Hot Corrosion behavior of CNT reinforced Al₂O₃ Coated T-11 boiler Steel in Na₂SO₄-60% V₂O₅ environment at 900°C under cyclic condition

Amandeep Singh^{*1}, Vikas Chawla² and NeelKanth Grover³ ¹Research Scholar, Singhania University, Jhunjhunu-333515, India ²Mechanical Engineering, I.K.G.P.T.U., Kapurthala -144603, India ³Mechanical Engineering, I.K.G.P.T.U., Kapurthala -144603, India *sharma_aman26@yahoo.com, Phone: +91-9872376156

Abstract-----In this work, hot corrosion surface behaviour of bare and CNT reinforced Al_2O_3 coated T-11 boiler steels has been studied. Cyclic oxidation studies in molten salt (Na_2SO_4 - $60\%V_2O_5$) environment were conducted at a temperature of 900°C for 50 cycles in the laboratory using silicon carbide furnace.. Each cycle consists of 1 hour heating at 900°C followed by 20 min of cooling in air at room temperature. X-ray diffraction (XRD) and scanning electron microscopy/energy dispersive X-ray (SEM/EDAX) techniques were used to characterise the oxide scales of surface. The results obtained showed the CNT reinforced Al_2O_3 coated T-11 steels were found to be more corrosion resistance than bare T-11 steel.

Keywords----- Hot Corrosion; Bare and CNT reinforced Al₂O₃ coated T-11boiler steel

I. INTRODUCTION

Hot corrosion was first recognized as a serious problem in 1940's in connection with degradation of fireside boiler tubes in coal fired steam generating plants and later with the severe attack of gas turbine air-foil materials [1]-[4]. It is a serious problem in power generation equipment, in gas turbines for ships and aircrafts and in other energy conversion and chemical process systems e.g. in boilers, internal combustion engines, fluidized bed combustion and industrial waste incinerators [5 and 6]. A study revealed that 25% to 30% of annual corrosion costs in the U.S. could be saved if proper corrosion management practices were employed [7].

Chromium and molybdenum constituents in ferritic steels are renowned for their excellent mechanical properties. In addition to this, they possess high temperature strength and creep resistance with high thermal fatigue life, as well as with good thermal conductivity, weldability, and resistance to corrosion. Due to these characteristics, this type of steels have shown their importance for application in industrial processes related to carbochemistry, oil refining, carbon gasification and energy generation in thermal power plants, where components like, heat exchangers, boilers and pipes operate at high temperatures and pressures for long periods of time [8]-[10].

Degradation of metals and alloys due to hot corrosion has been identified as a serious problem for many high temperature aggressive environment applications, such as boilers, internal combustion engines, gas turbines, fluidized bed combustion, and industrial waste incinerators [11 and 12]. Due to depletion of high-grade fuels and for economic reasons, residual fuel oil along with coal is used extensively in the energy generation systems such turbines. boilers, and industrial as waste incinerators. The impurities namely, Na, V, K and S present in the fuel oil, react together to form low melting point compounds on the surface of materials and induce accelerated oxidation (hot corrosion). The degradation of material occurs when these molten compounds dissolve the protective oxide layers that naturally form on materials during boiler/gas turbine operation, inability to either totally prevent the hot corrosion or its detection at an early stage has resulted in several accidents, leading to loss of life and/or destruction of engines/infrastructures [13]. Moreover, the vanadium compounds are also good oxidation catalysts and allow oxygen and other

gases in the combustion atmosphere to diffuse rapidly to the metal surface and cause further oxidation. As soon as the metal is oxidized, the cycle starts over again resulting into high corrosion rates [14 and 15].

Therefore, the evolution of wear and hightemperature corrosion protection systems in industrial applications is very significant topic from both engineering and an economic perspective [16].

So coatings play significant role to protect the materials used in aggressive environment against the oxidation and corrosion attack [17]. Recent studies reveal that coating application used 80% of the total cost for the protection of metals [18].Coatings extend the life of materials by protecting them against wear, oxidation and corrosion.

Various coating process are used to improve the surface property of various metals for different application. Thermal spray coating process is well known process to improve the wear resistance of surface. Thermal spray process carried out in a different way like plasma spray, arc spray, detonation gun spray, flame spray etc. [19 and 20].

