HIGH-TEMPERATURE BEHAVIOR OF AL_2O_3 AND STELLITE- 6 COATED ON ASTM-SA210-GRADE A1 BOILER STEEL IN THE ACTUAL INDUSTRIAL ENVIRONMENT OF COAL FIRED BOILER

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ABSTRACT

In this work, Al_2O_3 and Stellite-6 coatings were deposited on ASTM-SA210-Grade A1 boiler steel using Balzer's rapid coating system (RCS) machine (make Oerlikon Balzers, Swiss) under a reactive nitrogen atmosphere. The oxidation behaviour of Grade A1 coated steel in air has been studied under isothermal conditions at a temperature of 900°C in a cyclic manner. Oxidation kinetics was established for the Grade A1 Coated steel in air at 900°C under cyclic conditions for 10 cycles by thermogravimetric technique. Each cycle consisted of 1 hour heating at 900°C followed by 20 min of cooling in air. Stellite-6 coated Grade A1 steel showed minor amount of weight gain after each cycle. X-ray diffraction (XRD) and scanning electron microscopy/energy dispersive X-ray (SEM/EDAX) techniques were used to characterise the oxide scales. Stellite- 6 coated steel was found to be more corrosion resistance than Al_2O_3 Coated steel in air oxidation for 10 cycles.

Keywords: Hot corrosion, Stellite- 6, Thermal sprayed Coated

I. INTRODUCTION

In a wide variety of applications, mechanical components have to operate under severe conditions, such as high load, speed temperature and hostile chemical environment. Thus, their surface modification is necessary in order to protect them against various types of degradation [1]. Materials used for high temperature applications are subjected to various types of degradation phenomenon such as high temperature corrosion, erosion-corrosion, overheating, solid particle abrasion, wear, etc. High temperature corrosion of boiler tubes used for super -heaters and re-heaters in steam-generating systems has been recognized as a severe problem, resulting in tube wall thinning and premature failure [2][4]. High temperature erosion–corrosion of heat transfer pipes and other structural materials in coal fired boilers is recognized as being the main causes of downtime at power generating plants, accounting for 50–75% of the total arrest time [3][5]. Maintenance costs for replacing broken pipes in the same installations are also very high, and can be estimated at up to 54% of the total production costs. Superalloys have been developed for high temperature applications, but they are not able to meet both the high-temperature strength and the high-temperature erosion–corrosion resistance simultaneously [6]. One possible way to address these problems is by applying a thin layer of anti-wear and anti-oxidation coatings with good thermal conductivity, such as thermal sprayed nickel or iron based alloyed coatings [7][9]. Thermal sprayed coatings are economical, can be produced by means of relatively simple techniques and offer an excellent corrosion and wear protection. Moreover, other favourable properties can be produced at the coating or component surface. As a result, these coatings have found use in various industrial applications [10][4]. Detonation gun (D-gun) spray coating process is a thermal spray process, which gives an extremely good adhesive strength, low porosity and coating surfaces with compressive residual stresses [5][8].

This paper is intended as a contribution to the knowledge of the oxidation behaviour of the Al_2O_3 and Stellite-6 Coated on ASTM-SA210-Grade A1 Boiler Steel in an actual industrial environment of coal fired boiler. Techniques such as x-ray diffraction (XRD), scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS), and electron probe microanalysis (EPMA) were used to analyze the samples after testing in the industrial environment.

II. EXPERIMENTAL MATERIAL

2.1 Substrate Steels

Al_2O_3 and Stellite-6 coatings were deposited on steel ASTM-SA210-Grade A1, which has a wide range of applications in boilers, especially when the service conditions are stringent from the point of temperature and pressure. The nominal chemical composition is reported in Table 1. Specimens with dimensions of approximately 20mm x 15mm x 5mm were cut from the alloy sheet. Polished using emery papers of 220, 400, 600 grit sizes and subsequently on 1/0, 2/0, 3/0, and 4/0 grades, and then mirror polished using cloth polishing wheel machine with 1μm lavigated alumina powder suspension. The specimens were prepared manually and all care was taken to avoid any structural changes in the specimens.

Table 1. Nominal chemical composition (Wt %) for “ASTM-SA210-Grade A1” boiler tube steel used in present study

Alloy	C	Mn	Si	S	P	Cr	Mo	Fe
Grade A1	0.27	0.93	0.1	0.058	0.048	-	-	Bal.

