High Temperature Oxidation Behaviour of Plasma Sprayed Al_2O_3 Coating on ASTM-SA213- T-91 Boiler Steel

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Abstract
In the present study Al_2O_3 coating was deposited on ASTM-SA213 T-91 boiler steel by Plasma spray process. The performance of coated as well as uncoated T-91 steel was evaluated for high temperature oxidation in air at 700°C under cyclic conditions. The corrosion kinetics of the oxidation was evaluated by the weight change measurements made after each cycle for a total period of 50 cycles. Each cycle consisted of 1 hour heating in a silicon carbide tube furnace followed by 20 min cooling in ambient air. X-ray diffraction (XRD), scanning electron microscopy/energy dispersive X-ray analysis (SEM/EDAX) techniques were used to analyze the oxidized products. The uncoated alloy suffered intensive spalling in the form of removal of the oxide scale, which may be attributed to the formation of unprotective Fe_2O_3 dominated oxide scales. The Al_2O_3 coated alloy showed much lesser weight gain and the oxide scales remained intact till the end of the experiment. The phases revealed in the oxide scales of the coated specimen were mainly oxides of aluminum which is reported to be protective against the high temperature corrosion.

Keywords: *Oxidation; Scale; Plasma Spray process; Corrosion Kinetics; Al_2O_3 coating*

1. INTRODUCTION

A major drawback of metallic material is the deterioration in their properties with their interaction with the environments in which they are to perform. This leads to an early failure of metallic components with the allied hazards of plant halt and loss of economy. The annual direct loss of natural resources, i.e. metals, owing to environmental degradation is also significant [I].The degradation by oxidation is one of the main cause of failure of hot-section components in the gas turbines, so insight of the oxidation phenomenon is very essential [2]. Oxidation or dry-corrosion is a type of corrosion which involves the re) using various surface modification techniques. The protective action between a metal and oxygen at high temperature in the absence of water or an aqueous phase. The rate of oxidation of a metal at high temperature depends on the nature of the oxide layer that forms on the metal surface [3].

The formation of a stable and slowly growing protective oxide scale such as $A1_2O_3$, Cr_2O_3, or SiO_2 is must for alloys used at high temperatures. However, the protective $A1_2O_3$ scales formed at high temperatures may be prone to crack and spall during thermal cycling. Therefore, to look for a technique for enhancement of adhesion of $A1_2O_3$ scales, so that alloys and coatings can resist high temperatures, has become a major focus of research of high-temperature materials[4]. Ferritic steels, which contains chromium and molybdenum, possess excellent mechanical properties combined with high temperature strength and creep resistance with high thermal fatigue life along with good thermal conductivity, weldability, and resistance to corrosion. Due to these properties, these type of steels have found special place for their application in a number of industries related to carbo-chemistry, oil refining, coal gasification and energy generation thermal power plants, where components like, heat exchangers, boilers and pipes operate at high temperatures and pressures for long periods of time [5,6]. The alloys specicially developed for oxidation resistance usually form a protective layer of chromia or alumina. Better protection is achieved with the rapid formation of oxide layer of alumina or chromia. As this layer grows or as it reforms over areas from which the original layer was removed, it must be able to withdraw chromium or aluminium from the metal so as to facilitate for further scale growth [7]. Oxide scale is formed by a layered structure with variable composition and microstructure from the substrate to the outer interface [8–13]. Recent studies presented that the materials used for high temperature strength are highly susceptible to high temperature corrosion and the surface engineering plays a crucial role in effectively contending the high temperature corrosion problem [14]. One of the protective means to offset the problem of hot corrosion is to coat the base material with a protective layer (coating coatings are being used widely now a days; on structural alloys in energy conversion and utilization systems to protect their surface from high temperature oxidation [15]. Coatings are usually designed in such a way that on exposure to reactive environments, the selective oxidation of an element occurs to form a protective oxide scale such as Cr_2O_3, Al_2O_3 or SiO_2 [16]. Plasma spraying is one of the most versatile thermal spray process with respect to the sprayed materials. The high temperatures of plasma spray processes permit the deposition of coatings for applications in areas of liquid and high temperature corrosion and wear protection and also special applications for thermal, electrical and biomedical purposes [17].

