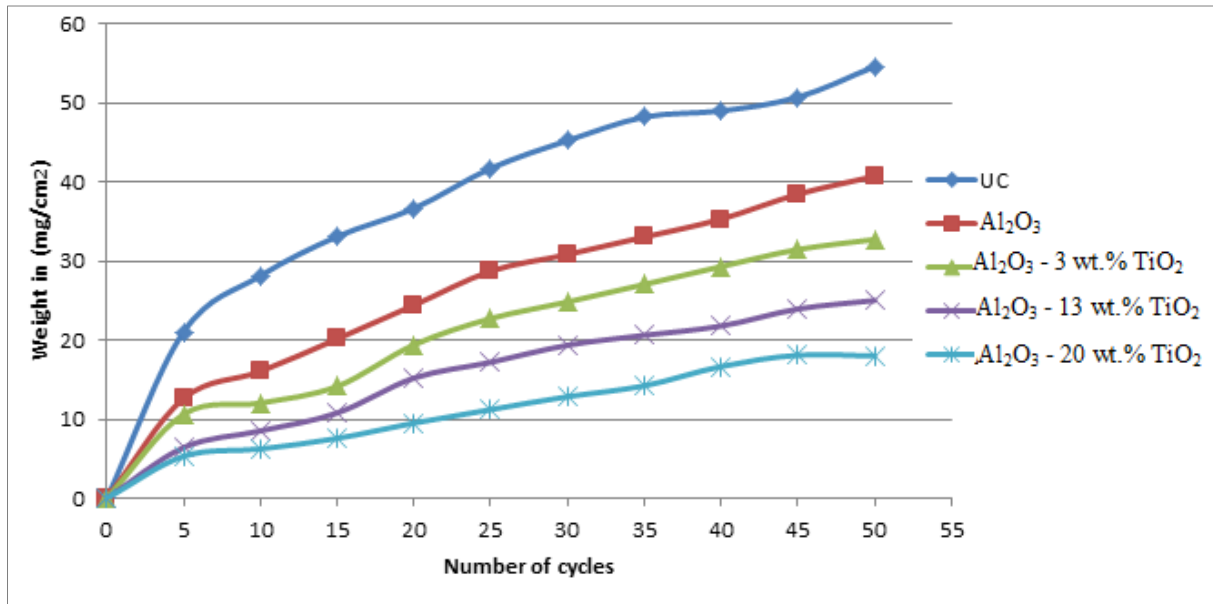
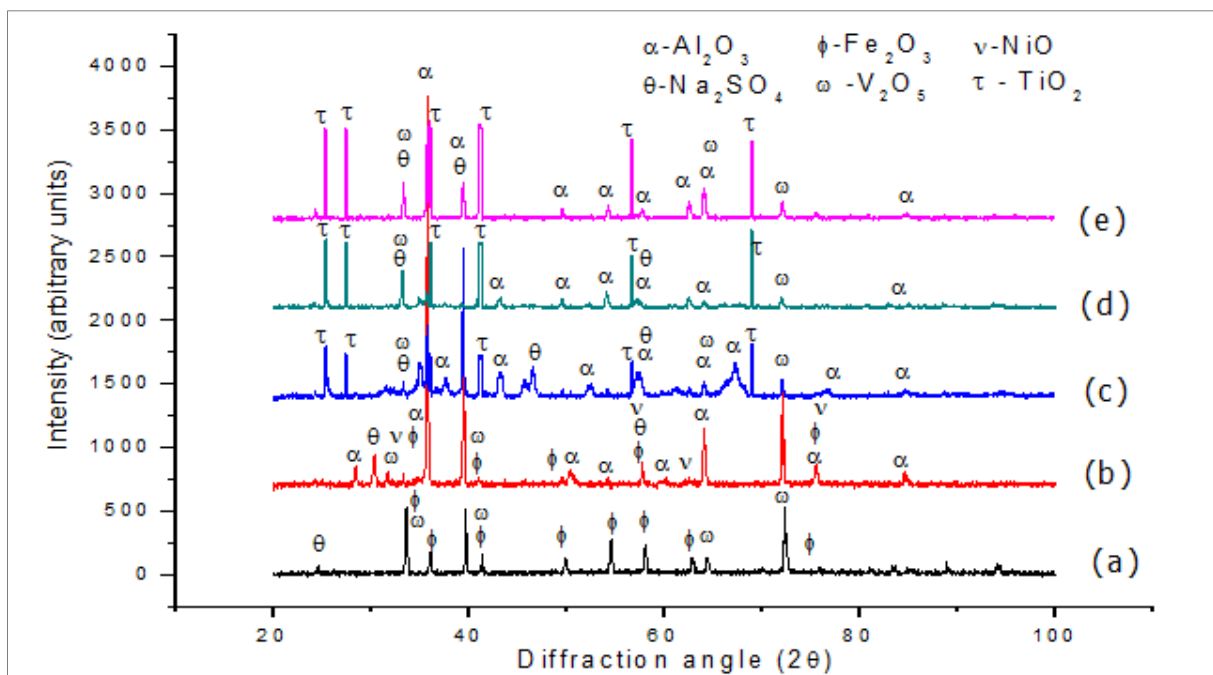


