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High Temperature Corrosion Study on NiCr Coated Low Alloy and Mild Steel

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D-gun spray, corrosion, elevated temperature, thermal spray coatings.

Abstract

Cyclic hot corrosion behaviour of bare and D-gun sprayed NiCr coated low alloy and mild steel sample having dissimilar coating thicknesses viz. 0.2-0.25 mm and 0.25-0.30 mm have been studied in molten salt environment mixture of Na_2SO_4 - 25wt. % NaCl at temperature of 750°C. Corrosion kinetics were analysed and parabolic rate constants (K_p) for both bare and coated sample were calculated and observed that 0.2-0.25 mm thick NiCr coated specimen showed better resistance against high temperature corrosion for both materials. SEM/EDX analysis was carried out to examine scale formed on the bare and coated sample after hot corrosion.

1. Introduction

A severe problem commonly observed in gas turbines, boilers, chemical industries etc is corrosion at high-temperature. At a high temperature formation of metal compounds such as oxides, chlorides, nitrides, sulphides etc. depending upon the exposed environment due to corrosion. Hot corrosion increases the corrosion rate over hundred times as compared to oxidation in heat resistant alloys. Maximum effect of hot corrosion is observed at high temperature when the material is in contact with fused salts viz. Na_2SO_4, NaCl, K_2SO_4, V_2O_5, KOH [1,2]. Hot corrosion causes the materials to be ingested at unusually high rate as compare to oxidation resulting frequent failures at high temperature application. Thus, to overcome such situation, thermal spray coatings has been suggested over the components to overcome elevated temperature oxidation and corrosion [3-4]. Gonzalez et al. [5] studied on grey cast iron coating with NiCrBSi and concluded that laser spray have higher adhesive straight than flame spray coating.

Hard, wear/corrosion resistant and dense micro-structured coatings were formed with the help of Detonation-gun (D-gun) spray technique. It was considered as one of the best coating in terms of higher bond strength, coating density, lower porosity and hardness along with fine grained structure [6-7]. Process parameters and their importance on the tribological behavior of detonation sprayed coatings were studied by various authors. The composition of coating, microstructure, porosity and residual stress has been used to determine coating component hardness, wear, erosion, oxidation, and corrosion resistance [8].

High temperature oxidation and hot-corrosion behaviour of a carbon-alloyed iron aluminide was studied in Na_2SO_4 environment and observed that the degradation rate of material in hot-corrosion was more than that of oxidation [9]. Cr_3C_2-25%NiCr coating of thickness 0.15-0.25 mm deposited on Super Ni 600 alloy using D-gun technique has been studied against oxidation at a temperature of 900°C. Results showed that the bare Super Ni 600 alloy gained four times weight as compared to that in the coated material [10]. Corrosion behavior of Cr_3C_2-NiCr, NiCr, WC-Co and Stellite-6 alloy coatings on ASTM SA213-T11 steel at 900°C in a liquefied salt media was studied and reported that coated steel specimen displayed improved resistance to hot corrosion as compared to that of uncoated steel specimen. NiCr coating was found to be best in protecting the substrate steel. Formation of Cr_2O_3, NiO and $NiCr_2O_4$ provide better resistance to hot-corrosion [11].

Ni- based alloy Superni 600 is studied in salt environment at high temperature with and without coating. The nature of rate constant is found parabolic for both the case [12]. Hot corrosion behaviour of NiCr and Cr_3C_2-NiCr coating in salt Na_2SO_4-V_2O_5 were studied and observed that NiCr coating exhibit better corrosive resistance properties as compared to Cr_3C_2-NiCr coating [13-15]. Zikin et al. [16] studied the wear behaviour of reinforcement TiC-NiMo and Cr3C2-Ni at elevated temperature. From this study they concluded that at elevated temperature reinforcement is wear out by oxidation.

Augmented corrosion rate has been observed [17-22] in different materials exposed to corrosion in the molten salt environment of Na2SO4/NaCl. Azakli et al. [23] studied oxidation of two different alloys (NiAlCr–Ca and NiAlCr–Sr) and stated that NiAlCr–Sr alloy exhibited the best oxidation resistance. A study on 0.2C-Mn (rolled), 0.2C-Mn, 0.15C-0.5Cr and 0.1C-0.5Cr-0.2Mo steels were conducted in CO2 environment and observed relatively lowest corrosion rate in 0.2C-Mn steel [24]. Author worked on (CoCrFeNi)100-xMox alloys to investigate the influence of Mo on corrosion behaviour in 3.5% NaCl solution and reported that corrosion resistance of the alloys gradually increased with increasing aging temperature and Mo contents [25]. To study on nano-particle NiCrCoAlY mixed with TiB2 coating was performed by Zou at al. [26], and concluded that coating shows small weight gain with excellent oxidation resistant. To study that effect of boiler component at 800°C T91 steel was coated with WC-CO and Cr3C2-NiCr. It was concluded from the result that after 50th cycle WC-CO coating was wear out where as Cr3C2-NiCr coating was adhere with the sample [27].