There is not much reported literature on high temperature oxidation manners of plasma sprayed Al_2O_3 coating on boiler steel substrates. Thus, the present work has been focused to study the influence of plasma sprayed Al_2O_3 coating on high temperature oxidation behavior of ASTM-SA213 T-91 boiler steel in air at 700°C under cyclic conditions. The corrosion kinetics of oxidation behavior of plasma sprayed Al_2O_3

coating and uncoated alloy substrate was investigated by thermogravimetric technique. X-ray diffraction (XRD) and Scanning Electron Microscopy/Energy-dispersive analysis (SEM/EDAX) have been used to characterize the oxidized products in order to have an insight in to the corrosion mechanisms.

2. EXPERIMENTAL PROCEDURE

2.1. Development of coating

2.1.1. Substrate material

The Fe-based substrate material selected for the present study, 9Cr-1Mo steel "ASTM-SA213-T-91was in tubular form . The chemical composition of the base material has been given in Table 1 which shows the nominal composition as provided by the supplier and the actual composition measured by optical spectroscopy.

2.1.2. Coating powder

A commercially available Ni-20Cr (Praxair NI-105) metallic powder was used for bond coat and the Al_2O_3 powder namely Alumina with minimum assay 98.0 % (AMPERIT® 740; H. C. Starck) was used for top coat. The coating powders were made available by Metallizing Equipment Co. Pvt. Ltd., Jodhpur (India). Scanning Electron Microscope (SEM) micrograph of the Al_2O_3 powder has been shown in Fig. 1. It is observed from SEM micrograph that the powder particles have irregular morphology with larger particle 42.16μm and smaller one with 6.79μm measured by back scattered electron image (BSEI), which is consistent with the nominal size range provided by the manufacturer.

2.1.3. Coating formulation

The specimens were cut from the alloy tube with approximate dimensions of 20mm×15mm×5mm.Then,specimens were polished using emery papers of 220, 400, 600 grit sizes and subsequently on 1/0, 2/0, 3/0 and 4/0 grades, then cleaned, degreased and shot grit blasted with alumina powder (Grit 60) prior to the deposition of the coating for having better adhesion between the substrate and the coating. The coating was deposited on the substrate using Plasma Spray (a thermal spray process) apparatus SG100 MASS FLOW PLASMA SPRAY. The coating work was carried out by commercial firm namely Metallizing Equipment Co. Pvt. Ltd., Jodhpur (India). The process parameters for the Plasma spray process employed for applying the coating are summarized in Table 2.

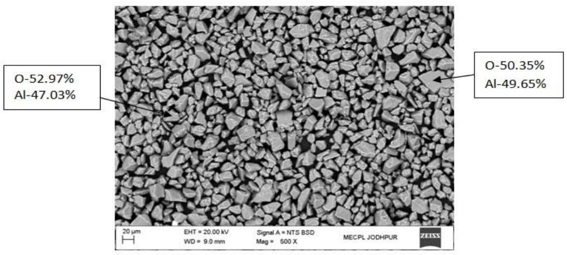

Fig.1 SEM/EDAX analysis of Al_2O_3 coating powder showing elemental composition (% wt)

Table 2: Spray Parameters Employed for Plasma Spraying

Parameters	Values
Arc Current (amp) Anode	730
Arc Current (amp) Cathode	720
Arc Voltage (V)	43
Spraying Distance (inch)	3-3.5
Powder Feed Rate (rev./min.)	6
Plasma Arc Gas (Argon)(slpm)	38.4
Carrier Gas (slpm)	4.15
Water Flow Rate (lpm)	26

2.2. Characterization of the as-sprayed coatings

The Field Emission Scanning Electron Microscope (FEI: CARL ZEISS EVO-18 Research; at Metallizing Equipment Co. PVT. LTD. Jodhpur) fitted with an EDAX attachment (Oxford, UK) was used to characterize the surface morphology of the coating. SEM micrographs along with EDS spectrum were taken with an electron beam energy of 20 keV. The XRD analysis was carried out using a PANalytical Empyrean Advance diffractometer (Netherlands) with Cu K alpha radiation at Metallizing Equipment Co. PVT. LTD. Jodhpur, India. The specimens were scanned with a scanning speed of 2^o/min in 2θ range of 20^o to 120^o and the intensities were recorded. The diffractometer interfaced with the diffraction software that provides the d-values directly on the diffraction pattern.

