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Oxidation performance of CNT-reinforced Cr_3C_2-NiCr coatings sprayed by HVOF method

The high-temperature oxidation performance of thermal sprayed nanocomposite coatings is discussed in the present study. The three combinations 3, 5, and 7% wt. of CNT reinforced Cr_3C_2-25% NiC coatings sprayed by HVOF on SAE-213 T12 boiler tube alloy steel working at 600^0C in silicon tubular furnace following 1hour heating and 20 minutes cooling in atmospheric temperature for 50 cycles. Thermally developed oxidized layer formation influences in structural variations on surface morphology, phases of elements of uncoated and CNT coated sample were analysed using SEM/EDS and XRD techniques. The thermogravimetric approach was used to estimate the kinetics of oxidation on all samples the weight gain measurement has been studied. The weight accumulation of bare substrate has a higher rate of oxidation than the CNT coated samples. The weight gain of the samples oxidation mechanism generally represents parabolic in nature. The oxidation growth rate minimizes when CNT reinforced in coatings and oxidizing scale deposited on CNT coated surface significantly lower than uncoated samples. The developed Cr_2O_3, NiO, and Fe_2O_3 oxidized layers were provided intensifying oxidation resistance.

Keywords : High velocity oxy-fuel (HVOF), carbon nanotube (CNT), oxidation, boiler tube alloy steel, thermo-gravimetric analysis, elevated temperature.

1.0 Introduction

The composite coatings need to develop resulting enhanced the performance, energy, and efficiency of power plant components. The CNT reinforced coatings results in improving thermal conductivity, porosity level in the microstructure reduces with increase in the CNTs added in coatings (Thakare J.G et al., 2017). These types of coatings applied on the supercritical boiler tube alloy steel of the steam producing plants to hindering erosion-corrosion complexities due to low-grade fuels were containing chlorides, sulphides, and thermodynamic concerns such as metal, gas temperature, gas thermal variations when fuel-burning by inhomogeneous waste and soot blowing affects thermal fluctuations causes corrosive oxides penetrating through the substrate material. The CNT reinforced coatings improve the layer properties leads to enhance hot-corrosion and reduction in weight gain than alumina and silicon-coated samples (Zainab Jalal et al., 2021).

This elevated temperature results in harmful effects on the attributes of the bare alloy steels. The HVOF thermal deposition process has been introduced to become the right option to spray well adhered, dense coatings, and with better mechanical functional properties. The WC-CO/NiCrAlY coatings has less oxidation rate compared to the uncoated sample. The protective layer Ni and Cr acts as a barrier against oxidizing species penetrating through the coating and WC-CrC-Ni coatings enhanced in oxidation resistance property than uncoated samples (Somusundaram et al., 2014; Somusundaram et al., 2015).

The HVOF thermal spray is a more extensive method for a high range of materials are now being focused on coatings achieved better protection against high oxidation situations in the HVOF process than the Tribomet^{TM} process (Raphael Goti et al., 2014). The 4% wt. of CNT addition in Al_2O_3 coating plays important role in oxidation resistance, reduces weight gain than the remaining % of CNT additions in Al_2O_3 coating (Goyal et al., 2018). The Ni-50Cr coatings have an oxidation resistant layer formed on the surface and decrease weight gain than Ni-20Cr sprayed by cold spray process deposited on T22 boiler steel (Bala et al., 2009). The YSZ coatings 0,5,10, and 15% wt. of added in Si-Mo coatings, the modified 10% wt. of YSZ in coatings performed better oxidation, achieved lower weight gain compared to other coatings due to protective layers formed sprayed by two-step spraying process (Cai et al., 2018). Reviewed on CoNiCrAlY-YSZ coatings on Inconel 617 specimen the coating has more pores in coated structure tend to oxidation resistance decreases. The stellite-6 coating deposited on titanium alloy have less oxidation resistance than cobalt superalloy, and special steel (MDN-121) working under hot-conditions used in gas turbine blades, these coatings sprayed by HVOF (Hatami, N et al., 2018;

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Jegadeeswaran et al., 2013).

The Fe-based 5V-10Nb, and 5V-10Co specimens have better oxidation, erosion resistance than specimen containing vanadium without Co, and CoNiCrAlYSi coatings sprayed by HVOF process rich chromium layer beneath the outer aluminum layers formed to act against penetrating oxides scales to improve the oxidation and corrosion resistance (Kusumoto et al., 2015; Mohammadi et al., 2016).

The effect of sandblasting on MCrAlY coatings aluminum oxide layer deposited on the surface was observed, isothermal oxidation rate decreases when increased amount of alumina compared to as-sprayed coatings, and the NiCrFeSiB coatings exposed under cyclic hot conditions protective layers Cr_2O_3, SiO_2 formed to act against oxidation results in less weight gain obtained due to kinetics of oxidation rate decreases, and the NiCrAlY coatings deposited on superni 76 superalloys Al_2O_3 layer formed between coating and oxide scale interface was observed during exposure to cyclic hot situations.