Senderowski and Bojar [28] worked with gas explosion splashed covering on plain carbon steel. The application property for example the level of request, the stage creation, grain morphology, outside covering bonds and so forth was investigated. Warm strength of Fe-Al covering is better after gas explosion showering and extra warming at 750 °C and 950 °C for 10 h. Audigie et al. [29] studied P91 alloy with coating aluminide and nickel-aluminide to measure corrosion in molten salt environment. They concluded that uncoated P91 showed considerable mass gains and spallation in both conditions. Senderowski et al. [30] worked austinite valve materials with D-gun spray Fe-Al type coating. At a high temperature range Fe-Al coating shows different oxidation behaviors in air environment. To simulate acid-rain environment material was tested in NaCl solution, and concluded that it is suitable for acidic environment. Kumar et al. [31] concluded that at the high temperature corrosion of FeAl- based alloy reduced due to formation of Al_2O_3 and TiO layers. Senderowski et al. [32] works with FeAl base intermetallic HVOF coatings in molten salt at 850°C. All coatings were described by Al-eliminated locales, interpolate oxidation and different stoichiometric proportions of iron aluminides. The outcomes are talked about as for the development of oxide scale on a superficial level after openness to destructive media, as well as the heterogeneity and imperfections of the showered coatings.

As per the available literature, there are many literatures that studied in different salt environments but no literature was found that evaluate cyclic corrosion behavior in mixture of salt environment using different thickness. Thus, there is need to comprehensive study of corrosion behaviour in salt mixture in different coating thickness. Therefore, corrosion behaviour of bare and D-gun spray NiCr coated low alloy (LA) and mild steel (MS) sample in molten salt environment mixture of Na_2SO_4 - 25wt. % NaCl at a temperature of 750˚C using different coating thickness were studied. The comparison of resistance offered by varying coating thicknesses (0.2-0.25 mm and 0.25-0.30 mm) NiCr coating over LA and MS sample is also studied using parabolic rate constant.

2. Materials and Method

Both materials were procured in form of plate of 6 mm thickness. Spark spectroscopy test was used to determine the chemical compositions of the LA-steel and MS (Table 1-2), respectively. Specimen were cut with the help of WEDM in the size of 15mm x15mm x5 mm, and polished using polishing papers to remove oxide layer. D-gun spray technique was used to coat NiCr coating on the specimen of thickness in the range of around 0.2-0.25 mm and 0.25-0.3 mm.

Table 1 *Results for spectroscopy test for LA steel*

Elements	Al	Ni	Cr	P	Si	Ti	Mn	C	Fe
Wt. %	0.038	0.033	0.036	0.024	0.219	0.013	1.13	0.086	Remining

Experiments were performed at temperature ranging from 750±10˚C for 50 cycles on bare and coated LA and MS sample using laboratory muffle furnace. The cycles have of time duration of 1 hr and 20 minutes in which 1 hr of heating thereafter 20 min of cooling to attain atmospheric temperature. The experiment was performed using alumina boats. These boats ware preheated for 10 hours before starting the experiment. The sample also heated at 230˚C before application of salt. Slurry of molten salt (Na2SO4 - 25wt. % NaCl) was prepared by the adding distilled water with salts in the given weight percentage. 3-5 mg/cm2 of salt slurry was applied uniformly over

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the sample followed by drying of sample in the furnace at temperature 140°C for 80 minutes for the proper adhesion of salt. Weight of sample after the application of salt were taken as the initial weight of the sample, and weight changes after each cycle were measured using digital electronic meter. Hardness of the sample is measured using Vickers hardness testing machine at 5 kg load. SEM and EDS report were generated by SEM machine (Carl Zeiss EVO 50).