2.3. High temperature Oxidation Study in Air

The high temperature oxidation study was performed under cyclic conditions for 50 cycles at 700°C. Each cycle consisted of 1 hr of heating at 700°C in a silicon carbide tube furnace followed by 20 min of cooling at room temperature. The alumina boats were used to keep the specimens for insertion in the furnace. The cyclic study provides the most severe conditions for testing of specimens and similar to the actual industrial environment, where frequent shutdowns for maintenance occur. A cyclic study for 50 cycles was chosen to be conducted as the study for 50 cycles is taken as a standard for attaining the steady-state oxidation for the materials [18-21]. The studies were performed for uncoated as well as coated samples for the purpose of comparison. The change in weight measurements were taken at the end of each cycle with the help of Electronic weighing machine with a sensitivity of 1 mg. At the time of measuring weight change to determine the total rate of corrosion, the spalled scale was also taken in to account for total weight measurement. Weight change data was analyzed to know the corrosion kinetics. After the experiment, the corroded specimens were subjected to the XRD and SEM/EDAX analysis for the surface as per the procedure mentioned in Section 2.2.

3. RESULTS

3.1. XRD analysis of the as sprayed coating

The XRD diffractrograms of the Plasma sprayed Al_2O_3 coating in as-sprayed condition on ASTM-SA213-T-91 boiler

Table No 1 Chemical Composition (wt %) of T - 91

Alloy	Composition	C	Mn	Si	S	P	Cr	Mo	V	Ni	Other Elements	Fe
T-91	Nominal	0.08 0.12	0.30 0.60	0.20 0.50	0.01 Max	0.02 max	8.00 9.50	0.85 1.05	0.18 0.25	0.40 Max	Nb=0.06 - 0.10	Bal.
	Actual	0.13	0.44	0.45	0.008	0.009	9.12	1.02	0.10	0.27	Nb=0.08	Bal.

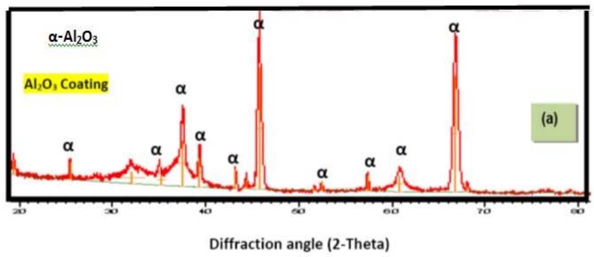

Fig.2 X-ray diffraction patterns for Al_2O_3 coating on T-91 steel

steel are depicted in Fig. 2 on reduced scale. The analysis indicates Al_2O_3 as the principal phase for the Al_2O_3 coatings on T91 boiler steel.

3.2. SEM/EDAX analysis of the as-sprayed coating

The SEM micrographs along with EDS spectrum reveal the surface morphology of the as-sprayed Al_2O_3 coating on ASTM-SA213-T-91 boiler steel showing elemental composition (%) at selected points are shown in Fig. 3. The micro-structure consists of interlocking of particles where the sprayed particles appear to be deformed significantly showing a flattened appearance. The deformed shapes of particles are due to impact of particles having higher velocities during the Plasma spray process. The microscopic features indicate that the coatings are homogeneous and massive, free from cracks (Fig. 3). Presence of some oxide stringers as well as open pores has been noticed in general in the coatings. The dark regions in between may be porosity. The EDAX analysis reveals the domination of Al and O in the grey region (Fig. 3). The composition is nearly same as that of sprayed powder.