The Ni_3Ti and $Ni_3Ti+Cr_3C_2$-NiCr coatings sprayed on AISI stainless steel exposed at high-temperature situations Nio and Cr_2O_3 protective layer forming to stabilize against oxidations (Naeimi et al., 2016; Ramesh et al., 2011; Rana et al., 2014; Reddy et al., 2018). The Cr_3C_2-50%NiCMoNb coatings sprayed by HVOF has better performing property under high-temperature condition play important role act against inner carbide oxidation than other coatings (Sarka Houdkova et al., 2017). A. Shamsipoor et al. reported on YSZ coatings on the Cr_2AlC substrate have better oxidation resistance property at elevated temperature than CoNiCrAlY substrate coated sample, reduces abrasion resistance in all samples, these coatings sprayed by the atmospheric plasma spray process (A. Shamsipoor et al., 2020).

The graphite grades materials are GLM50, GLM, and SPSS behaves more sensitive in temperature environment and GLM50 sample has more degree of graphitization leads to excellent oxidation resistance than GLM, SPSS graphite material (S. Ariharan et al., 2021). The research work carried out on CoNiCrAlY coatings performed at 1100 ^{0}C conditions the internal oxidation concave-shaped oxides were observed in the coated structures (H. Chen et al., 2020). The CoNiCrAlY coatings sprayed by CGDS and HVOF method produced same microstructure with low porosity and low oxide were observed leads to minimum oxidation rate deposited compared to the APS method (P. Richer et al., 2010). Revealed on TIG welded GrA1 steel less amount of oxides formed than SMAW and the presence of Fe_2O_3 oxide was found analyzed by XRD and EDS techniques (Kumar. R et al., 2019). The shielded oxides are NiO, Fe_2O_3 and Cr_2O_3 are developed working under air oxidation at 900^{0}C observed by XRD and EDS analysis and the Superni 750 alloy has more weight gain than Superni 76, Superfer 800 alloys (R.A Mahesh et al., 2008). The Ni-20Cr coated samples enhanced in hardness, density compared to bare substrates and reduction in weight gain kinetics on coated samples due to NiO, Cr_2O_3 and $NiCr_2O_4$ layers developed act to against corrosive species into the substrate (Kaur et al., 2015). The oxidation studies was carried out at 800, 900, and 1000ºC on Ni(Cr)-TiB_2 coatings sprayed by the HVOF method, thin adherent oxides formed on the coating surface at 800ºC, and when the oxidation was studied at a temperature of 1000ºC thick oxides was developed leading to detached from the coating (Behnam Lotfi et al., 2010).

The Superni75 and Superni718 the oxide layers NiO, Cr_2O_3, and $NiCr_2O_4$ are developed tend to better oxidation and corrosion resistance than Superfer 800H alloy due to affected from non-protective oxide layers (Subhash Kamal et al., 2010). The Ni-5Al coating coated sample reduction in weight accumaltion during air oxidation studies at 900ºC and Cr_2O_3 layer formed to protect from oxides at coating and substrate interface. (Saladi.S et al., 2014). Nano Y_2O_3 uniformly distributed in ferritic alloys leads to development of infinite protective and adhered oxides are Cr and Fe leads to increased oxidation resistance (Meharwal.A et al., 2021). Increased cobalt content the Mo crystal structure effected on adherent loss in refining atoms and decreased weight loss at different temperature (Bao.Z et al., 2021). IN 625 coating sprayed by HVOF on T22 subtrate, NiO, Cr_2O_3 and $NiCr_2O_4$ oxides exhibited excellent oxidation resistace compared to uncoated sample (Verma and Kaushal, 2020). The Ni-20Cr coatings studied at 900ºC has more improvement in oxidation compared to WC-12Co coatings (Kumar. A. M et al., 2018).

The present research work is investigated on oxidation behaviour of uncoated and CNT reinforced Cr_3C_2-25%NiC coatings sprayed on T12 boiler alloy steel substrate, performed in silicon tube furnace at 600ºC. The cyclic oxidation 1h heating, 20min cooling in room temperature situations the kinetics of weight gain measurements and changes in microstructural attributes of the coatings were examined. After review of previous and the existing literature the investigation was not carried out on this type of nano-composite coatings combination which were adopted for T12 boiler tube alloy steel.

2.0 Experimentation

2.1 SELECTION OF SUBSTRATE MATERIAL

The ASTM SAE-213 T12 boiler alloy steel choosen for the present research study was procured from Karnataka power corporation limited, Government of Karnataka, India. The substrate which is generally uses supercritical boiler tube material for incinerated elevated-temperature regions of coal-fired boilers in steam generating thermal power plants. The chemical composition of the substrate is shown in Table 1 and physical dimensions of the samples were cut from supercritical boiler tube 25mm × 25mm × 4mm. The samples were polished with silicon carbide abrasive paper down to 200 grit. Finally the samples blasted with alumina oxide powder using grit size of 16µm at a pressure of 7 Kg-cm^{-2}, stand-off

TABLE 1: CHEMICAL COMPOSITION OF BOILER TUBE ALLOY STEEL

ASTM SA213 T-12 Steel	Chemical composition, wt.%									
	C	Ni	Cr	S	P	Si	Mo	V	Mn	Fe
	0.07	0.68	1.35	2.172	0.020	0.64	1.039	0.43	0.455	Balance.