Figure 2. Weight gain in (mg/cm<sup>2</sup>) for uncoated and coated substrates



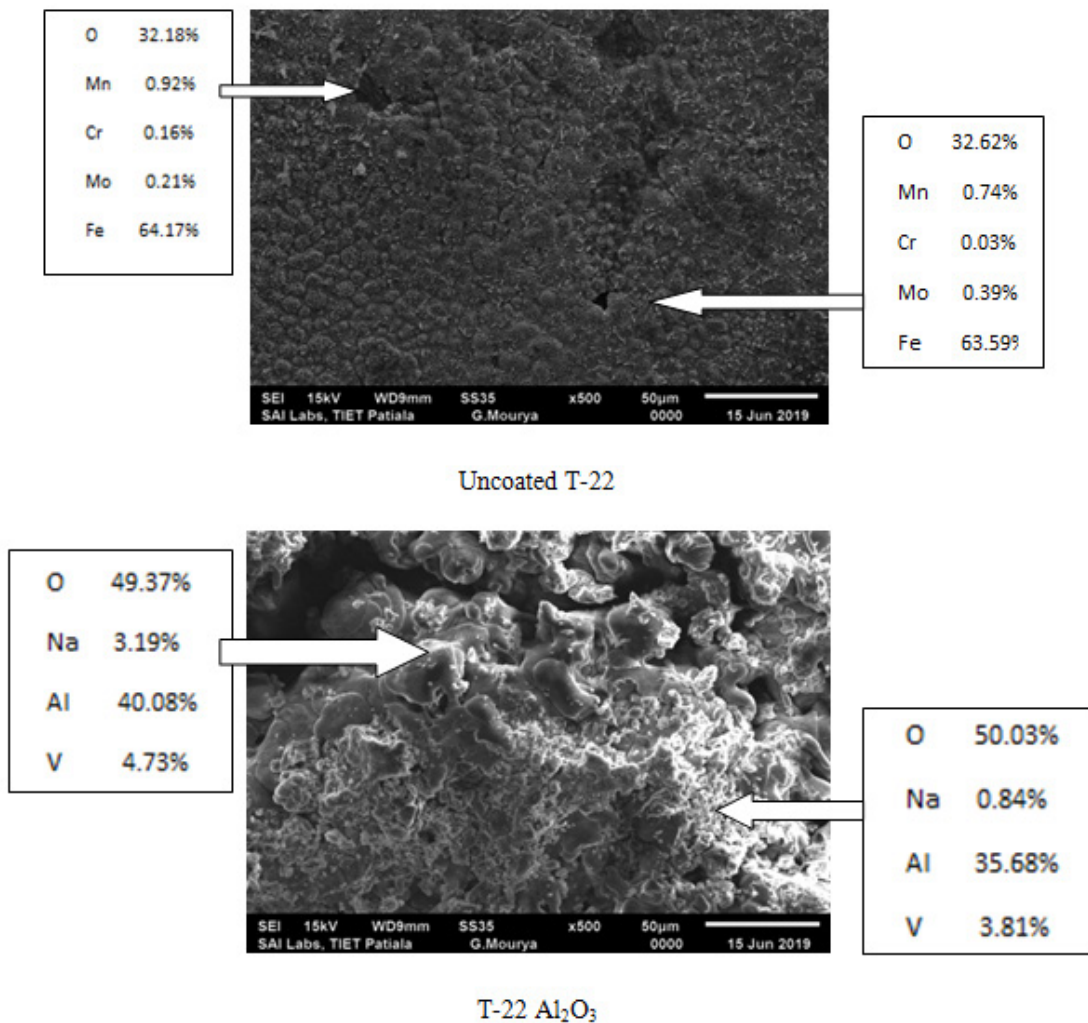
**Figure 3.** Xrd spectrum of (a) uncoated, (b) Al<sub>2</sub>O<sub>3</sub> (c) Al<sub>2</sub>O<sub>3</sub> - 3 wt.% TiO<sub>2</sub>, (d) Al<sub>2</sub>O<sub>3</sub> - 13 wt.% TiO<sub>2</sub>, and (e) Al<sub>2</sub>O<sub>3</sub> - 20 wt.% TiO<sub>2</sub> coated substrates