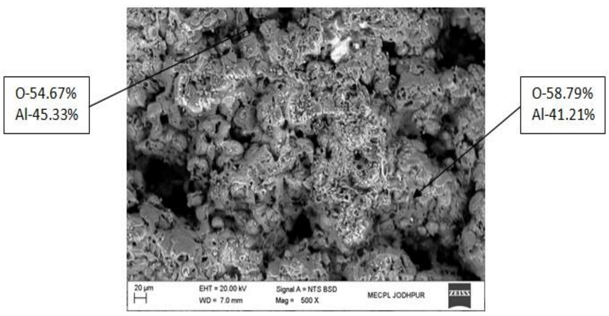

Fig. 3 SEM micrograph and EDAX analysis of Al_2O_3 coating on T-91 showing elemental composition (%wt) at selected points

3.3. High temperature oxidation study in air

The macrographs of the uncoated and coated specimens subjected to high temperature oxidation in air at 700°C for 50 cycles have been represented in Fig. 4. In the case of the uncoated T-91 boiler steel; a minor fragile scale appeared on the surface right from the 10th cycle & there after the scaling was gradual.

Spalling & sputtering of the scale with layers peeling off from 20th cycle onwards. The inner layers, which were rusty in colour till 15th cycle, turned into shiny silver colour after 20th cycle.. At the end of cyclic study, irregular and fragile scale of thin layers was observed (Fig. 4. a). The Al_2O_3 coating on T-91 steel, showed no much visible change during the study. Very minor cracks appeared along the edge in 20th cycle on sides only. The colour of the surface turned into light pale yellow after 50 cycles. No other scaling or spalling was observed. (Fig. 4.b). The coating was found to be adherent to the substrate with very less spallation tendency.

3.4. Corrosion kinetics of the oxidation study

Weight change data calculated during the high temperature oxidation testing has been compiled in Fig. 5. In the case of the Al_2O_3 coated specimen, it can be seen from the plots (Fig. 5.a) that the necessary protection against high temperature oxidation has been provided by the Al_2O_3 coating as the weight gain value for the coated steel is very less as compared to the uncoated steel. The (weight gain/area)^2 versus number of cycles plot (Fig. 5.b.) is shown for the samples to ascertain conformance with the parabolic rate law. The parabolic rate constant Kp were obtained from the slope of the linear regression fitted line (cumulative weight gain/area)^2 vs. number of cycles and are shown in Table 3. It is clear that the data conforms to the parabolic rate law to an acceptable limit for the alloy. The coating also followed the parabolic rate law of oxidation for all the 50 cycles of study, as can be seen from the plots (Fig. 5.b). The parabolic rate constant (Kp) value for the coated substrate is very less than the bare counterpart.

Table 3 Parabolic rate constant Kp values of uncoated and coated T-91 subjected to oxidation in air for 50 cycles at 700^0 C

	Kp (gm^2 cm^{-4} s^{-1})
Uncoated T-91 Boiler Steel	130.86×10^{-10}
Al_2O_3 coated T-91 Boiler steel	16.21×10^{-10}

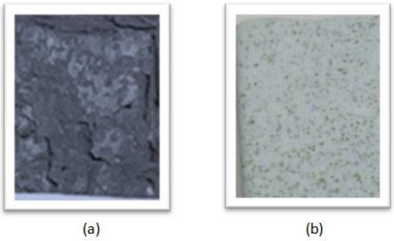

Fig.4 Macrographs of uncoated and coated substrate T-91 steel subjected to oxidation in air for 50 cycles at 700^0 C (a) Uncoated T-91 (b) Al_2O_3

The value of parabolic rate constant (Kp) observed in case of Al_2O_3 coated T-91 boiler steel was 16.21 x 10^{-10} $gm^2cm^{-4}s^{-1}$ and for uncoated T-91 boiler steel was 130.86 x 10^{-10} gm^2 cm^{-4} s^{-1}. Also the overall weight gain (Fig. 5.c) after 50 cycles of oxidation studies for uncoated T-91 steel is 49.73 mg/cm^2 and for the Al_2O_3 coated steel is 16.34 mg/cm^2. Thus it can be interpreted from the weight change plots that the weight gain in case of coated T-91 steel is less than the uncoated T-91 steel.