Table 2 *Results for spectroscopy test for MS-sample*

Elements	C	P	S	Si	Mn	Fe
Wt. %	0.18	0.017	0.018	0.22	0.88	Remining

3. Results and Discussion

3.1 Bare and NiCr Coated LA Steel Behaviour During Hot Corrosion

3. From starting to 49th cycle, higher weight gain in LA steel was observed during hot corrosion. Whereas low weight gain was observed in NiCr coated specimen after 50 cycles. During hot corrosion scale formation was started with the end of first cycle and inflammation was detected after 11th cycle in bare LA steel specimen. Breaking of scale from corners was started after 1st cycle in case of bare samples and it was continued till 50th. Irregular scale and cracks at the corner were observed in hot-corroded bare specimen. The delicate scale on the surface of the specimen could not stand and began to peel off. The oxides formed immediately after the 1st cycle on the bare steel specimen were dark gray in color and persisted until the completion of the 50th cycle, while green oxides were observed on the surface of the NiCr coated specimen. Thus, coated specimen showed better hot corrosion resistance as compared to bare. Figure 1 shows the surface morphology of bare and NiCr coated low alloy steel samples at a temperature of 750 ± 10˚C in a molten salt atmosphere of Na2SO4 - 25wt.% NaCl. The scale formed on the bare low alloy samples after 50 cycles shows the presence of dense and delicate oxides with Fe and O being the major amounts and C, Na, S and Cl in minor amounts as in Figure 1a. NiCr coated low alloy steel samples showed Cr and O in major amounts and C, Ni, Na, S, Fe, and Cl in minor amounts as shown in Figure 1b and 1c. The continuous and dense layer of Cr and Ni oxides acts as a diffusion barrier and prevents oxygen from entering the substrate. Excellent corrosion resistance was provided by the coated samples due to the formation of Ni and Cr oxides. Furthermore, the coating was dense, compact and showed no dispersion or peeling of scales.

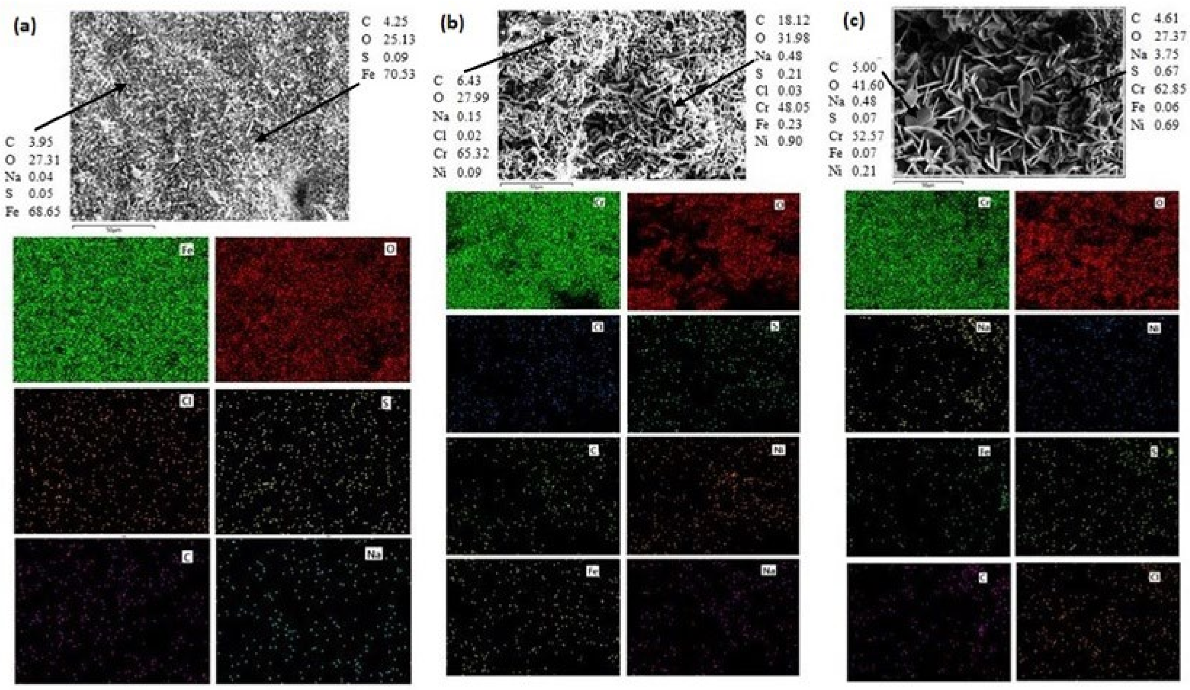

Fig. 1 *EDS and elemental mapping of (a) bare; (b) 0.2-0.25 mm; (c) 0.25-0.30 mm, thick NiCr coated low alloy steel sample of hot corrosion after 50 cycles*