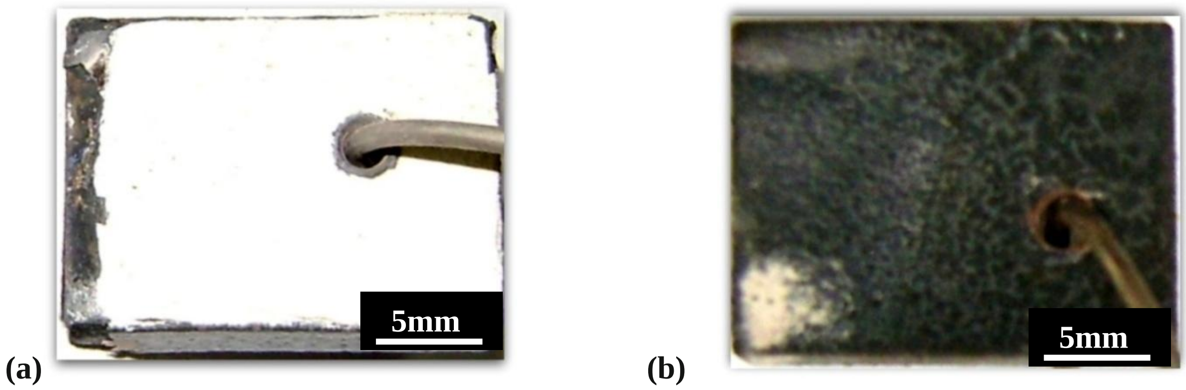

Fig. 1 Macrographs of the ASTM-SA210-Grade A1steel samples exposed to a platen superheater of the coal-fired boiler for 1000 h: (a) coated Al_2O_3 steel,and (b) Stellite- 6 coated ASTM-SA210-Grade A1steel

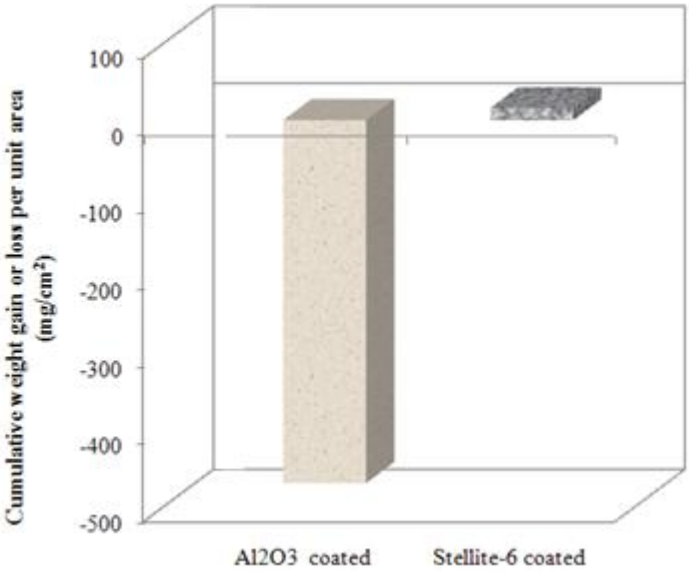

Fig. 2 Bar chart showing cumulative weight gain or loss per unit area for Al_2O_3 and Stellite- 6 Coated ASTM-SA210-Grade A1 steel exposed to a platen superheated of the coal-fired boiler for 1000 h at 900^o C

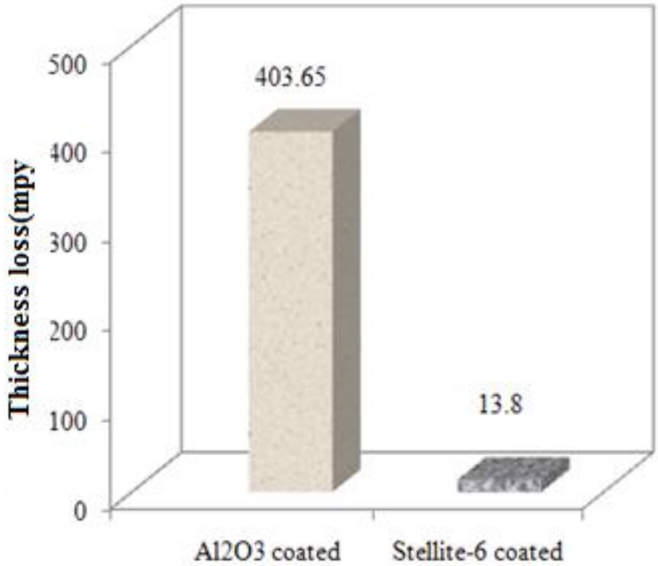

Fig. 3 Bar chart indicating the thickness change in mpy by the Al2O3 and Stellite- 6 coated ASTM-SA210-Grade A1 steel exposed to a platen superheated of the coal-fired boiler for 1000 h at 900^o C

2.2 Development of Coatings

Specimens were grit blasted with alumina powder (Al_2O_3) before coating. Commercially available, Al_2O_3 and Stellite-6 alloy in the powder form were used as the coating material in the study. The coating powders were made available by SVX POWDER M SURFACE ENGINEERING Pvt. Ltd., Greater Noida (India). All the process parameters, including the spray

distance, were kept constant throughout the coating process. The standard spray parameters used for the deposition of the coating are given in Al_2O_3 were oxygen flow rate, 4800 L/H, Fuel (Acetylene) Flow Rate 1920 L/H, Carrier Gas(Nitrogen) Flow Rate 800 L/H, Spray Distance 200mm, Flame Temperature 3900 ^{0}C, Detonation Frequency 3 Shots/Sec, and Stellite-6 oxygen flow rate, 3100 L/H, Fuel (Acetylene) Flow Rate 2400

L/H, Carrier Gas(Nitrogen) Flow Rate 720 L/H, Spray Distance 165mm, Flame Temperature 3900 ^{0}C, Detonation Frequency 3 Shots/Sec.