TABLE 2: COATING POWDERS MIXED WITH CNTS IN DIFFERENT (WEIGHT %) PROPORTIONS

	Coating powder Cr_3C_2–25% NiCr in wt.%	CNT in wt.%	Mix proportion in gm	
			Cr_3C_2-25%NiCr	CNT
1	97	3	970	30
2	95	5	950	50
3	93	7	930	70

distance for alumina blasting 150 mm, and cleaned with acetone to remove dust particles before deposition of the coatings.

2.2 COATING POWDER FORMULATION AND CHARACTERIZATION

The nano-composite coatings are developed using Cr_3C_2-25%NiC mixed with three varying 3, 5, 7% weight of CNT reinforced in coating powder. The addition of 3 wt.% of CNT mix in coating powder, 3g of CNT were mixed with 97 g of Cr_3C_2-25%NiCr. Furthermore, continued remaining varying combinations prepared are shown in Table 2. These combinations mixed and rolled for atleast 4h continuously with a speed of 200 rev min^{-1} using planetary ball rolling mill, rollers and jar are made up of zirconium, rollers have 5mm diameter.

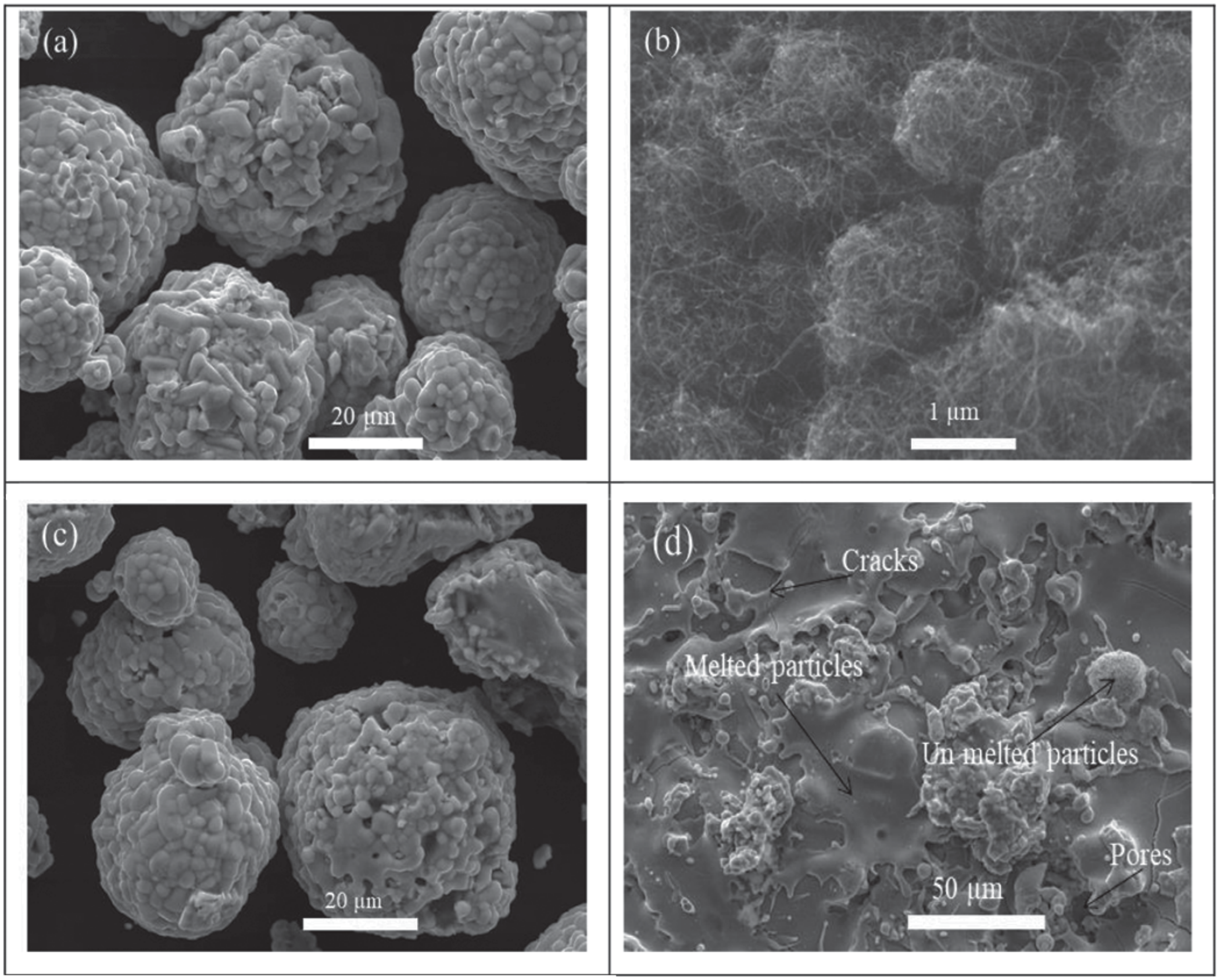

Fig.1(a-c): SEM images of coating powders and Fig.1(d) Coated sample; (a) Cr_3C_2–25%NiCr: (b) Pure CNT: (c) 7% CNT in Cr_3C_2–25%NiCr: (d) 7% wt. of CNT coated sample