3.2 Evaluation of Corrosion Rate

The mean of corrosion at 750 ± 10˚C under a molten salt atmosphere for bare and NiCr coated LA steel was shown in the Fig. 2 for every cycle. The Fig. 2 shows that NiCr coated samples gained small weight as compared to bare. After 50 cycles of hot-corrosion, the total weight increases for samples of bare, 0.2–0.25 mm, and 0.25–0.3 mm thick NiCr coated LA steel were calculated to be 147.02094, 8.61723, and 10.2975 mg/cm^3, respectively. Besides, the explanatory rate consistent (Kp) was assessed for the low combination steel. In the Fig.3, the diagram between square of weight gain (mg^2/cm^4) and number of cycles were plotted. The linear regression was utilized to gauge parabolic rate constant (K_p). When the data are plotted as weight squire versus time, metals exhibiting parabolic corrosion rates produce a straight line. It can be concluded that the NiCr coated samples almost obey a parabolic rate law. Correlation coefficients (R^2) for bare, 0.2-0.25 mm, and 0.25-0.30 mm thick NiCr coated LA steel sample were 0.99, 0.99, and 0.93, respectively. The parabolic rate constant (Kp) for bare, and NiCr coating is given in Table 3. The K_p values of the coatings were found to be much lower than that of bare steel. The sample shows that, the coatings protected the samples against hot corrosion, the lower the value of K_p, the better the corrosion resistance.

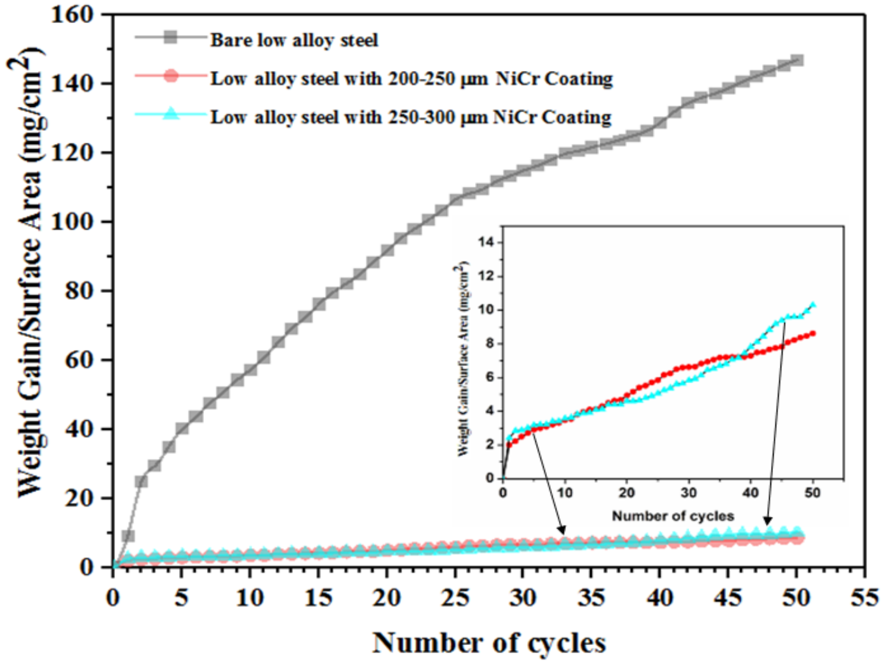

Fig. 2 *Change of weight with number of cycle in low alloy steel subjected to cyclic hot-corrosion*