2.3 Studies in Coal-Fired Boiler

The studies were performed for Al_2O_3 and Stellite- 6 coated specimens in the middle zone of platen superheated of the Stage-II boiler of Guru Nanak Dev Thermal Plant, Bathinda, Punjab (India). The specimens were hung through the soot blower dummy points at 27-m height from the base of boiler, using a nichrom wire passed through a 1.5-mm hole drilled in the samples. The average temperature was about 900^{o}C with a variation of 10°C. The average volumetric flow of flue gases was 231 m^3/s. The SOx and NOx values of the flue gases were 290 and1183l g/m^3, respectively. Flue gases contain 12% CO_2 and 7% O_2 by volume. The samples were exposed to the combustion environment for a total of 10 cycles, each consisting of a 100-h exposure followed by 1-h cooling at ambient conditions. Physical observations were made after each cycle. The weight change was measured at the end of 10 cycles. However, because of suspected oxide spalling and ash deposition on the samples, the weight change data could not be used directly for predicting degradation behavior. Therefore, the extent of degradation of the specimens was assessed by the thickness loss and depth of internal corrosion attack. After the cyclic study in actual boiler environment, the samples were analyzed using XRD, SEM/EDS. The oxidized samples were cut using Isomet 1000 precision cutter across the cross section and mounted in transoptic powder for the cross-sectional analysis.

III. RESULTS

3.1 Thickness of the sprayed coating analysis

Average thickness of the coatings was measured from the Back Scattered Electron Images (BSEI) for the Al_2O_3 and Stellite- 6 coating on ASTM-SA210-Grade A1 steel shown in Fig. 4. In the micrograph, three regions namely substrate, coating and epoxy are visible. Average thickness of the coating was measured as 260-330 μm.

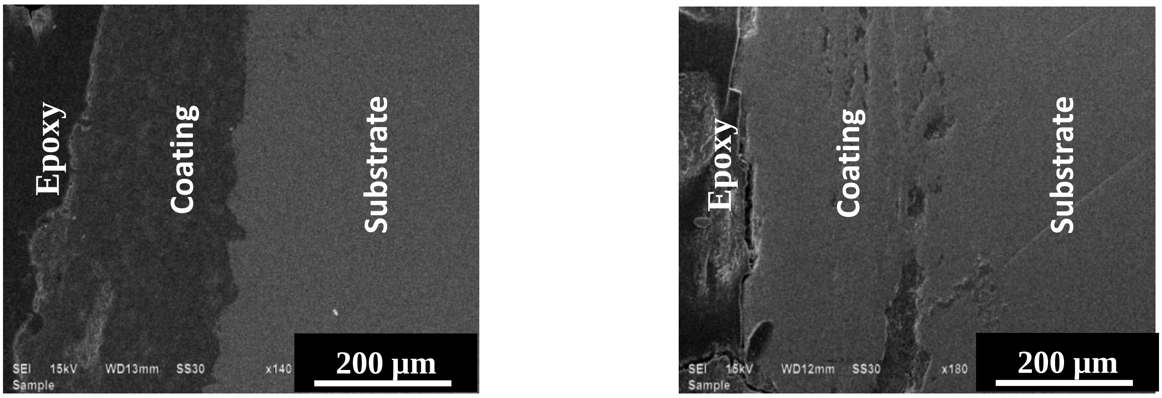

Fig. 4. BSEI micrographs showing cross-sectional morphology of D-gun sprayed a). Al_2O_3 and b). Stellite- 6 coating on ASTM-SA210-Grade A1 boiler steel specimen.

3.2 Visual observations and weight-change analysis

Photographs of the D-gun spray Al_2O_3 and Stellite- 6 coated ASTM-SA210-Grade A1 steel after exposure to the oxidation studies in air at 900 °C for 10 cycles are shown in Fig. 1. In case of Al_2O_3 coated "ASTM-SA210-Grade A1" steel, coating started peeling off from edges after 1st cycle. Cracks developed at edges and a part of base material was seen after 2nd cycle. Base material was peeled off layer after other from the surface where coating was damaged till 5th cycle. There was swelling of material surface where there was no coating after 5th cycle. Cracks appeared all over the bare surface during cooling after 6th cycle while some part of the material having coating remained same Fig. 1(a). On the other hand In case of Stellite-6 coated "ASTM-SA210-Grade A1" steel, there was no change seen upto 3rd cycle. At the end of 4th cycle small crack was seen on the surface. The cyclewise macrographs of high temperature erosion corrosion of Al_2O_3 coated "ASTM-SA210-Grade A1" steel are shown in Fig. 1(b). Surface condition remained same upto 10th cycle as it was after 4th cycle. It shows that the coating has protected the material.