This paper is intended as a contribution to the knowledge of the hot corrosion behavior of CNT reinforced Al_2O_3 Coated T-11 boiler Steel in molten salt (Na_2SO_4 -60% V_2O_5) environment at 900°C in cyclic manner. In this study, detonation gun spray method is used for coating process to enhance the micro hardness of coated surface, the adhesive property and also for the sliding applications.

II. EXPERIMENTAL MATERIAL AND PROCEDURE

A. SUBSTRATE STEEL

The boiler tube steel T-11(ASTM -A199/A450 -T-11) has been selected as the substrate materials for the experimental work. The T-11 steel alloys were acquired from Guru Nanak Dev Thermal Power Plant, Bathinda (Punjab, India) for protecting them against high temperature corrosive environment applications. The chemical composition of T-11 boiler steel is as reported in Table 1.

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B. PREPARATION OF SUBSTRATE MATERIALS

Specimens with dimensions of approximately 20mm X 15mm X 5mm were cut from the boiler tubes. The specimens were polished using emery papers of 220, 400, 600 grit sizes and later on 1/0, 2/0, 3/0 and 4/0 grades. These substrates are then grit blasted with alumina powders (Grit 45) prior to the deposition of the coatings by detonation gun thermal spray process.

C. FEEDSTOCK MATERIALS FOR THE COATINGS

Four types of coatings were selected in this study. The Al_2O_3 powder was chosen for D-Gun spray coating with reinforcement of CNT (1.5% wt, 2% wt and 4% wt) on T-11 boiler steel. An additive CNT of different composition with 99.99% purity and Al_2O_3 powder were dry-ball milled in a conventional rotating ball mill with stainless steel balls as a grinding medium for 10 hrs to form a uniform and homogeneous mixture.

D. FORMULATION OF COATINGS

The D-gun spray process was used to apply coatings on the alloys at SVX Powder M Surface Engineering Pvt. Ltd, Greater Noida (UP, India) as shown in Fig.1. Standard spray parameters were designed by the above mentioned firm for depositing the coatings in the present work. All the process parameters, including the spray distance (200mm), were kept constant throughout the coating process. The standard spray parameters as mentioned in the manual of Detonation gun spray system were used for the deposition of the coating as shown in Table 2.

TABLE 1: CHEMICAL COMPOSITION (WT %) OF T-11 BOILER STEEL (ASTM - A199/A450- T-11)

	Standard	Steel	CHEMICAL COMPOSITION								
				С	Si	Mn	P	S	Cr	Ni	Mo
Nominal	ASTM	T-11	min	0.05	0.5	0.3	-	-	1		0.44
	1		max	0.15	1	0.6	0.025	0.025	1.5		0.65
Actual	ASTM	T-11		0.12	0.7	0.44	0.014	0.010	1.22		0.5

E. HOT CORROSION STUDIES IN MOLTEN SALT (NA₂SO₄-60% V₂O₅)

Hot corrosion studies were conducted at 900° C in a silicon carbide tube furnace in laboratory. The

furnace was calibrated to an accuracy of $\pm 5^{\circ}$ C using Platinum/Platinum-13% Rhodium thermocouple fitted with a temperature indicator of Electromek (Model-1551 P, Ambala Cantt, India). The bare as well as the coated specimens were polished down to 1µm alumina wheel cloth polishing to obtain similar condition of reaction before being subjected to corrosion run. The physical dimensions of the specimens were then recorded carefully with digital vernier caliper to evaluate their surface areas. Subsequently, the specimens were washed properly with acetone and dried in hot air to remove the moisture. During experimentation, the prepared specimen was kept in an alumina boat and the weight of boat and specimen was measured. The alumina boats used for the studies were pre-heated at a constant temperature of 1200°C for 12 hours and it was assumed that their weight would remain constant during the course of high temperature cyclic corrosion study.

TABLE 2 SPRAY PARAMETERS OF D-GUN SPRAY	ING
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Variant	D-Gun Spraying
Oxygen flow rate	4800 l/h
Fuel (acetylene) flow rate	1920 l/h
Carrier gas (nitrogen) flow	800 l/h
Spray distance	200 mm
Flame temperature	3900°C
Detonation frequency	3 shots/sec