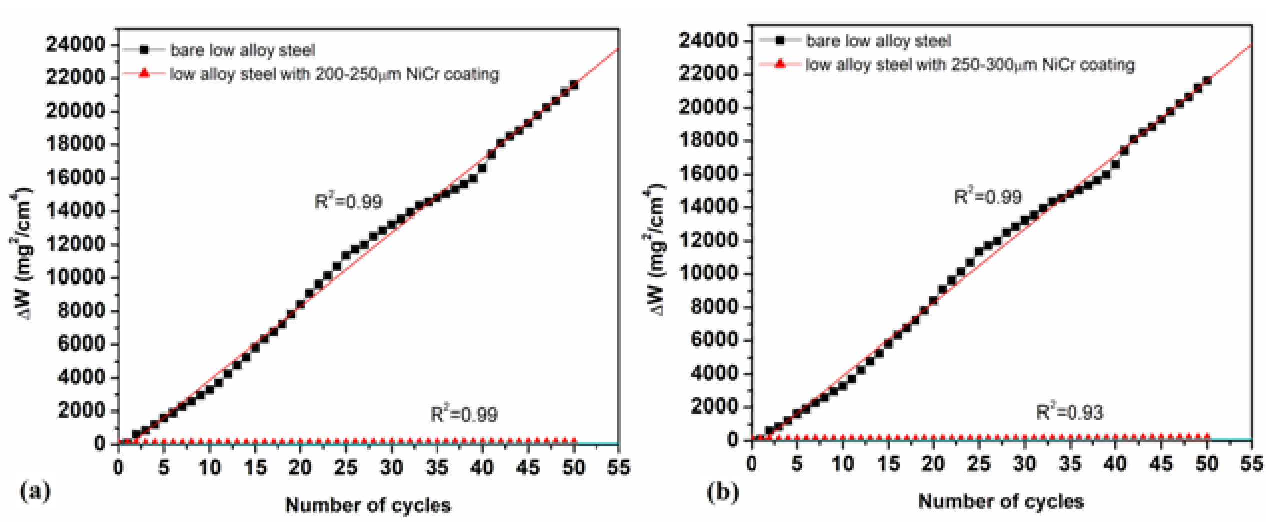

Fig. 3 *Weight gain square vs. No. of cycle of bare and (a) 0.2-0.25 mm; (b) 0.25-0.30 mm, thick NiCr coated LA steel sample subjected to hot-corrosion*

Table 3 *Parabolic rate constants (K_p) of corroded LA steel sample*

Sample No.	Sample (LA steel)	K_p (10^{-10} $g^2/cm^4/s$)
S1	Bare	1232.47
S2	0.2-0.25 mm thick NiCr coating	3.98677
S3	0.25-0.30 mm thick NiCr coating	5.05509

Table 4 *The parabolic rate constants (K_p) of corroded MS sample*

Sample No.	Specimen (Mild steel)	K_p (10^{-10} $g^2/cm^4/s$)
S4	Bare	736.003
S5	0.2-0.25 mm thick NiCr coating	1.22216
S6	0.25-0.30 mm thick NiCr coating	10.1276

3.3 Bare and NiCr Coated MS Steel Behaviour During Hot Corrosion

NiCr coated MS samples showed slight weight gain until the 50th cycle, while bare MS samples started from the first cycle and continued until the 50th cycle. During hot-corrosion in molten salts of bare MS, scale formation was observed at the end of the 1st cycle and scale swelling was observed at the end of the 10th cycle. A slight sorting of the scale for bare samples began at the end of the 17th cycle; However, the construction of the scale continued until the 50th cycle. Irregular scales with cracks at the edges were observed in hot-corrosive bare MS samples. The delicate scale could not stand on the surface of the sample and began to peel off the specimen surface. The oxides formed after the first cycle on the bare mild steel sample were dark brown until the completion of the 50th cycle, whereas green oxides were observed on the surface of the NiCr coated samples.

Figure 4 depicts the bare and D-gun sprayed surface morphology of MS samples with NiCr coatings that have corroded at a temperature of 750 10°C in a molten salt environment. In contrast to the naked mild steel samples, which displayed increasing weights beginning with the first cycle and lasting until the 50th cycle, the NiCr coated mild steel samples barely increased in weight. Scale development was noticed during hot corrosion of bare mild steel at the conclusion of the first cycle, and scale swelling was noticed at the end of the tenth cycle. Additionally, irregular scaling and cracks were seen at the specimen's rough edges made of mild steel. The sample' surface developed a fragile scale that couldn't stand and started to fall off. The scale's minor spelling started at the end of the 21st cycle, but construction didn't finish until the 50th. The hot-rusted bare sample's elemental mapping analysis revealed a Fe and O-rich scale, as shown in Fig. 4a, that was nearly equivalent to that of the low alloy steel samples. The samples that had been coated revealed a consistent and error-free interfacial connection with the substrate steel.