The SEM images of Cr_3C_2–25% NiCr powder particles are in spherical, irregular in shape with granularity of a powder as shown in Fig.1(a), and CNT powder has long thin cylinders of carbon hexagonal in shape with an average dimensions of 20±5nm depicted in Fig.1(b). The 7 wt.% of CNTs are uniformly dispersed in the Cr_3C_2-25%NiCr coating powder, the CNT have nano sizes spread equally in between the coating particles observed in Fig.1(c). The 7% wt. of CNT coated sample has melted, partially melted particles along with some pores and hair line crackes were observed in SEM image after oxidation studies depicted in Fig.1(d).

2.3 Coating development

The composite coating powders sprayed by high velocity oxy-fuel (HVOF) thermal spray process on ASTM SA-213 T12 boiler tube alloy steel substrate, using METCO gun DJ-2600 equipment, and nozzle DTH-2603 in Spraymet Industries Ltd, Bangalore, India. The parameters are defined during the thermal spraying process the oxygen and hydrogen flow rate is 250 L/min, 60 L/min and pressure 10 bar, 7 bar, and air flow rate 1000 LPM, pressure 5.5 bar, the coatings powder feed rate 82 g/min, spraying distance 200 mm kept constant during deposition and after deposition the specimens were cooled with compressed air jets.

In this method the product of oxygen and hydrogen burns in combustion region; then expands through nozzle, the gas velocities may be supersonic, nano-composite coating powder fed axially, the coating particles heated and accelerated through nozzle, the coatings are in a molten or semi-molten state to impinge on substrate get distorted quickly crystallize to grow splat. The thickness of coating measured along cross-section of the coated specimens in range of 280-290μm, thickness of the coatings was measured by backscattered electron image..

2.4 Oxidation studies

The cyclic oxidation examinations were carried out in experimental silicon tube furnace at high-temperature environmentof 600°C. The specimen size width, height, and thickness are measured precisely with a digital vernier-caliper to estimate surface areas. Accordingly the samples were cleaned thoroughly by acetone and to remove moisture content dried by hot-air. The ceramic boat including samples were pre-heated at a isothermal-temperature of 800°C for 4h and it was concluded that weight would neutralize while conducted at elevated-temperature for oxidation study. The uncoated and coated specimens were weighed with ceramic boat and held in elevated-temperature zone of the furnace at 600°C. After 1h heating the specimens were taken out from the silicon tube furnace including ceramic boat and 20 min cooling in air it reached to ambient temperature and the specimens including ceramic boat again weighed this constitute one cycle of oxidation study. An electronic balance model CB-120 (Contech Mumbai India) with a sensitivity 10^{-3} was used to record weight gain studies. Visual examinations were also done after completion of each cycle concerned the physical appearance of oxide scale formation on surface, lustor, colour, adherence/spallation tendency were observed. The uncoated and coated samples were studied to cyclic oxidation under hot-zone situation for 50 cycles with weight gain being recorded after completion of each cycle. The weight gain plots were found against specific weight gain of the specimens and 50 number of cycles was conducted in boiler hot-condition environment. The oxidized specimens were expressed kinetic oxidation in weight gain measurement, and oxidized surfaces, sectioned surface of the backscattered image examined by SEM/EDS, and XRD techniques.

3 Results and discussions

3.1 Visual examination

The surface morphology examined on all oxidized samples after oxidation in air at 600°C for 50 cycles shown in Fig.2(a). Dark colour observed on uncoated surface after completion of third cycle and then silver dots shiny dark-grey colour was observed after fifteenth cycle, and then after 50th cycle black, light purple colour appeared on the surface. The CNT reinforce coated samples colour turns into light brown, purple, and blue colour depicted in Fig.2(b-d) respectively. These samples have no spallation on the surface of the coating.

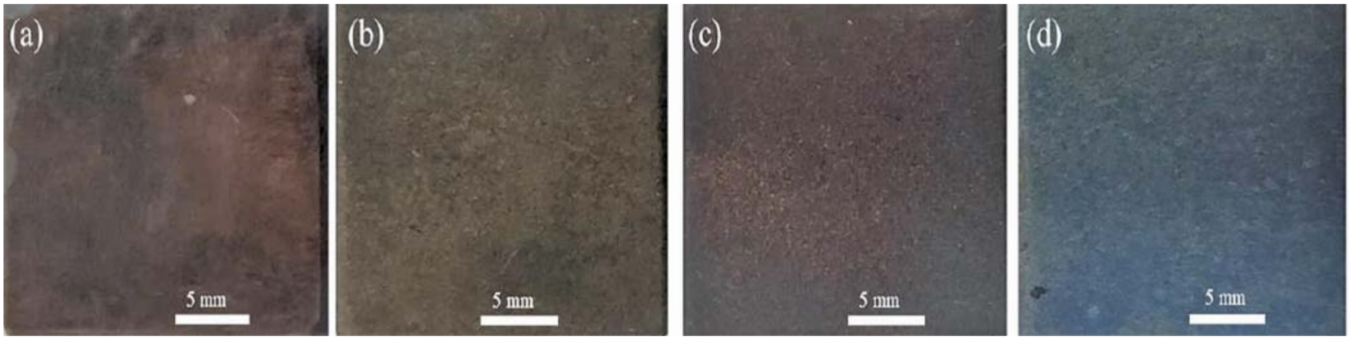

Fig.2(a-d): Macrographs of all samples after oxidation at 600°C (a) Uncoated sample: (b) 3% CNT: (c) 5% CNT : and (d) 7% CNT coated sample