The XRD graph shows that, the uncoated T-22 Specimen shows the formation of Fe<sub>2</sub>O<sub>3</sub> at oxide scales. The specimen with the coating of pure Al<sub>2</sub>O<sub>3</sub> shows the Al<sub>2</sub>O<sub>3</sub> peaks along with Na<sub>2</sub>SO<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub>. The specimen containing Al<sub>2</sub>O<sub>3</sub>-3TiO<sub>2</sub> shows formation of TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>SO<sub>4</sub>, and no presence of Fe<sub>2</sub>O<sub>3</sub> is found on the surface of specimen. The specimen coated with Al<sub>2</sub>O<sub>3</sub>-13TiO<sub>2</sub> shows the peak of TiO<sub>2</sub>, Na<sub>2</sub>SO<sub>4</sub> and no presence of Fe<sub>2</sub>O<sub>3</sub> is found on the surface and specimen coated with Al<sub>2</sub>O<sub>3</sub>-20TiO<sub>2</sub> shows presence of TiO<sub>2</sub>, Na<sub>2</sub>SO<sub>4</sub> and Al<sub>2</sub>O<sub>3</sub> along with of V<sub>2</sub>O<sub>5</sub> and no peak of Fe<sub>2</sub>O<sub>3</sub> is found on coated surface of specimen.