Cumulative Weight change (mg/cm^2) versus number of cycles plots for the Al_2O_3 and Stellite- 6 sprayed ASTM-SA210-Grade A1 boiler steel after oxidation studies in air environment at 900 °C up to 10 cycles are shown in Fig. 2. The weight change data is usually used to establish the kinetics of the oxidation process. A higher weight gain represents higher rates of oxidation. Therefore, the oxidation rates of various materials can be compared with the help of weight change data. Based on these facts, It can be inferred from the plots that the Al_2O_3 coated Grade A1 boiler steel have shown high rate of erosion-corrosion as compared to Stellite-6 coated Grade A1 boiler steel. The cumulative weight gain or loss per unit area (mg/cm^2) after completion of 10 cycles is presented in Fig. 2. The cumulative weight loss after completion of 10 cycles of erosion and oxidation for Al_2O_3 coated Grade A1 steel are 470 mg/cm^2 respectively, while cumulate weight gain for Stellite-6 coated Grade A1 steel is 15.77 mg/cm^2. From thickness loss data the erosion-corrosion rates were evaluated in mils/year as shown in Fig. 3.

3.3. Thickness change in actual boiler environment

The extent of oxidation–erosion loss was measured in terms of metal layer lost due to scaling after 1000 h exposure Fig.3. The thickness of metal lost in oxidised–eroded Al_2O_3 coated Grade A1 boiler steel is 1.31 mm. Based upon these values, the thickness loss rate indicated by Al_2O_3 coated steel is calculated as 403. 65 mils per year (mpy).On the other the thickness loss value for the Stellite-6 coating is 0.47 mm and the corresponding thickness loss rate is found as 13.8 mpy

3.4. X-ray diffraction (XRD) analysis

XRD for Al_2O_3 coated "ASTM-SA210-Grade A1" boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment shown in Fig. 5. As is clear from the diffractograms, Al_2O_3, Fe_2O_3 & SiO_2 are the main phases present in the oxide scale of Al_2O_3 coated and Stellite-6 coated Grade A1 boiler steel. The XRD diffractograms for the Stellite-6 coated "ASTM-SA210-Grade A1" boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment shown in Fig. 6. respectively. Fe_2O_3, Al_2O_3 and SiO_2 are identified as the strong intensity phases in Grade A1 steel.

3.5 SEM / EDAX analysis

SEM micrograph and EDAX analysis for Al_2O_3 coated "ASTM-SA210-Grade A1" boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment, (X200). In case of Al_2O_3 coated "ASTM-SA210-Grade A1" steel after high temperature erosion-corrosion at 900°C for 1000 hours, SEM micrograph shows grey phases along with whit phase globules in Fig. 7. The EDAX point analysis indicates that grey phase (point 3) is rich in O, Si and Al, while globules (point 4) shows maximum amount of O and Si with small amount of Al. SEM micrographs and EDAX analysis of Stellite-6 coated "ASTM-SA210-Grade A1" steel after high temperature erosion-corrosion at 900°C for 1000 hours is shown in Fig. 8. SEM micrograph indicates grey and dark phases. The grey phase (point 5) is rich in O, Al and Si. Small amount of Na, P, Ca, Cr and Fe are also present. The dark phase (point 6) shows maximum oxygen and chromium content along with Al, Si, Ca and Fe.

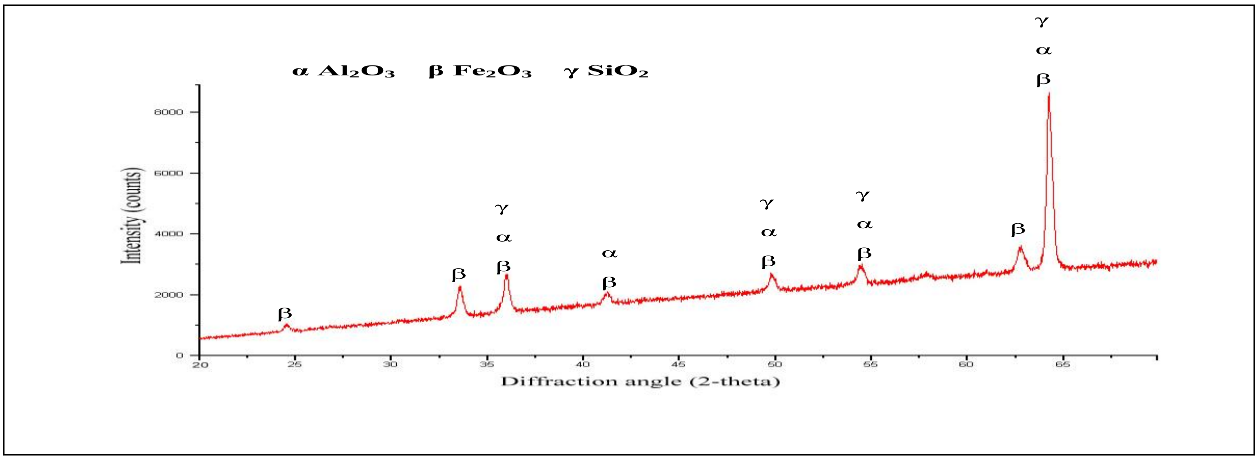

Fig. 5 XRD patterns for Al_2O_3 coated “ASTM-SA210-Grade A1” boiler steel exposed to a platen superheater of the coal-fired boiler for 1000 h at 900^{o}C.