The plots for the uncoated sample show higher weight gain at initial cycles followed by gradual weight gain. In case of Al_2O_3 coated T-91 steel; the weight change is negligible throughout the study.

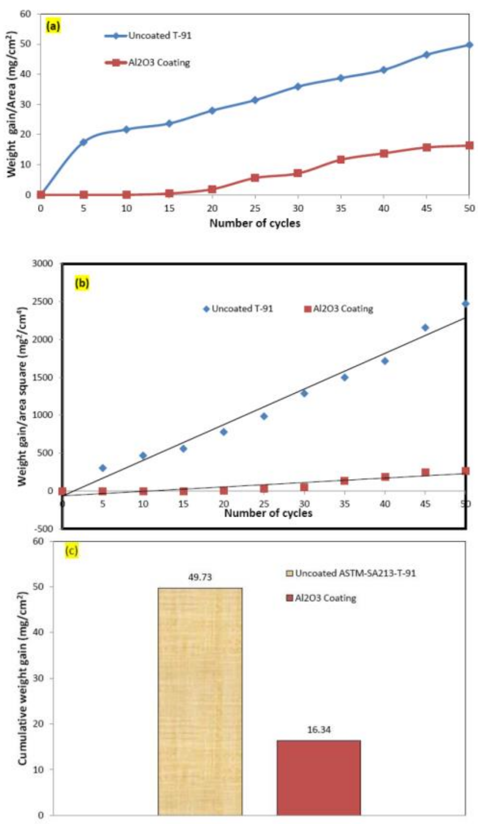

Fig 5 (a)Weight gain/Area vs number of cycles plot (b)(Weight gain/Area)^2 vs number of cycles plot (c) Cumulative weight gain bar chart for uncoated and Al_2O_3 coating on T-91 substrate steel subjected to high temperature oxidation in air for 50 cycles at 700^{0}C

3.5. X-ray diffraction analysis of the scale

The XRD diffractograms (on reduced scale) of the oxide scale for the coated and uncoated steel samples exposed to the high temperature oxidation at 700°C for 50 cycles are depicted in Fig. 6. The scale of uncoated T-91 steel indicated mainly the presence of Fe_2O_3 phase and V_2O_3 and MnO phases (Fig. 6.a). Whereas in case of the Al_2O_3 coated alloy the top scale indicated the presence of Al_2O_3 as main phase (Fig. 6.b).

3.6. SEM/EDAX analysis of the scale

The SEM micrographs indicating morphology of the uncoated and the Al_2O_3 coated specimens after being subjected to the high temperature oxidation in air have been shown in Fig. 7. In the case of uncoated T-91 boiler steel (Fig. 7.a) the micrograph shows corroded oxide scale with significant spalling of its top layer. The surface scale shows distorted grains like microstructure. The exposed area of the scale seems to be dense and presence of cracks is also being noticed. At some other points, the scale has an amorphous appearance. The EDAX analysis shows that the scale is found to be rich in Fe and O, indicating the possibility of Fe_2O_3 formation along with minor traces of Mo, Nb, Cr and Mn. On the other hand, in the case of the Al_2O_3 coated steel, it reveals the formation of a homogeneous, massive and free from cracks structure (Fig. 7.b). The EDAX analysis of the dark grey regions (Fig. 7.b.) of the coating reveals the domination of Al and O.

4. DISCUSSION

The plasma spray process was successful in the deposition of Al_2O_3 coating on the Fe-Cr based alloy i.e. T-91 boiler steel; the coating has nearly uniform, adherent and dense microstructure. The microscopic features indicate that the coating is homogeneous and massive, free from cracks. (Fig. 3). Al_2O_3 have been identified as the main phase by the XRD analysis (Fig. 2) for as-sprayed coating for Al_2O_3 coating on T-91 boiler steel by plasma process. This is further recognized by the EDAX analysis which shows the dominance of Al and O in the coating (Fig. 3). There is a presence of some pores in the micro-structure.