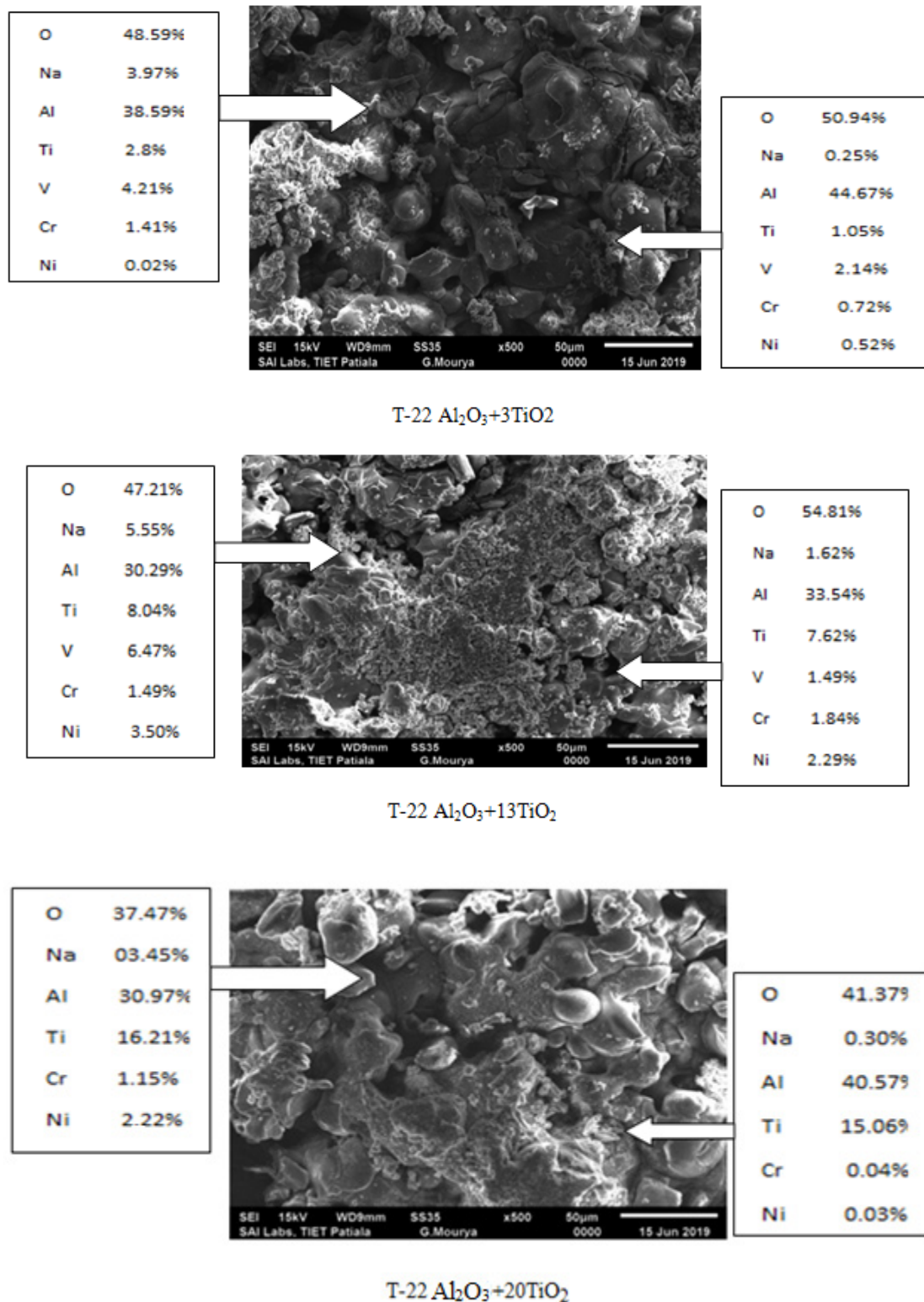
The XRD graph clearly shows that the uncoated specimen mostly affected by the formation of Fe<sub>2</sub>O<sub>3</sub> and nearly no formation of Fe<sub>2</sub>O<sub>3</sub> found on the surface of the specimen coated with TiO<sub>2</sub>- Al<sub>2</sub>O<sub>3</sub>.

### 3.4. SEM/EDS Analysis

Figure 4 shows the SEM micrograph of T-22 uncoated steel substrate after exposing it in molten salt environment for 50 cycles of heating inside a silicon tube muffle furnace. The EDS analysis on spectrum indicates the presence of Fe and Cr which might be the reason of formation of Fe<sub>2</sub>O<sub>3</sub> and Cr<sub>2</sub>O<sub>3</sub>. SEM Micrograph shows the spallation in the coating layer. The Fe and O found along with small percentage of Cr. For Al<sub>2</sub>O<sub>3</sub> coated T-22 specimen the EDS analysis spectrum shows the presence of Al, O, Cr and Fe. The presence of Al and Fe might cause the formation of Al<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. In case of coated specimens 3Wt% TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>, 13Wt% TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> and 20Wt% TiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> respectively shows the morphology of surface coating of specimens and the EDS analysis shows the major formation of Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>. The TiO<sub>2</sub> was well spotted with bright particle in darker region of Al<sub>2</sub>O<sub>3</sub>. The presence of Na and V might be due to the presence of molten salt elements in the oxide scale.