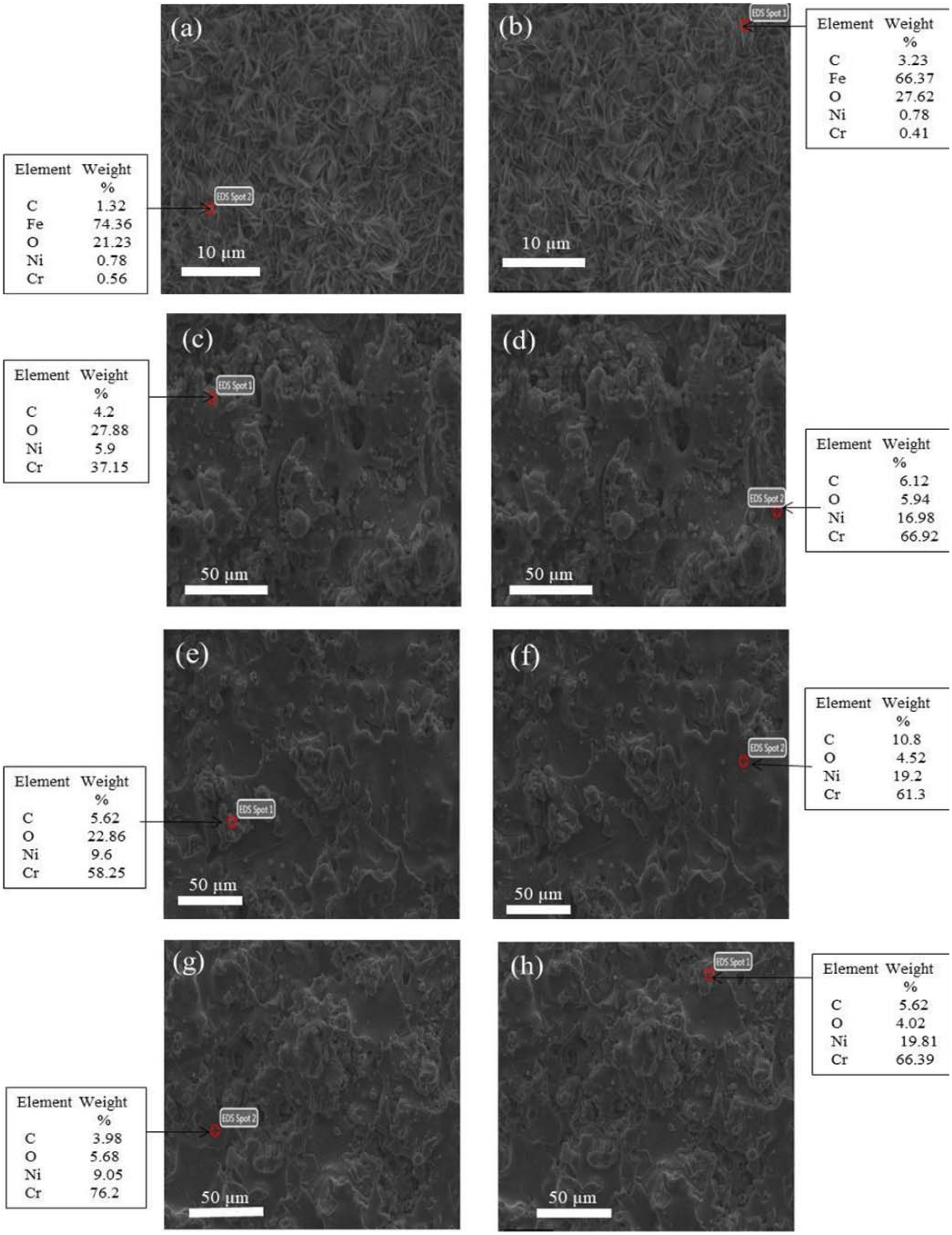

Fig.3(a-h): SEM/EDS images of uncoated and coated samples after exposed to oxidation at 600°C: (a-b) Uncoated sample: (c-d) 3% CNT: (e-f) 5% CNT: (g-h)7% CNT coated samples