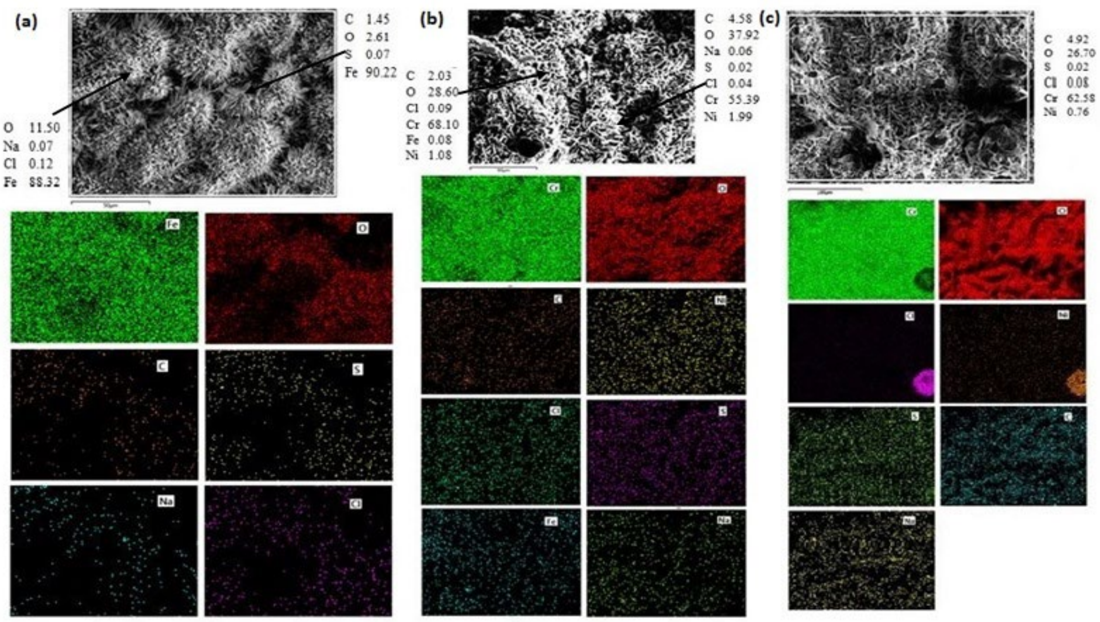

Fig. 4 *EDS spectrum and elemental mapping of (a) bare, (b) 0.2-0.25 mm;(c) 0.25-0.30 mm, thick NiCr coated MS-sample subjected to high temperature corrosion*

3.4 Evaluation of Corrosion Rate

Figure 5 displays the cumulative weight increases per unit surface area vs. cycle count for MS sample that were either uncoated or coated with NiCr and subjected to high temperature corrosion at 750±10°C in a molten salt environment. The total weight gain for the uncoated, 0.2-0.25mm, and 0.250–0.300 mm thick NiCr coated MS sample was calculated to be 114.92829, 5.60327, and 14.1813 mg/cm^2, respectively. The graph between square of weight gain vs. number of cycles graph were plotted as shown in Fig. 6, and it used to determine Kp for the MS sample. The correlation coefficients (R^2) between bare and coatings were 0.98, 0.96, and 0.94, respectively. Parabolic rate constants (K_p) of bare and NiCr coatings (0.2-0.25 mm, and 0.25-0.30 mm) MS sample are given in Table 4 for corrosion. The value of Kp of 0.2-0.25 mm thick NiCr coated MS was found to be lowest as compared to other, indicated that 0.2-0.25 mm thick NiCr coated has better bond strength than that of others NiCr coated specimen.