**Figure 4.** SEM/EDAX results of different corroded specimens

The SEM/EDS result showed that the coated specimens formed preventive elements such as Al, Cr and Ti on their surface, but in uncoated specimen the formation of Fe and Mo was found. The presence of Fe and Mo on the surface of uncoated specimen, might have caused the formation of

iron oxide layer on the surface, and led to maximum mass gain. The corrosion occurred at very early stages of the cycles in molten salt the environment at 900°C. On the other hand, the coated specimen showed absence of Fe and molybdenum.

## 4. Discussion

In this experiment, the hot corrosion behaviour of different reinforced coatings of  $\text{TiO}_2\text{-Al}_2\text{O}_3$  on T-22 boiler steel was examined. Each specimen was kept in molten salt environment (40wt%  $\text{Na}_2\text{SO}_4\text{-60wt% V}_2\text{O}_5$ ). After the successful completion of experiment the result were obtained.

The macrograph showed the rate of corrosion was high in uncoated T-22 specimen as compared to coated specimens. The change in colour to brownish was found in early stage in uncoated T-22 specimen. After 25<sup>th</sup> cycle the development of cracks were observed on the corner of uncoated specimen in visual observations. The thermo gravimetric study revealed that the uncoated specimen gained maximum mass in the experiment as compared to other specimens. The change in mass showed the kinetics of corrosion in the molten salt environment at elevated temperature. The thermo gravimetric graph (Fig. 2) showed that mass gained by the uncoated specimen was  $54.61\text{mg/cm}^2$ . The XRD (Fig. 3) graph indicated the formation of  $\text{Fe}_2\text{O}_3$  in oxide scale on the surface of uncoated specimen. The SEM/EDS analysis indicated the presence of Fe, Mo and V on the surface of uncoated T-22 specimen (Fig. 4). These elements are highly responsible for the hot corrosion [2]. The molybdenum oxide is found in molten state at  $550^\circ\text{C}$ . At  $900^\circ\text{C}$  the liquid molybdenum oxide dissolves the protective layer of chromium, which causes corrosion on the surface of substrate. The protective oxide layer of chromium helps to resist the corrosive species to react with the surface of metal [9]. The iron oxide reacts with the base metal with time, due to this the mass of substrate increases with respect to time. The thick iron oxide layer forms on the surface of specimens which is responsible for corrosion [13].

$\text{Al}_2\text{O}_3$  coated specimen showed 25.41% less mass gain as compared to uncoated specimen. Small pores were noticed on the surface of the specimen after exposing it to the molten salt environment for 50 cycles. XRD graph indicated the presence of  $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  oxide layers. The less mass gained by the specimen is might be due to the formation of  $\text{Al}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$  which resist the corrosive element to attack the base metal. However, porosities in  $\text{Al}_2\text{O}_3$  coatings might have abled the corrosive elements to attack the base metal. SEM/EDS analysis also showed presence of Al, Cr and Fe which validated the XRD results. The spallation produced on the specimen was might be due to the formation of  $\text{Fe}_2\text{O}_3$  oxide layer.

After exposing the 3wt% $\text{TiO}_2\text{-Al}_2\text{O}_3$ , 13wt%  $\text{TiO}_2\text{-Al}_2\text{O}_3$  and 20wt% $\text{Ti-Al}_2\text{O}_3$  coated substrates in molten salt environment (40wt%  $\text{Na}_2\text{SO}_4\text{-60wt% V}_2\text{O}_5$ ) at  $900^\circ\text{C}$ . These coatings were found safe to hot corrosion. There was no colour change found till 30<sup>th</sup> cycle and no cracks were visualized in the coated specimen after completion of all 50 cycles in molten salt environment at elevated temperature. The specimen coated with

20 $\text{TiO}_2\text{-Al}_2\text{O}_3$  coating showed least mass gain. The mass gained by specimen coated with  $\text{Al}_2\text{O}_3\text{-3TiO}_2$  was 40.06% less than that of uncoated T-22 steel. The specimen coated with  $\text{Al}_2\text{O}_3\text{-13TiO}_2$  gained less mass by 54.18% and the specimen coated with  $\text{Al}_2\text{O}_3\text{-20TiO}_2$  showed least mass gain of  $18.01\text{ mg/cm}^2$  and gained 67.02% less mass as compared to uncoated specimen showed by thermo gravimetric method shown in. The XRD graph showed the presence of  $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$  and  $\text{TiO}_2$  oxides on the surface of coated specimens. These elements are corrosion resistant in nature. In SEM/EDS analysis, the coated specimen showed the presence of Ti, Al in major phase. There were no traces of Fe and Mo found on the surface of coated specimens even after 50 numbers of cycles in molten salt environment at elevated temperatures.