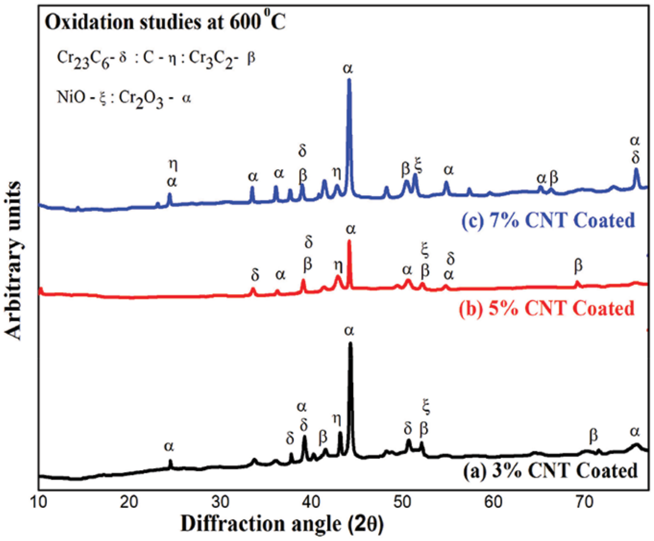

Fig.4 XRD spectrum of uncoated sample after cyclic oxidation test at 600°C

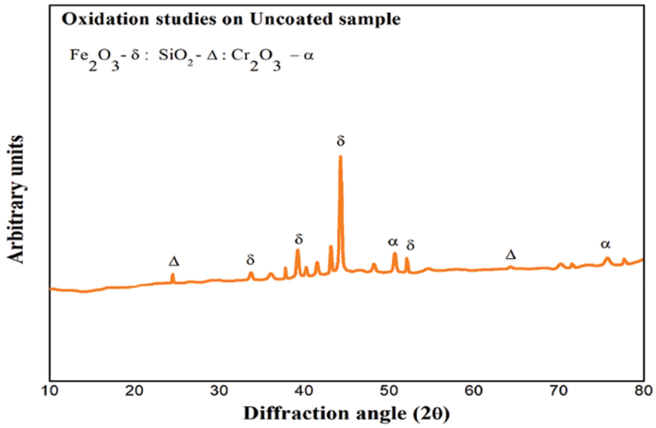

Fig.5 XRD spectrum of all CNT coated samples after cyclic oxidation test at 600°C

3.2 SEM/EDS Analysis

The SEM/EDS analysis was carried out after exposed to hot condition under cyclic oxidation in air at 600°C. The surface morphology and elemental compositions was examined for all the samples shown in Fig.3(a-h). The oxidation studies were conducted up to 50 cycles, oriented whisker or fibre like structure on the surface were observed in Fig.3(a-b). The EDS spectrum at selected points on the substrate rich in FeO content and Cr, C and Ni are in meagre amount. In coated samples the presence of Cr, Ni and O are in major amount and C containing little amount analyzed by EDS techniques depicted in Fig.2(a-h).

3.3 XRD Analysis after Oxidation

The XRD analysis of uncoated and coated samples performed under cyclic oxidation in air at 600°C for 50 cycles elemental phases were represented in Figs.4 and 5. The formation of oxide scales are Fe_2O_3 in major phase, SiO_2 and Cr_2O_3 are in minor phases identified on uncoated substrate sample represented in Fig.4. In coated samples the presence of oxides are Cr_3C_2, C are in major substance and these phases act as a barrier against oxides to the substrate. The Cr_2O_3, NiCr and NiO oxidised products are in meager amount were developed at higher temperature oxidation environment were analysed by XRD spectrums depicted in Fig.5.

3.4 Cross-sectional analysis

SEM Back Scattered Electon (BSE) images and elemental composition were analysed by SEM/EDS across the cross-sectin at different points for uncoated and coated samples as shown in Fig.6(a-d). SEM images of oxidized surface on uncoated sample represents black dots, cracks or distortions of elements due to oxidized products penetrating into the substrate. The presence of Fe_2O_3 phase formed in vital amount of scale on top surface observed in Fig.6(a). The presence of elements such as Fe chief constituent Mo, Ni, C and Mn are in meagre amount. In case of CNT coated samples, the coatings has dense, amorphous surface morphology, well adhered and interlocked with the substrate, along with Cr, Ni, O and C elements are confirmed, these elements dispersed equally in the coatings. The presence of carbon in the coated microstructure, which is proved that presence of CNTs in the coated matrix analysed by EDS techniques.

3.5 Weight gain kinetics

The weight gain curves for oxidised specimens after oxidation in air at 600°C for 50 cycles are shown in Fig.7(a-b). It is evident from the Fig.7(a) the T12 substrate material has maximum weight gain compared to coated samples. The wt.% of CNT increases in coated samples, weight gain rate decreases when samples exposed in hot-zone environment. The parabolic rate constant (K_p) is determined from the slope of the linear regression fitted line (weight gain/area)^2 versus number of cycles, and it is shown in Table 3. The nature of fit, a parabolic rate law for oxidation measures, is also depicted in Fig.7(b). It is suggested that all the coatings on the T12 alloy steel have relatively matched the parabolic rate law.