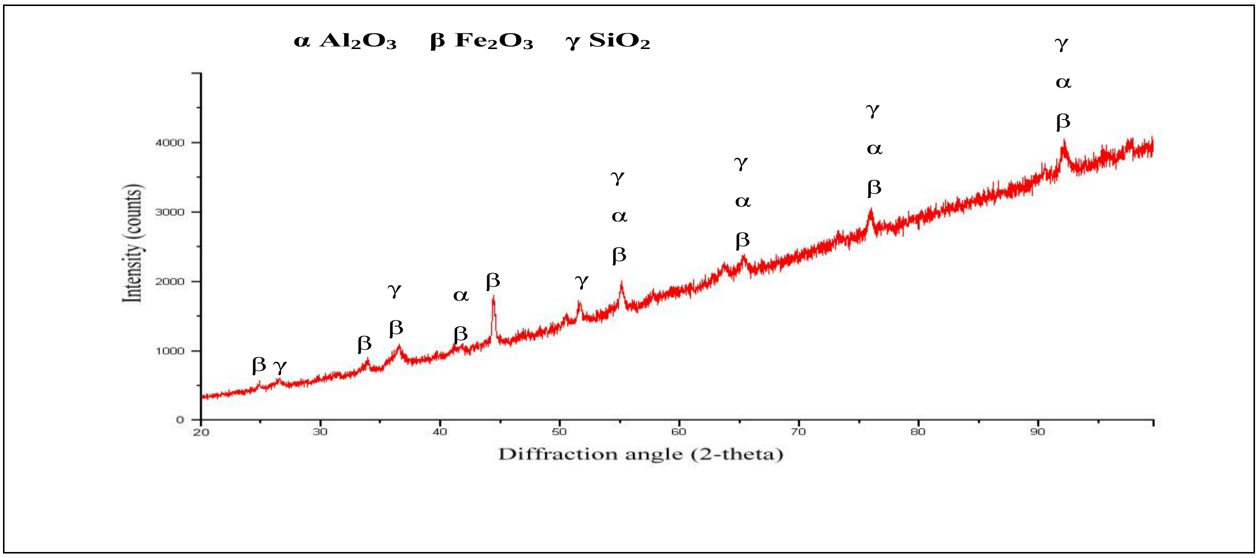

Fig. 6 XRD patterns for Al_2O_3 coated “ASTM-SA210-Grade A1” boiler steel exposed to a platen superheater of the coal-fired boiler for 1000 h at 900^{o}C.