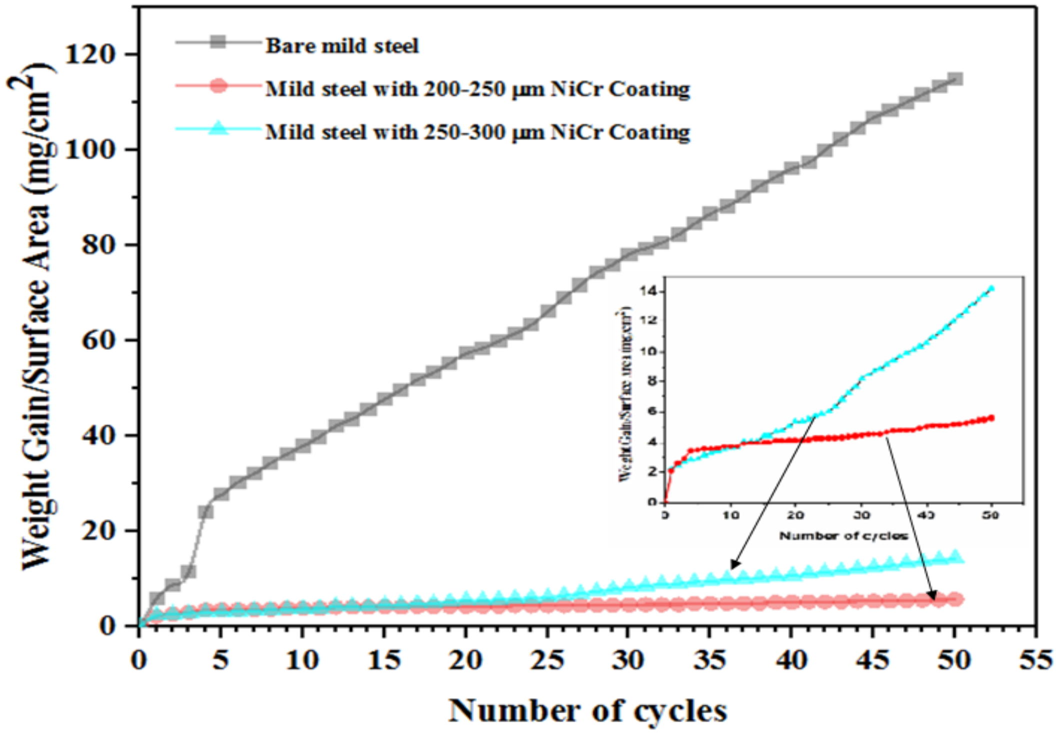

Fig. 5 *Weight change/surface area (mg/cm^2) vs. number of cycle graph of MS sample subjected to cyclic high temperature-corrosion*

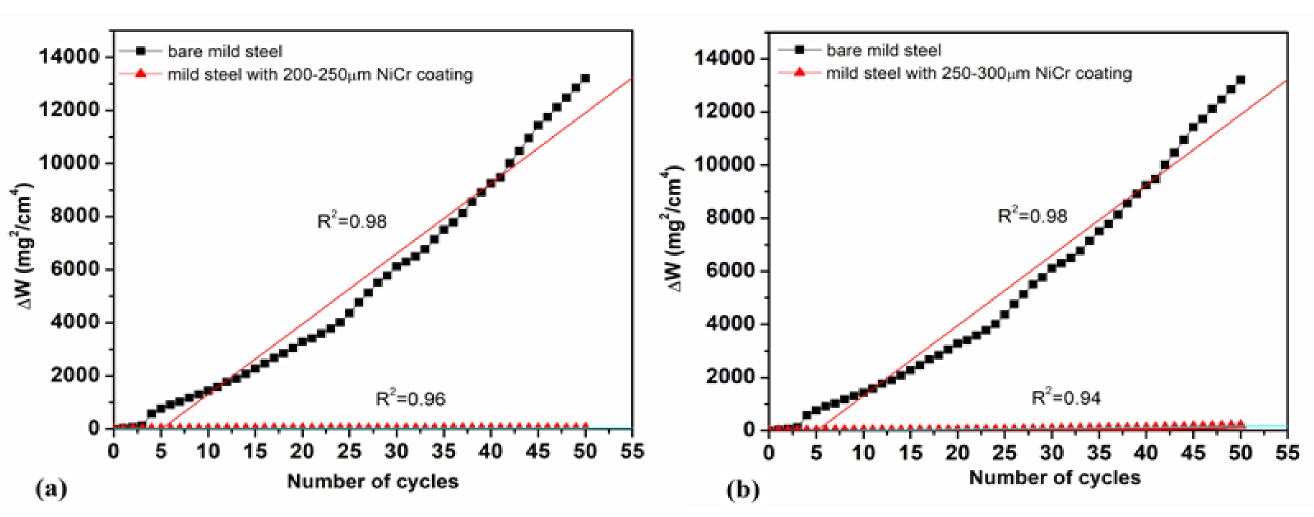

Fig. 6 *Weight gain square (mg/cm^2)^2 vs. number of cycle graph of bare and (a) 0.2-0.25 mm, and (b) 0.25-0.30 mm, thick NiCr coated MS sample subjected to high temprature-corrosion*

3.5 Oxidation Kinetics of Low Alloy Steel and Mild Steel

Thicker NiCr coated low alloy steel sample (0.2-0.25 mm, and 0.25-0.30 mm) showed a weight gain that was 17.06% and 14.27% smaller than that of bare LA steel sample, respectively. Additionally, NiCr-coated MS sample that were 0.2-0.25 mm, and 0.25-0.30 mm thick demonstrated 20.51% and 8.10%, respectively, less weight growth when compared to bare mild steel sample. It was found that both sample adhered to the parabolic rate law, which denotes a consistent weight growth and slows the progression of corrosion. There was an initial increased weight gain, which may have been caused by the hastened interfacial reaction. Additionally, oxidation of air trapped within the coating may contribute to weight increase [6]. Ni is less reactive than Cr, thus the coating of NiCr forms $NiCr_2O_4$ scale which is more resistant to NaCl induced corrosion. In early stages of corrosion, Ni and Cr react with O_2 to form NiO and Cr_2O_3 [33]. Cl^- generation mechanisms have been described as the initiation mechanism to activate corrosion in sodium chloride. The released chlorine reacts at the metal-oxide interface travelling through the protective oxide (Eq. 1) [34].