The weight change plots (Fig.5 a & b) for the uncoated and coated T-91 boiler steel indicated that the oxidation behavior has shown conformance to the parabolic rate law. The parabolic kinetic behavior is due to the diffusion controlled mechanism operating at 700 °C under cyclic conditions [22-27]. The higher weight gain during the first few cycles might be credited to the rapid formation of oxides at the splat boundaries and within the open pores due to the penetration of the oxidizing species. Further, the successive increase in weight is gradual [28]. Also in case of coated T-91 steel; once the oxides are formed at places of porosity and splat boundaries, the coating becomes dense and the diffusion of oxidizing species to the internal portions of the coating gets slowed down and the growth of the oxides becomes limited mainly to the surface. This will make the weight gain and hence the oxidation rate steady with the further progress of exposure time [29]. The uncoated T- 91 steel shows the forming of scale with spalling. Spalling of the scale of the bare T-91 may be credited to the strain developed by the precipitation of Fe_2O_3 from the liquid phase during the cooling period of thermal cycles and inter diffusion of intermediate layers of iron oxide [30]. The identification of Fe_2O_3 by the EDAX and XRD analysis showed that non-protective conditions were established on the surface. The formation of Fe_2O_3 in the spalled scale has also been reported to be non-protective [31]. Similar results were reported by Tiwari and Prakash [32-34] and Singh et al. [29] during high temperature corrosion studies of iron-base super alloy at 900°C. The smaller peaks of Cr_2O_3 along with strong peaks of Fe_2O_3 in the case of uncoated T-91 alloy may be due to the existence of Cr in the alloy steel. Similar results have been reported by Chawla et al. [22-25] , Singh [29] and Saddique et al. [35]. The XRD results are further sustained by the surface EDAX analysis (Fig. 7.a.). The authors further reported that Fe–Cr alloys at higher temperature form spinel ($FeCr_2O_4$ and Cr_2O_3) on the inner side and Fe_2O_3 on the outer side of the scale in the presence of oxygen. Both the uncoated and coated alloys showed parabolic behavior with a transition in their Kp values (Table 3). The parabolic rate constant Kp were obtained from the slope of the linear regression fitted line (cumulative weight gain/area)^2 vs. number of cycles. It is clear that in spite of some deviations in the data for the uncoated steel; it follows the parabolic rate law to an acceptable limit. The plasma sprayed Al_2O_3 coated T-91 steel has shown better corrosion resistance than its uncoated counterpart. The Al_2O_3 coating was successful to reduce the oxidation rate of the T-91 steel by 67.14%, in terms of overall weight gain (Fig. 5.c.). In case of Al_2O_3 coated T-91 steel; the weight change was almost negligible throughout the study. The Al_2O_3 is generally regarded as the best protective oxides, with relatively slow diffusion compared to other oxides [36]. Schutze et al. [37] reported a common feature of Al-based coatings that they act as a reservoir phase for the formation of protective and slow growing Al-based oxide scales by reaction with the operation environment, thereby providing an environment barrier against the inward diffusion of aggressive species down to the metal to be protected and slowing down consumption of metal rate by the oxidation process itself. Also, the coated T-91 steel followed the parabolic law, which predicts that the scale formed have shown the tendency to act as diffusion barrier to corrosive species. This depicts the protective behavior of the Al_2O_3 coating against corrosion. The value of parabolic rate constant (Kp) was observed in case of Al_2O_3 coated T-91 boiler steel i.e. 16.21 x 10^{-10} $gm^2cm^{-4}s^{-1}$ and for uncoated T-91 boiler steel i.e. 130.86 x 10^{-10} $gm^2cm^{-4}s^{-1}$.