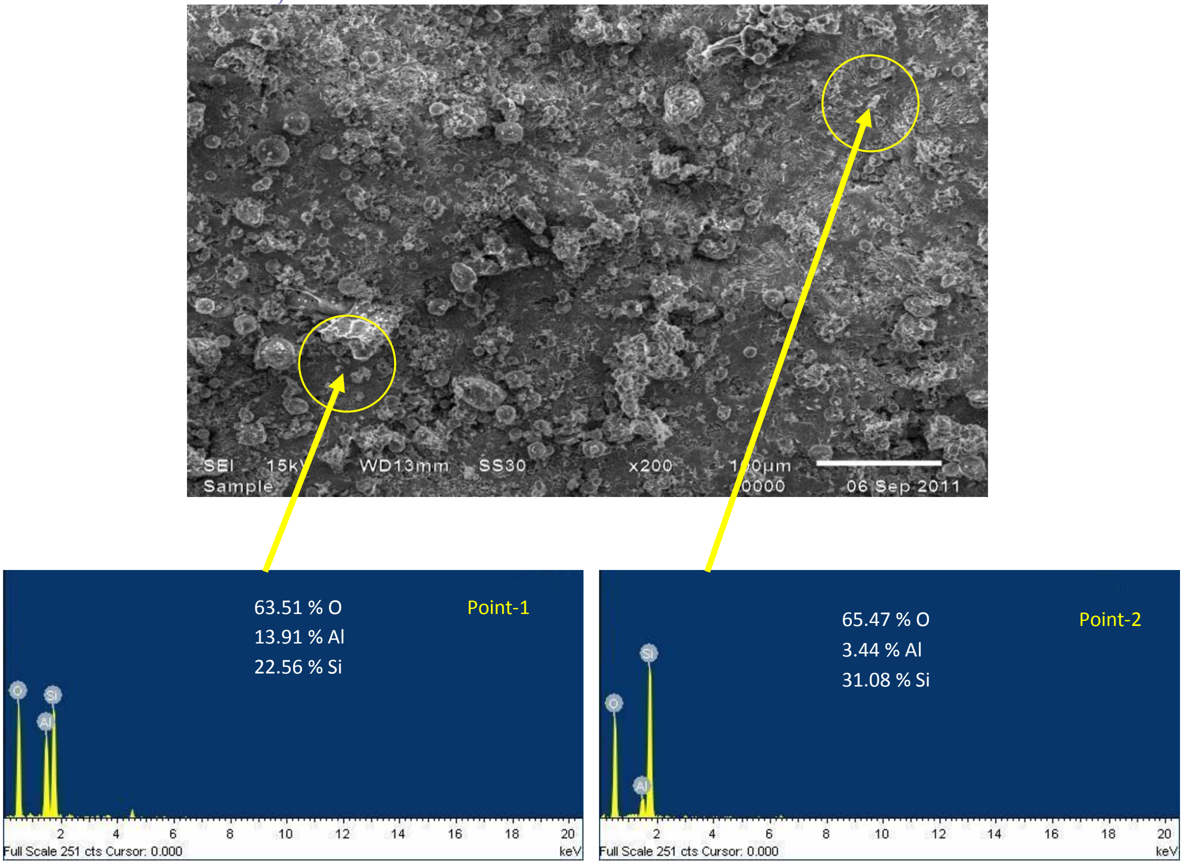

Fig. 7 SEM micrograph and EDAX analysis for Al_2O_3 coated “ASTM-SA210-Grade A1” boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment, (X200).

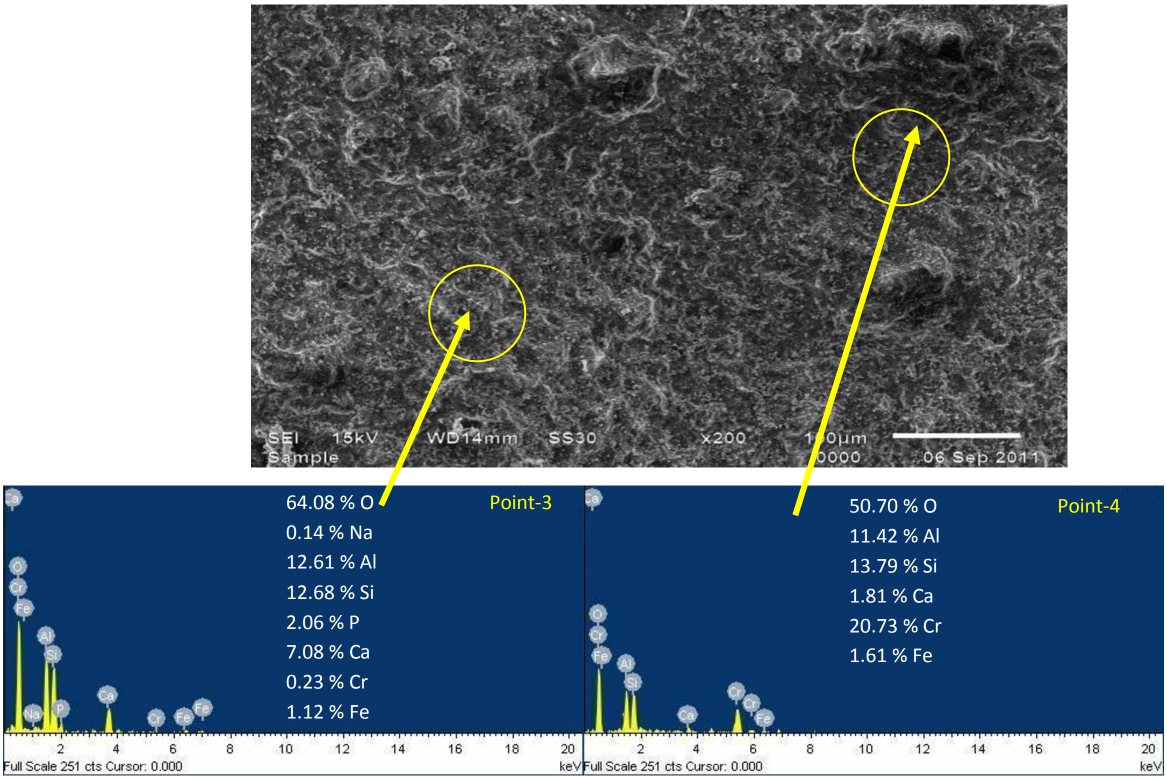

Fig. 8 SEM micrograph and EDAX analysis for Stellite-6 coated "ASTM-SA210-Grade A1" boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment, (X200).

3.6 Cross-sectional scale morphology

BSEI and elemental variation across the cross-section of coated "ASTM-SA210-Grade A1" boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment is shown in Fig.8 BSEI micrograph in case of Al_2O_3 coated Grade A1 boiler steel is shown in Fig.9 (a). Micrograph shows the formation of thick scale. EDAX analysis reveals that scale is rich in Fe and O along with minor amount of C. The top portion (point 7) of the scale mainly consists of Fe and O and little amount of C, Ni and Al. Outer portion of the scale point out the possibility of Fe_2O_3. In case of satellite-6 coated Grade A1 boiler steel shown in Fig.9 (b) EDAX analysis reveals the presence of oxygen in greater amount in scale. At bottom (point 4) scale consist of oxides of chromium and iron. Moving towards outermost layer of the scale, the presence some amount of Ni, W and significant amount of Co is revealed by the EDAX analysis.