The prepared specimens were then heated in an oven up to 250°C and a salt mixture of Na₂SO₄-60% V₂O₅ dissolved in distilled water was coated on all the six surfaces of the warm polished specimens with the help of a camel hair brush. Amount of the salt coating was kept in the range of 3.0 -5.0 mg/cm². The salt coated specimens as well as the alumina boats were then dried in the oven for 3 hours at 100°C and weighed before being exposed to hot corrosion tests. Then, the boat containing the specimen was inserted into hot zone of the furnace maintained at a temperature of 900°C. The weight of the boat loaded with the specimen was measured after each cycle during the corrosion run, the spalled scale if any was also considered during the weight change measurements. Holding time in the furnace was one hour in still air followed by

cooling at the ambient temperature for 20 minutes. Following this, weight of the boat along with specimen was measured and this constituted one cycle of the hot corrosion study. Precision weighing Balance Model PEWT-M1 (High scale weighing machine manufacturer, India) having a sensitivity of 10⁻³g was used to conduct the weight change studies. The specimens were subjected to visual observations carefully after the end of each cycle with respect to color or any other physical aspect of the oxide scales being formed. All hot corrosion studies were carried out for 50 cycles i.e. 50 cycles were made for each sample. Corroded samples from hot corrosion were analysed by XRD (BRUKERbinary V3) and SEM/EDAX and the oxide scale which fell into the boat were also analysed by XRD.



Fig.1 D-Gun Thermal Spray Process Used In the Current Research Work

III. EXPERIMENTAL RESULTS

A. BEHAVIOUR IN MOLTEN SALT (NA₂SO₄-60%V₂O₅) ENVIRONMENT AT ELEVATED TEMPERATURE

1) Visual Examination

The macrographs of bare and coated T-11 boiler tube steel after hot corrosion studies in molten salt environment of $Na_2SO_4-60\% V_2O_5$ at high temperature of 900°C for 50 cycles are shown in Fig. 2

Colour of oxide scale formed on the bare alloy T-11 [Fig. 2(a)], after hot corrosion in the molten salt enviorenment(Na_2SO_4 -60% V_2O_5) for 50 cycles

at 900°C was mixture of dark black and light grey scale along with large destruction on surfaces. Large cracks was observed on the corners and edges of specimen. Intially, colour of scale apppeared as brownish. Subsequently, it converted into light black and grey mixture along some cracks on the surface on bare T-11. The scale formation was noticed at the end of 3rd cycle and swelling of which was observed at the end of 14th cycle. Spalling of scale started at the end of 19th cycle; however, the scale formation continued till 50th cycle.

Whereas Al_2O_3 coating alloy [Fig. 2(b)] show initially white colour. As the hot corrosion went on, the colour of coating turned into light golden colour on one side of surface while the other side shows coating of light gold colour on maximum surface and small part visualize black colour along with small cracks after 50 cycles. The scale formation observed at the end of 7th cycle and swelling of corners was observed at the end of 20th cycle.The coating suffered spallation along the corners and edges of the specimen. After 50 cycles, specimen's corners and edges found destructed and swelled very largely.

Alloy with coating $Al_2O_3+1.5\%$ CNT [(Fig. 2(c)] also show initially white colour. At the end of 50 cycles of hot corrosion in molten salt environment ($Na_2SO_4-60\%V_2O_5$) at 900°C, colour of oxide appeared on two sides of surface become brownish with grey colour background. Some white and gold colour mixture also appeared on the surface. Small spallation of coating suffered along the corners and edges of the specimen.

 $Al_2O_3+2\%$ CNT [Fig. 2(d)] coating posses white colour initialy. As the hot corrosion in molten salt ($Na_2SO_4-60\%V_2O_5$) went on, after 17th cycles spallation of coating start and the colour of oxide scale turned into grey colour appeared with brownish backgrounds. After 50 cycles, small grey colour appearance remained on both sides of surface with brownish colour background. Here, coating suffered spallation along the corners and edges of specimen with brownish colour. But in case of $Al_2O_3+4\%$ CNT [Fig. 2(e)] spallation of coating start after 23rd cycles and also colour of oxide scale turned into small light grey mixture with brownish background. Collapsing of coating appeared on the surface after 50 cycle of hot corrosion in molten salt environment (Na₂SO₄-60%V₂O₅) along both sides. All the edges and corners shows the mixture of brownish and light grey colour.