The top scale indicated mainly the presence of Al_2O_3 phase in the surface XRD analysis for the Al_2O_3 coated alloy after oxidation studies. No sign of diffusion of the substrate elements have been identified in the surface XRD analysis (Fig 6.b). The surface EDAX analysis (Fig. 7.b) also supports the XRD analysis results. Al and O have been recognized as the main phases by the analysis. The presence of Al_2O_3 phase in the oxide scales of the coated steel provides protection to

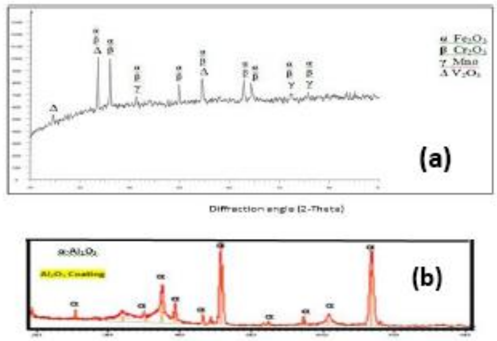

Fig 6 X-ray diffraction patterns for (a) uncoated ; (b) coated T-91 steel subjected to oxidation in air for 50 cycles at 700^0 C

base metal against any diffusion of oxidizing species. These oxide phases are stable [38] up to elevated temperatures due to its high melting point as well as it forms a dense and adherent layers that grow relatively slow [39]. The scale of this type forms a solid diffusion barrier that inhibits interaction of oxygen of underlying coating. Coatings are usually designed in such a way that on exposure to reactive environments, the selective oxidation of an element occurs to form a protective oxide scale such as Cr_2O_3, Al_2O_3 or SiO_2 [16]. The formation and growth of oxide scales are determined by an extensive range of parameters, including the alloy composition and the oxidizing environment. Therefore, it may be very difficult to establish a continuous, protective scale under some conditions [40].These oxides are very protective as reported by Ul-Hamid [19] and Sundarajan et al. [41]. Thus, based on the present study, it can be concluded that plasma sprayed Al_2O_3 coating can provide a very good high temperature oxidation resistance to the T-91 boiler steel.

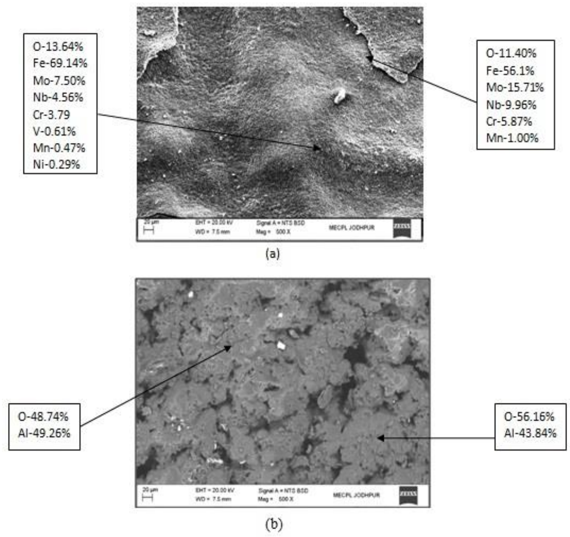

Fig.7 SEM micrographs and EDAX analysis of uncoated and coated T-91 subjected to oxidation in air for 50 cycles at 700^0 C (a) Uncoated T 91 (b)Al_2O_3

5. CONCLUSIONS

- Fe based alloys namely T-91 boiler steel was successfully sprayed by **Al_2O_3** ceramic powder with the help of plasma spray process, which resulted in a coating which has nearly uniform, adherent and dense microstructure.
- The uncoated T-91 boiler steel showed weight gain, high corrosion rate, irregular and fragile scale with cracks, shiny silver colour surface appearance and spallation of their oxide scale during high temperature oxidation studies at 700°C. The plasma sprayed **Al_2O_3** coated T-91 substrate when subjected to high temperature oxidation at 700°C; was found to be effective for maintaining its adherence with the substrate steel. Also, the oxide scale was found to be intact and there was no indication of spalling.
- The **Al_2O_3** coating successfully reduced the rate of corrosion of the T-91 steel by 67.14%, in terms of overall weight gain. In case of **Al_2O_3** coated T-91 steel; the weight change is less throughout the study than the bare counterpart.
- Thus, the plasma sprayed **Al_2O_3** coating was proved to be beneficial in enhancing high temperature oxidation resistance of T-91 alloys.

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