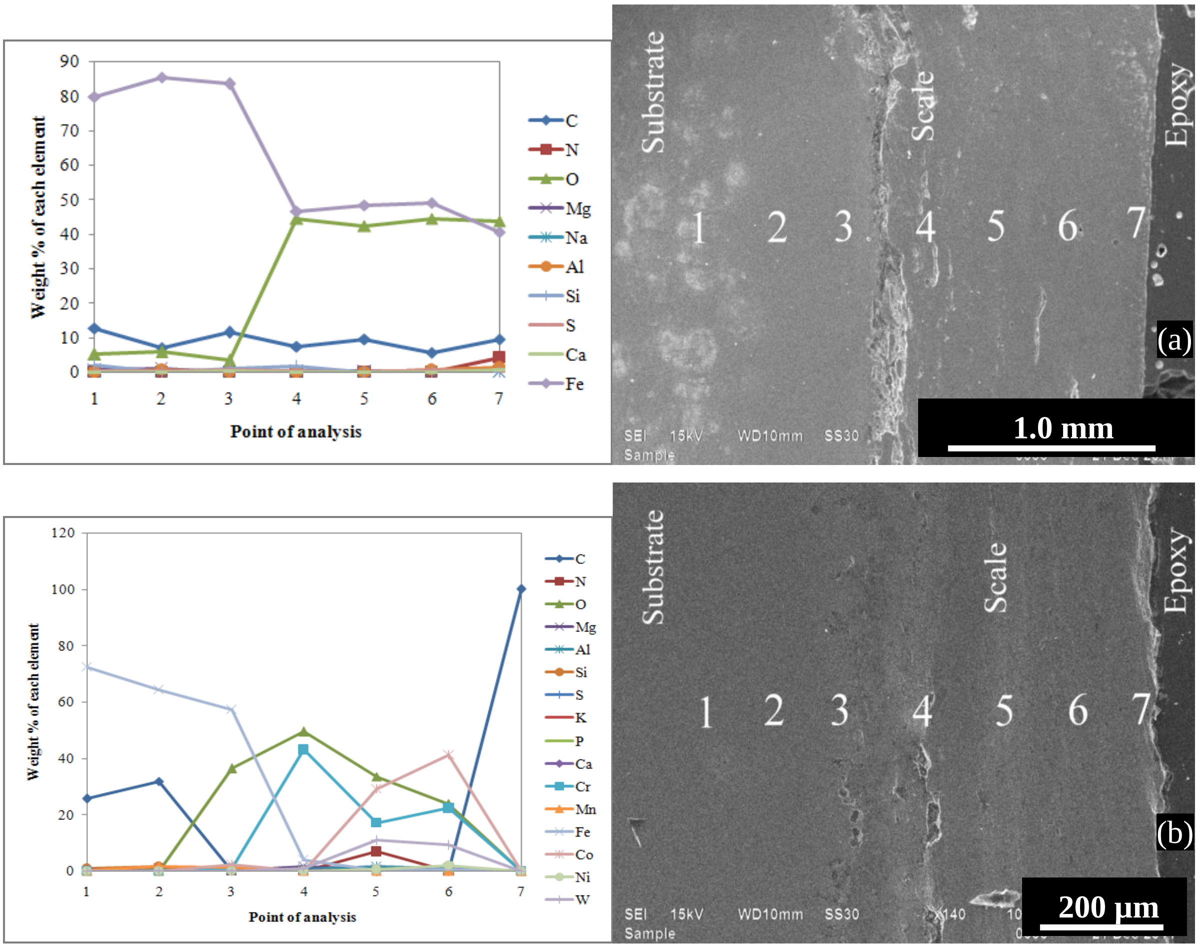

Fig. 9 Oxide scale morphology and variation of elemental composition across the cross-section of coated "ASTM-SA210-Grade A1" boiler steel after high temperature erosion-corrosion at 900°C for 1000 hours in coal fired boiler environment, (a) Al_2O_3 coated; (b) Stellite-6 coated.