$$Cr + Cl_2 \rightarrow CrCl_2 \quad (1)$$

Chlorine preferentially attack chromium due to greater affinity of chromium for chlorine as compared to iron [35]. Chromium chloride converted into chromium oxide and chlorine after reacting with oxygen (Eq. 2). Chlorine then migrates back to the corrosion front to form metal chlorides again and so on.

$$2CrCl_2 + 1/2O_2 \rightarrow Cr_2O_3 + 2Cl_2 \quad (2)$$

Equations 1 and 2 have been termed the “active corrosion” in which chlorine acts as a catalyst in converting the metal to its oxide resulting acceleration in corrosion. SEM analysis shows the presence of various elements like Cr, Ni, Cl, S, Na, C and O with different wt.% over the sample after 50 cycles of hot corrosion. Elemental mapping of corroded bare low alloy and mild steel sample show that Fe and O co-exist which confirm the formation of iron oxides. Presence of some cracks or inclusions on the coated surface are also noticed through which the aggressive species penetrate into the substrate and breakes the protective oxide layer and starts reacting with the substrate. Hot corroded bare low alloy and mild steel sample showed porous scales over the surface which indicates non-protective behaviour of scales. Presence of Cr and O was confirmed by the needle/platelet type of oxides. Oxide structure with similar platelets was observed while studying NiCrAlY coating exposed to Na_2SO_4-10%NaCl environment [36].

Due to the production of NiO, Cr_2O_3, Na_2CrO_4, and other materials that aid in providing resistance to high temperature corrosion, the surface morphology of the coated sample revealed larger amounts of Cr and O. The best oxide to give resistance against corrosion at high temperatures in molten sulphates is chromate, which is formed preferentially when chromate interacts with oxygen to create chromate [37]. By adjusting the spray process parameters (thickness), bond strength is assessed. It is found that bond strength of the coating reduced with increasing thickness. The metallic ions and oxidising species cannot penetrate the substrate due to the continuous thick band of Cr2O3 covering the coated sample. The oxide layer functions as a diffusion barrier, aiding in the reduction of future corrosion.

4. Conclusions

In this study, corrosion behaviour of bare and D-gun spray NiCr coated LA and MS sample in molten salt environment mixture of Na_2SO_4 - 25wt. % NaCl at a temperature of 750˚C using different coating thickness were studied from this research, the following conclusions was made.

0.2-0.25 mm, and 0.25-0.30 mm, thick NiCr coated hot-corroded LA steel sample showed 17.06%, and 14.27%, respectively, less gain in weight as compared to that of bare LA steel specimen. Also 0.2-0.25 mm, and 0.25-0.30 mm, thick NiCr coated hot-corroded MS sample showed 20.51%, and 8.10%, respectively, less weight gain as compared to that of bare MS specimen. The inward diffusion of corrosive species was restricted due to the oxide of chromium along the nickel-rich splats boundaries.

Parabolic rate constants (K_p) for hot-corrosion of bare, 0.2-0.25 mm, and 0.25-0.30 mm thick NiCr coated LA steel were calculated to be 1232.47, 3.98677, and 5.05509, (x 10^{-10} g^2 /cm^4 s^1) respectively, whereas for MS sample were calculated to be 736.003, 1.22216, and 10.1276, (x 10^{-10} g^2 /cm^4 s^1) respectively. The value of k_p of the coatings was found smaller then bare steel, representing the defending nature of the coatings counter to hot-corrosion

NiCr coated (0.200-0.25mm) steel showed better resistance against high temperature corrosion than 0.25-0.30 mm thick NiCr coated LA and MS. Corrosive media penetrate more easily in 0.25-0.3mm than in 0.2-0.25mm.

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Author Contribution

In this work ***conceptualization and methodology*** *was developed by Jitendra Kumar and Sunil Kumar Rajput,* ***Reviewing and editing*** *was done by Tarun Soota, supporting by Anshika Kushwaha.*

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