2) Weight change measurements

Wieght gain/unit area(mg/cm²) versus number of cycles plot for the bare as well as D-gun sprayed coated boiler tube steel T-11 alloys after 50 cycles of hot corrosion in molten salt environment (Na₂SO₄-60% V₂O₅) at 900°C are shown in Fig. 3. Uncoated T-11 shows the maximum weight gain among all the alloys. The coated Al₂O₃+1.5% CNT steel alloy show lower weight gain among all the coated specimens and much resisitance to given aggressive environment than uncoated alloy at 900°C. Al₂O₃ coated T-11 alloy indicate higher weight gain among all the T11 coated alloys in given environment. The weight gain by coated T-11 (Al₂O₃), after 50 cycles is nearly 62.3% less than that of uncoated T-11.



Fig. 2 Macrograph Of Bare And Coated T-11 Specimen Subjected To Hot Corrosion in Na₂SO₄-60%V₂O₅ at 900°C For 50 Cycles;
(a) Uncoated T-11 Boiler steel; (b) Al₂O₃ Coating; (c) (Al₂O₃+1.5%CNT) Coating; (d) (Al₂O₃+2%CNT) Coating (e)(Al₂O₃+4%CNT) Coating



Fig. 3 Weight gain plot for uncoated and coated T-11 boiler tube steel subjected to hot corrosion in molten salt (Na₂SO₄-60% V₂O₅) environment at 900° C for 50 cycles



Fig. 4 Weight gain square plot for uncoated and coated T-11 boiler tube steel subjected to hot corrosion in molten salt $(Na_2SO_4-60\%V_2O_5)$ environment at 900°C for 50 cycles

It is found that maximum 91.8% saving in weight gain for $Al_2O_3 + 1.5\%$ CNT coated, 87.04% saving in weight gain for $Al_2O_3 + 2\%$ CNT coated and 87.3% weight saving for $Al_2O_3 + 4\%$ CNT coated T-11 in comparion with uncoated T-11.

Table 3 Parabolic Rate Constant (Kp) Values For Bare And D-Gun Sprayed Coated Grade T-11 Boiler Steel Subjected To Hot Corrosion in Na₂SO₄-60% V₂O₂ Environment for 50 Cycles At 900°C

T-11	Kp $(g^2 cm^{-4} s^{-1})$
Uncoated	7133.34×10 ⁻¹⁰
Al ₂ O ₃	1348.94×10 ⁻¹⁰
Al ₂ O ₃ +1.5%CNT	51.46×10 ⁻¹⁰
Al ₂ O ₃ +2%CNT	147.75×10 ⁻¹⁰
Al ₂ O ₃ +4%CNT	135.01×10 ⁻¹⁰

Further, the weight gain square (mg^2/cm^4) data plotted as a function of time (number of cycles) is shown in Fig. 4 to establish the rate law for the hot corrosion. All the CNT reinforced Al₂O₃ coated T-11 followed a parabolic rate law of oxidation at 900°C (Fig. 4). The values of the parabolic rate constant (kp) are shown in Table 3. It is inferred that the kp values for the coated T-11 steel are less than the bare T-11.

B. X-RAY DIFFRACTION ANALYSIS

The samples after hot corrosion were removed from boat and their oxide scales which were separated from surface were also removed. Then they were analysed separately by XRD and after that only oxidised sample were analysed by SEM /EDAX. The results of XRD analysis contained graph indicating peak values (i.e. d values) which were used to identify various phases with the help of inorganic X-ray Diffraction data card from Powder diffraction file of JCPDS. Help of software named Philips X'pert High score and Eva was also taken for finding out compounds at respective peaks.

1) XRD result for bare and Coated T-11 samples

The various phases identified from X-ray diffraction patterns on un-coated and D-gun sprayed coated boiler tube steel alloys of T-11 after hot corrosion in molten salt environment (Na₂SO₄-60%V₂O₅) at 900°C for 50 cycles are shown in Fig 5. As obvious from the analysis, the uncoated T-11 steel has indicated the formation of mainly iron oxide (Fe₂O₃) [Fig. 5(a)]. The Al₂O₃ coated T-11 boiler steel shows the formation of mainly Alluminium oxide (Al₂O₃). Also the main phase identified for the coated Al₂O₃+1.5%CNT,

 $Al_2O_3+2\%$ CNT and $Al_2O_3+4\%$ CNT, T-11 alloys are Al_2O_3 [(Fig. 5(b-e)].



Fig. 5 X-ray diffraction profiles of the (a) uncoated T-11 and (b) Al₂O₃ coated T-11 boiler steel subjected to hot corrosion in molten salt (Na₂SO₄-60% V₂O₅) environment at 900° Cfor 50 cycles (contd.)