IV. DISCUSSION

At elevated temperatures, oxidation resistance of coatings in prevailing conditions is one of the major factors in determining the erosion loss rate, but interlamellar cohesion, porosity, ductility, and original splat size of the coating have a great effect, as well [11]. In general, spraying systems using high particle velocities produce dense coatings with small splat size, high bonding strength and large contact area between individual splats. Under erosion exposure, the size of the disintegrated pieces is determined by original splat size of the coating. Thus, high velocity coating systems have good erosion resistance because disintegration of a single platelet requires high energy and loosened pieces are small [12]. Coatings produced by D-gun spraying techniques are with very high bond strength and generally porosity lower than 2% with thickness ranging from 50 to 500 μm as reported by [14]. Al_2O_3 and Stellite-6 coating has been formulated successfully by D-gun spraying technique on "ASTM-SA210-Grade A1" boiler tube steel. The coating thickness has been measured along the crossection of samples and found to be in the range of 260- 330 μm as taken by [17]for study of performance of coatings on boiler tube steel in actual industrial environment. Coating microstructure presented in Fig 4 (a) and Fig 4 (b) indicates dense coating of Al_2O_3 and Stellite-6 on Grade A1 steel respectively. The oxides required for the purpose of corrosion and oxidation resistances are Al_2O_3, Cr_2O_3 and SiO_2 [17]. Al_2O_3 coating is already oxide which is corrosion resistant. In SEM/EDAX micrograph of Stellite-6 coating minor dark phases consist of O, Cr, Co, W along with C and Fe whereas in grey phase C and Fe are absent. However both the phases are rich in Cr, Co and O. Minor peaks of CoO along with high peaks of CrCo and FeNi have been observed in XRD analysis of Stellite-6 coating as observed by [16] for Stellite-6 coating deposited by HVOF coating technique.Observing the BSEI of cross-section, the coating substrate interface has no gaps, which indicates good adhesion between coating and substrate. The elemental variation is shown in mapping which indicates Al and O in Coating of Al_2O_3 coated sample and Cr, Co as main elements in Stellite-6 coating.

During erosion-corrosion study of coated "ASTM-SA210-Grade A1" boiler tube steel in actual industrial environment, weight loss of Al_2O_3 coated Grade A1 steel increased with progress of cycles. In case of Al_2O_3 coated specimen one side coating was separated after 2nd cycle while other side coating was seen depleting slowly upto 10th cycle. Al_2O_3 coated specimen gained some weight after 5th cycle, and then loosed weight continuously upto 9th cycle and finally gained minor amount of weight after 10th cycle. Higher erosion rate of Al_2O_3 coated Grade A1 steel might be responsible for weight loss. Minor increase in weight after some cycles might be due to the blockage of pores of specimens by oxides, which acted as diffusion barrier to inward diffusion of oxidizing agents. Stellite-6 coated Grade A1 steel showed minor amount of weight gain after each cycle. Erosion-corrosion rate in terms of mpy for coated Grade A1 steel followed the sequence: Al_2O_3 coated > Stellite-6 coated

There is uniform deposition of ash (Si, Mg, Ca etc) on the surface of coated Grade A1 steel which is revealed by the EDAX analysis. Al_2O_3 coated Grade A1 steel shows the formation of Al_2O_3, Fe_2O_3 and SiO_2 as indicated by SEM/EDAX and supported by XRD analysis. During high temperature corrosion, initially the corroding agents react with top surface of the coating and starts mitigating through the intersplat interface and diffusion of elements of substrate for e.g. iron moves upward along this inter splat space at the coating substrate interface as reported [20]. In case of Stellite-6 coated Grade A1 steel, EDAX analysis reveals the presence of O, Al, Si and Cr along with minor amount of Na, P, Ca and Fe on upper surface of the scale. XRD analysis shows the formation of Al_2O_3, Cr_2O_3 and SiO_2. Cross-sectional analysis of the Stellite-6 coated specimen after boiler exposure reveals that scale is rich in Cr, Co and O with presence of minor amount of W and Si. Cobalt provides toughness, chromium provides corrosion resistance and wear resistance is provided by complex carbides of W, Co and Cr (Souza and Neville, 2007). The protection shown by this coating may be due to the formation of oxides of chromium and cobalt as reported [18].

The maximum resistance to erosion-corrosion indicated by Stellite-6 coated specimen as compared to Al_2O_3 coated specimen might be due to dense and less porous structure obtained by D-gun spraying technique and presence of thick chromium and cobalt rich band in the scale.

The porosity of the coatings is of prime importance in the hot corrosion studies. Dense coatings are supposed to provide a very good corrosion resistance compared to porous coatings as reported [19].

V. CONCLUSIONS

Performance of Al_2O_3 and Stellite-6 coatings deposited on "ASTM-SA210 Grade A-1" boiler steel by D-gun spraying technique Grade A1 steel has been investigated in coal fired boiler environment at 900°C for 1000 hours (10 cycles). The conclusions from the present study have been mentioned below:

- Al_2O_3 and Stellite-6 coatings have been successfully deposited by D-gun spraying technique with coating thickness of range 260- 330 μm.
- During coating process oxygen diffused into coating.
- Erosion-corrosion resistance for coated Grade A1 steel at high temperature followed the sequence, Stellite-6 coated > Al_2O_3 coated
- Coated Grade A1 steel specimens have shown ash deposition on their surface.
- Al_2O_3 coated Grade A1 steel undergone intense spalling and peeling of scale from the part of where coating was separated from specimen after 2nd cycle along with weight loss.
- Stellite-6 coated Grade A1 steel gained minor amount of weight up to 10th cycle.
- Stellite-6 coating has provided better protection to Grade A1 steel against high temperature erosion-corrosion as compared to Al_2O_3 coating. This may be due to the formation of protective oxide Cr_2O_3.

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