The oxidation rate increased comparatively higher during initial cycles, which might be attributed to the formation of cracks in the top scale. The value of parabolic rate constant K_p value was found to be 0.08236 $g^2cm^{-4}s^{-1}$ and total weight gain after 50 cycles was 32.9191 mg cm^{-2}, the appearance of fragile non-protective Fe_2O_3 scale having cracks recognized by microstructural analysis on the uncoated corroded substrate surface. The addition of CNT in coatings is able to act as a barrier against corrosive species into the T12 alloy steel substrate during oxidation at 600°C was observed from weight gain curves. CNT coated samples free from micro-holes, hairline cracks, intact, and without spallation when exposed during 50 cycles. The cumulative weight gain/unit area for 3, 5, and 7% wt. of CNT coated T12 steel after air oxidation at 600°C for 50 cycles was found to be 23.1461 mg cm^{-2}, 11.6556 mg cm^{-2}, and 4.2028 mg cm^{-2} respectively and were able to reduce cumulative weight gain of T12 steel by 29.68, 64.59, and 87.23% respectively.

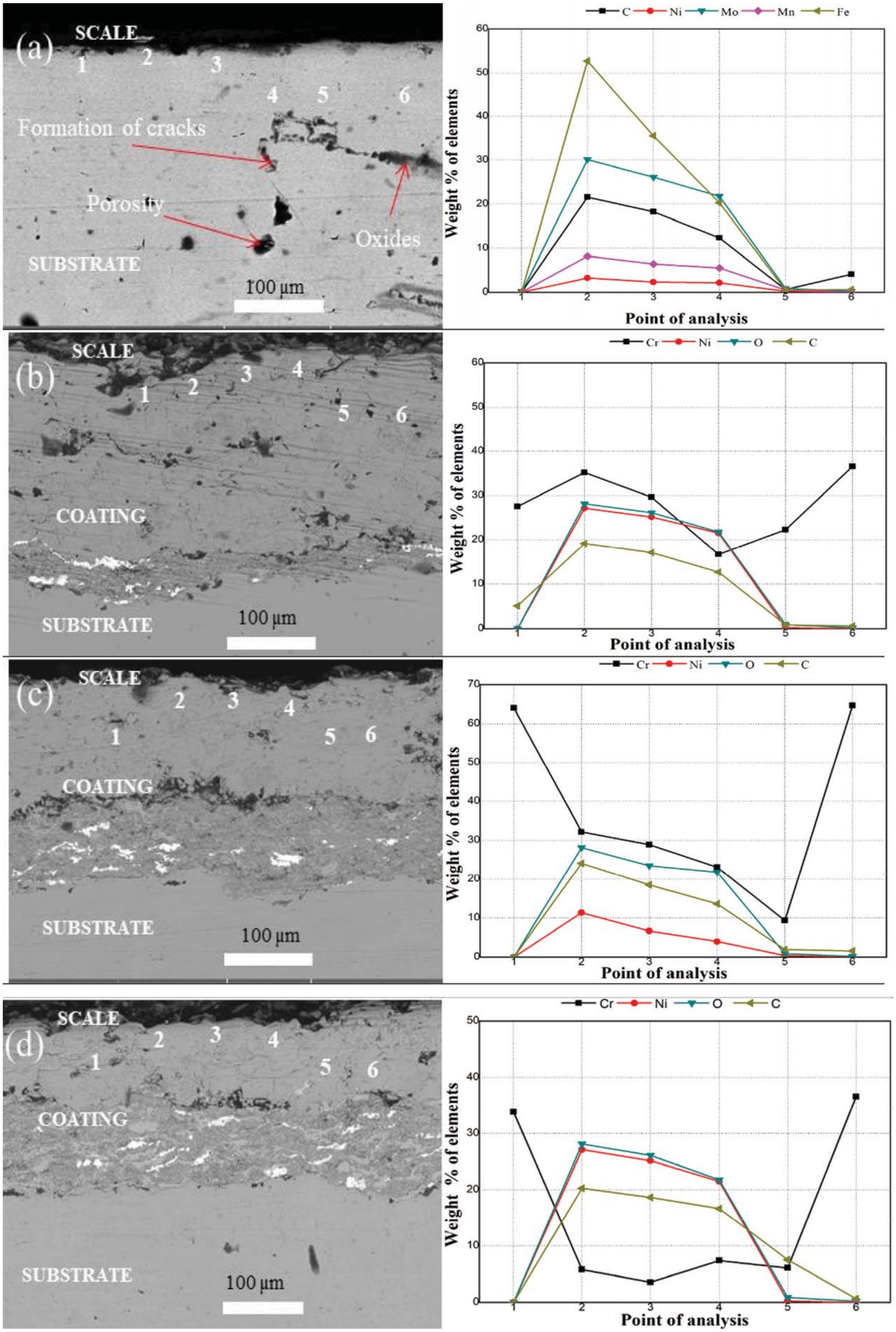

Fig.6(a-d) SEM BSE Images and EDS Analysis of (a) Uncoated: (b) 3% CNT: (c) 5% CNT: (d) 7% CNT coated T12 subtrate after exposure at 600°C.