This reinforcement of  $\text{TiO}_2$  in  $\text{Al}_2\text{O}_3$  might have helped to reduce the porosity in the coating layer. It is observed that the composition of  $\text{Al}_2\text{O}_3\text{-20TiO}_2$  showed better results. It has been analyzed that by increase in percentage of  $\text{TiO}_2$  with  $\text{Al}_2\text{O}_3$  coating, the hot corrosion resistance increases. The  $\text{TiO}_2$  along with  $\text{Al}_2\text{O}_3$  showed better adhesion properties with base metal and found preventive from hot corrosion. The rate of hot corrosion resistance showed the following trend:

Uncoated T-22 steel <  $\text{Al}_2\text{O}_3$  < 3Wt%  $\text{TiO}_2\text{-Al}_2\text{O}_3$  < 13Wt%  $\text{TiO}_2\text{-Al}_2\text{O}_3$  < 20Wt% $\text{Ti-Al}_2\text{O}_3$

## 5. Conclusions

- Different coatings of 100 $\text{Al}_2\text{O}_3$ , 3 $\text{TiO}_2\text{-Al}_2\text{O}_3$ , 13 $\text{TiO}_2\text{-Al}_2\text{O}_3$  and 20 $\text{TiO}_2\text{-Al}_2\text{O}_3$  were deposited on SA213-T22 with plasma spray coating technique.
- The uncoated specimen showed maximum mass change where as the specimen coated with  $\text{TiO}_2\text{-Al}_2\text{O}_3$  showed least mass change.
- The XRD and SEM/EDS analysis indicates that uncoated steel specimen contained  $\text{Fe}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$  as major phase in oxide scale.
- In the case of coatings of 3 $\text{TiO}_2\text{-Al}_2\text{O}_3$ , 13 $\text{TiO}_2\text{-Al}_2\text{O}_3$  and 20 $\text{TiO}_2\text{-Al}_2\text{O}_3$ , the major phases of  $\text{TiO}_2$  and  $\text{Al}_2\text{O}_3$  were found in XRD, SEM/EDS analysis.
- The better resistance was provided by  $\text{Al}_2\text{O}_3\text{-20TiO}_2$  among all other coatings when reacted to molten salt environment (40wt%  $\text{Na}_2\text{SO}_4\text{-60wt% V}_2\text{O}_5$ ) for 50 cycles at  $900^\circ\text{C}$ .
- The 100 $\text{Al}_2\text{O}_3$ , 3 $\text{TiO}_2\text{-Al}_2\text{O}_3$ , 13 $\text{TiO}_2\text{-Al}_2\text{O}_3$  and 20 $\text{TiO}_2\text{-Al}_2\text{O}_3$  coated specimen indicated the mass gain reduction by 25.41%, 40.06%, 54.18% and 67.02 % respectively, as compared to uncoated specimen.
- The  $\text{TiO}_2\text{-Al}_2\text{O}_3$  specimens showed no formation of cracks after experimentation done in molten salt environment (40wt%  $\text{Na}_2\text{SO}_4\text{-60wt% V}_2\text{O}_5$ ) at  $900^\circ\text{C}$ .

- The sequence of the coating provided better resistance of corrosion in decreasing order is Al<sub>2</sub>O<sub>3</sub>-20TiO<sub>2</sub> coating > Al<sub>2</sub>O<sub>3</sub>-13TiO<sub>2</sub> coating > Al<sub>2</sub>O<sub>3</sub>-3TiO<sub>2</sub> coating > 100 Al<sub>2</sub>O<sub>3</sub> coating > uncoated T-22

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