Fig . 5 X-ray diffraction profiles of the (c) Al₂O₃+1.5% CNT Coated T-11 and (d) Al₂O₃+2% CNT Coated T-11 (e) Al₂O₃+4% CNT Coated T-11 boiler steel subjected to hot corrosion in molten salt (Na₂SO₄-60% V₂O₅) environment at 900°C for 50 cycles

C. FE-SEM/EDS ANALYSIS OF SURFACE MORPHOLOGY

Fe-SEM micrographs with EDS spectrum showing the surface morphology of uncoated T-11 boiler tube steel sample as well as four types of coated T-11 boiler tube steel samples i.e Al_2O_3 , $Al_2O_3+1.5\%$ CNT, $Al_2O_3+2\%$ CNT and $Al_2O_3+4\%$ CNT after cyclic hot corrosion in molten salt environment (Na₂SO₄-60%V₂O₅) for 50 cycles at 900°C as show in Fig. 6, 7, 8, 9, 10 respectively.

Micrograph of corroded uncoated T-11 reveals the oxide scale developed on the surface is massive and with large cracks. The oxide scale produced is of white contrast and dark black region which is irregular in size and non-uniformly distributed. The EDS analysis of bare corroded T-11 steel alloy at 900°C reveals the scale rich in Fe and O, which indicates the possibility of formation of iron oxide. Carbon (C) and small amount of Mo element are also found at point of analysis, thereby predicting the possibility of formation of CO_2 and Mo_2O_3 . Besides, EDS analysis of bare corroded T-11 steel alloy also show poor scales in Na₂O, SiO₂, P₂O₅, SO_3 , V_2O_5 , Cr_2O_3 and MnO. On the other side, Micrograph of corroded Al_2O_3 and $Al_2O_3+1.5\%$ CNT coated T-11 steel alloys are with small cracks and appears to be dense and continuous. EDS analysis revealed the scale rich in Al and O, which shows the possibility of Al₂O₃. Similarly, the scale formed coated Al₂O₃+2%CNT on and Al₂O₃+4%CNT, is dense and regular like other coatings and surfaces show some cracks..

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Fig. 6 SEM micrograph with EDS spectrum of the uncoated T-11 boiler tube steel sample showing surface morphology after hot corrosion in molten salt (Na₂SO₄-60% V₂O₅) environment for 50 cycles at 900°C



Fig. 8 SEM micrograph with EDS spectrum of the Al₂O₃+1.5% CNT coated T-11 boiler tube steel sample showing surface morphology after hot corrosion in molten salt (Na₂SO₄-60% V₂O₅) environment for 50 cycles at 900°C



Fig. 7 SEM micrograph with EDS spectrum of the Al_2O_3 coated T-11 boiler tube steel sample showing surface morphology after hot corrosion in molten salt (Na2SO4-60% V2O5) environment for 50 cycles at 900°C

The EDS analysis clearly shows the formation of Al and O as predominant elements in both the cases, which shows the possibility of Al_2O_3 like other coatings sample studied above



Fig. 9 SEM micrograph with EDS spectrum of the $Al_2O_3+2\%$ CNT coated T-11 boiler tube steel sample showing surface morphology after hot corrosion in molten salt ($Na_2SO_4-60\%$ V₂O₅) environment for 50 cycles at 900°C



Fig. 10 SEM micrograph with EDS spectrum of the Al₂O₃+4% CNT coated T-11 boiler tube steel sample showing surface morphology after hot corrosion in molten salt (Na₂SO₄-60% V₂O₅) environment for 50 cycles at 900°C

IV. DISCUSSION

During hot corrosion surface study of CNT reinforced Al₂O₃ coating of T-11 boiler steel in molten salt(Na₂SO₄-60%V₂O₅) at 900°C for 50 cycles resemble that corrosion resistance property of CNT reinforced coated T-11 specimen is better than uncoated T-11 boiler steel as weight gained by bare T-11 is much more throughout the 50 cycles than the all four types of coated T-11 samples Al₂O₃+1.5%CNT, Al₂O₃+2%CNT. $(Al_2O_3,$ Al₂O₃+4%CNT). The rapid increase in the weight gain during the initial period of exposure to Na₂SO₄-60%V₂O₅ (molten salt environment) at 900°C may be due to the rapid diffusion of oxygen through the molten salt layer. A study proposed by [21] that in the temperature range of 900°C, the Na_2SO_4 and V_2O_5 will combine to form $NaVO_3$ (having a melting point of 610° C) as follows:

 $Na_2SO_4 + V_2O_5 = 2NaVO_3 + SO_2 + \frac{1}{2}O_2$ (1)

This $NaVO_3$ acts as a catalyst and also serves as an oxygen carrier to the base alloy through the open pores present on the surface, which will lead to the rapid oxidation of the base elements of the substrate to form a protective oxide scale. Hence, an increase in the weight gain of the alloys occurs in the early stages of the hot corrosion. During the subsequent cycles, the formations of oxides have blocked the diffusion of corrosive species by covering the pores and the splat boundaries. The rapid increase in the weight gain during the initial period was also reported by [22]-[25] during studies on the hot corrosion of alloys.

The coated $Al_2O_3+1.5\%$ CNT steel alloy show much lower weight gain among all the coated specimens and Al_2O_3 coated T-11 alloy indicate higher weight gain among all the T-11 coated alloys in given enviornment. The weight gain by coated T-11 (Al_2O_3), after 50 cycles is nearly 62.3% less than that of uncoated T-11. It is found that maximum 91.8% saving in weight gain for $Al_2O_3 + 1.5\%$ CNT coated, 87.04% saving in weight gain for Al_2O_3 +2% CNT coated and 87.3% weight saving for $Al_2O_3 + 4\%$ CNT coated T-11 in comparion with uncoated T-11.. The oxidation rate (total weight gain values after 50 cycles) of bare and coated T-11 boiler steel follows the sequence as given below:

Due to different value of thermal coefficient, cracks in the oxide scale (Fig.6) and spalling of coating might be occurred during cyclic testing of coating and substrate as reported by [11].

Various elements have different value of thermal coefficient of expansion. Due to this, much stress produced which will be responsible for more cracks and spalling and corrosive gas can easily penetrate through these cracks which will lead to significant grain boundary corrosion attack [26]-[29]. Also the more weight gain value in case of bare T-11 steel attribute to the existence of molybdenum in the substrate steel [9]. The Al₂O₃ coating layer on surface of substrate suppresses the diffusion of oxygen from the outer environment and hence plays a significant role in the resistance [30]-[32]. The Al₂O₃ layer also suppresses the surface from degradation. The D-Gun sprayed CNT reinforced

 Al_2O_3 coated T-11 and Grade A-1 boiler steels when subjected to hot corrosion in Na_2SO_4 -60% V_2O_5 molten salt environment at 900°C develop a scale mainly consisting an aluminum oxide, which are supported by XRD [Fig. 5(b-e)], EDAX (Fig. 6-10). The aluminum oxide is very protective as reported by [33 and 34]

Hence, the existence of large amount of Al_2O_3 in Na_2SO_4 -60% V_2O_5 molten salt environment will suppress the further oxidation of coating surface supportively and this is main lead to protection of surface from degradation.

V. CONCLUSIONS

The hot corrosion behaviors of uncoated and CNT reinforced Al_2O_3 coated T-11 boiler steel have been investigated in Na_2SO_4 -60% V_2O_5 environment at 900 °C for 50 cycles. The following conclusions are made:

- 1. The D-Gun sprayed CNT reinforced Al_2O_3 coatings on T-11 boiler steel have developed a protective scale mainly consisting an aluminum oxide.
- 2. The CNT reinforced Al₂O₃ coated T-11 steel have shown resistance to hot corrosion as the overall weight gain is less than as compared to the uncoated T-11 boiler steel.
- 3. The oxidation rate (total weight gain values after 50 cycles) of the uncoated and CNT reinforced Al_2O_3 coated T-11 boiler steel follows the sequence as given below:

- 4. Al₂O₃ coated T-11 alloy indicate higher weight gain among all the T-11 coated alloy in given enviornment.
- 5. Uncoated T-11 boiler steel posses highest weight gain. Some cracks are also observed in both cases.
- Al₂O₃+1.5%CNT coating on T-11 boiler steel boiler provide a much corrosion resistance in Na₂SO₄-60%V₂O₅ (molten salt environment) at high temperature.

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Amandeep Singh graduated with Mechanical Engineering and post graduated with Thermal Engineering from Punjab Technical University, Jalandhar, Punjab, India. Currently he is a research Scholar of Singhania University, Rajasthan, India, From 2015 to present he is doing his research work under the supervision of Dr Vikas Chawla and Dr. Neelkanth Grover.