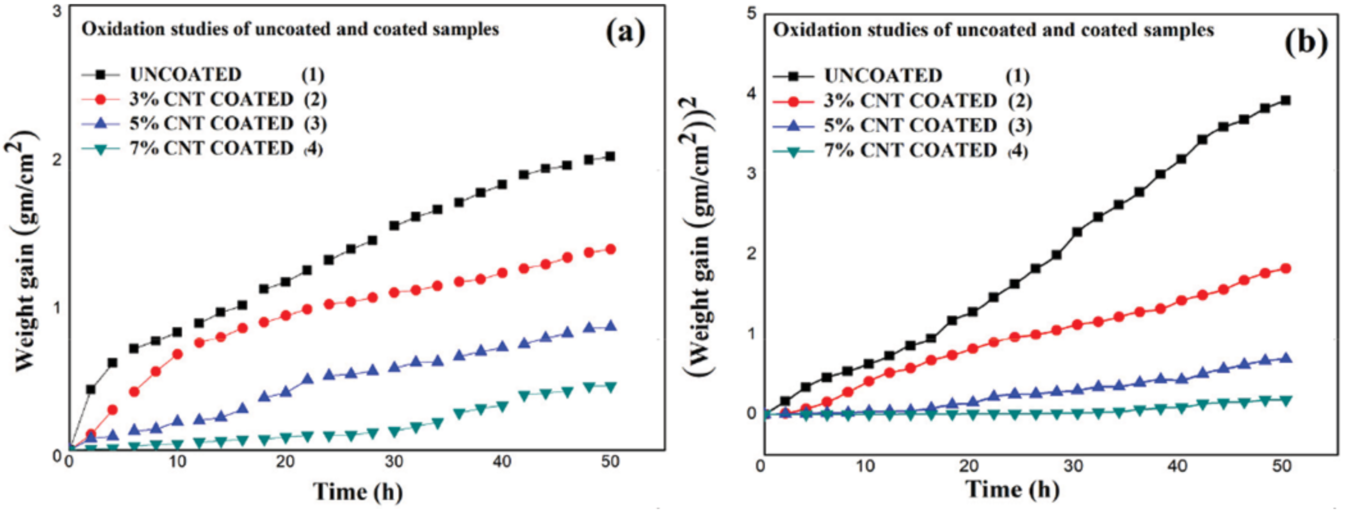

Fig.7(a-b): Plots of thermogravimetric analysis: (a) Weight gain/area versus number of cycles: (b) (Weight gain/area)^2 versus number of cycles for all samples exposed to air oxidation

Furthermore, the CNT reinforced coated samples have followed the parabolic law, which indicated that the scales formed have shown the tendency to act as a diffusion barrier to corrosive species. The values of parabolic rate constant (Kp) for CNT reinforced coating were found to be significantly lower than uncoated T12. The minimum value of K_p was observed for 7% wt. of CNT coated sample has 0.00367 $g^2cm^{-4}s^{-1}$, and it represents the highest oxidation resistance than all other coatings in air oxidation at 600°C for 50 cycles.The nanoparticle has smaller in size the CNT effectively to fill these residues in voids, pores in the coating matrix due to this cumulative weight gain decreases. The corresponding to this least weight accumulation was noticed in 7% wt. of CNT reinforced in a coated sample, and due to this it possessed maximum air oxidation resistance attributes compared to uncoated, and 3, 5% wt. of CNT coated samples. All coated samples examined by XRD technique $Cr_{23}C_6$, Cr_2O_3, as the vital amount, and NiO, carbon has little peaks was recognized due to presence of CNT in coating structure. The formation of shield oxide Cr_2O_3-NiO layers on coated samples, these layers has strong hot-corrosive resistance, act against penetrating species into base substrate material.

Table 3: Parabolic rate constant and cumulative weight gain for all samples

Type of coating	Corrosion rate K_p valuesin $10^{-10}g^2cm^{-4}s^{-1}$	Cumulative weight gain in $mgcm^{-2}$
Uncoated sample	0.08236	32.9191
3% wt. of CNT coated sample	0.03607	23.1461
5% wt. of CNT coated sample	0.01434	11.6556
7% wt. of CNT coated sample	0.00367	4.2028

4.0 Conclusions

In this research work, air oxidation behaviour of CNT reinforced Cr_3C_2-25%NiC coatings on T12 steel was investigated at 600°C the results as follows.

- The uncoated steel indicated higher weight gain, higher oxidation rate and results in formation of thick, porous, and non-protective scale during exposed to hot conditions observed by SEM/EDS analysis.
- The 3% wt. of CNT coated steel successfully reduced weight gain by 29.68% than uncoated steel due to the formation of protective oxides of chromium. Minor cracks with some spallation of scale were observed after air oxidation studies due to the formation of iron oxide.
- The development of Cr_3C_2, C, NiO, NiCr and Cr_2O_3 phases were found in CNT coated samples these elements leads to good oxidation resistance to the substrate.
- All CNT reinforced coatings showed comparatively lower weight gain than uncoated sample. The CNT reinforced coating samples were intact, without cracks and spallation after 50 cycles of studies. 3, 5, and 7% wt. of CNT coated samples reduced cumulative weight gain of by 29.68, 64.59, and 87.23% respectively. 7% wt. of CNT coated sample has highest oxidation resistance than all other coatings at 600 °C for 50 cycles.

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