

Cyclic oxidation behavior of bare and Cr₃C₂-25 (NiCr) coated super alloy at elevated temperature

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ABSTRACT

High temperature oxidation is the main cause for the degradation of materials used at elevated temperature which is responsible for the major shut down of turbines, boilers and incinerators used in large units. An attempt has been made to use Cr₃C₂-25%NiCr coating deposited by D-gun technique on Superni 600 alloy. These coatings are generally used for wear applications. This coating has been studied for oxidation at 900C under cyclic conditions. Sound and adherent coatings of 150-250 μm can be obtained by D-gun process. The bare and coated alloys were subjected to cyclic oxidation in air for 100 cycles. Weight change/area has been plotted against number of cycles and to monitor the oxidation kinetics. The scale formed after oxidation both on the bare and coated alloys has been examined using FESEM/EDX, XRD, and Cross-sectional/Mapping analysis. The weight gain by coated Superni 600 was found to be about 80% less than that of bare Superni 600. D-gun sprayed Cr₃C₂-NiCr coating has contributed to increase in the oxidation resistance of coated superalloy. These types of coatings can be developed for high temperature applications for both corrosion and erosion resistance. Copyright © 2013 VBRI press.

Keywords: Oxidation; Ni-based superalloys; D-gun; Cr₃C₂-NiCr coating.



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Introduction

The oxidation of materials at elevated temperature may be referred to high temperature corrosion. High temperature is considered to be 500 °C and above [1]. Superalloys have shown their potential to withstand high temperature. Therefore when extreme temperature and corrosive environments are present, superalloys are used [2]. Coatings are widely used to increase the life of superalloys. Modern thermal spray processes such as plasma spraying, HVOF, D-Gun etc. are usually used to deposit chromium, nickel–chromium coatings on the surface of the superalloys to enhance their high temperature oxidation resistance. Thermally sprayed chromium carbide-nickel chromium coatings are used in various applications involving sliding, abrasion and erosion over a wide range of temperatures up to 900°C [3]. The high resistance of high-chromium, nickel–chromium alloys to high-temperature oxidation and corrosion makes them widely applicable as welded and thermally sprayed coatings in fossil fuel-fired boilers, waste incineration boilers, and electric furnaces [4].

Detonation gun (D-gun) technology is one of the most promising and well known to produce coating of excellent quality. It provides low porosities, high bond strength & hardness.

Lot of studies has been done on friction and wear behavior of Cr₃C₂-25(NiCr) coating sprayed by D-gun and it was reported that coating results in producing high quality wear resistance properties [5-10]. In the practical application of the detonation gun sprayed coatings in Scandinavia it was concluded that the service life of the components coated in this way can be increased 2-3 and more times [11].

In a study, T. Sundararajan et al. [12] reported that two layered Al/Ni-Cr coating was deposited on 9Cr-1Mo steel by APS process and it was observed that the coating exhibited the excellent performance against steam oxidation. The Ni-Cr undercoat was found to be intact. The oxides at the splat boundaries mainly consisted of chromium. S. Matthews et al. [13] reported that Cr₃C₂-NiCr coating provides superior oxidation resistance to WC-Co composites, which have been applied extensively to protect these components against high temperature oxidation and erosion. G.Y. Lai [14] performed oxidation studies on Plasma sprayed and D-gun sprayed Cr₃C₂-NiCr coated 800H alloy in Helium environment at 871C under isothermal conditions for 4000 h. It was observed by them that the oxygen activity at the coating-substrate interface of D-gun sprayed coating was significantly reduced due to decrease in interconnecting porosity of the coatings and further increasing the spallation resistance of D-gun coating as compared to plasma sprayed coating. D-gun sprayed coatings are much superior than plasma sprayed coatings. D-gun sprayed chromium carbide coatings have been used to protect the heat exchanger tubes from the wear damages.

The main aim of the present work is to determine the oxidation resistance of D-gun sprayed Cr₃C₂-NiCr coating at high temperature so as to evaluate its sustainability. Therefore, in present study, D-gun Cr₃C₂-NiCr coated Superni 600 and bare Superni 600 (SN 600) have been subjected to cyclic oxidation for 100 cycles at 900C. Cyclic oxidation has been used so as to create the severe condition and moreover cyclic conditions provide more realistic

approach towards solving the problems of high temperature oxidation in actual applications.

Experimental

Materials

The substrate alloy i.e. SN 600 was procured from Mishra Dhatu Nigam Limited, Hyderabad (India) in the form of hot rolled and annealed sheet. The chemical composition of the alloy is shown in **Table 1**.

Table 1. Chemical composition of substrate.

Elements	Ni	Cr	Fe	C	Mn
Composition (wt. %)	73.8	15.5	10	0.2	0.5

D-gun sprayed coating

A commercially available 75% Cr₃C₂-25%NiCr(Ni₂OCr) powder having particle size 10-45 µm size (Praxair Surface Technology) is used in this study. SEM image of powder is shown in **Fig. 1**. Cr₃C₂-25(NiCr) Coating was deposited on SN 600 substrate by D-Gun process at SVX Powder M Surface Engineering Pvt. Ltd, New Delhi (India).

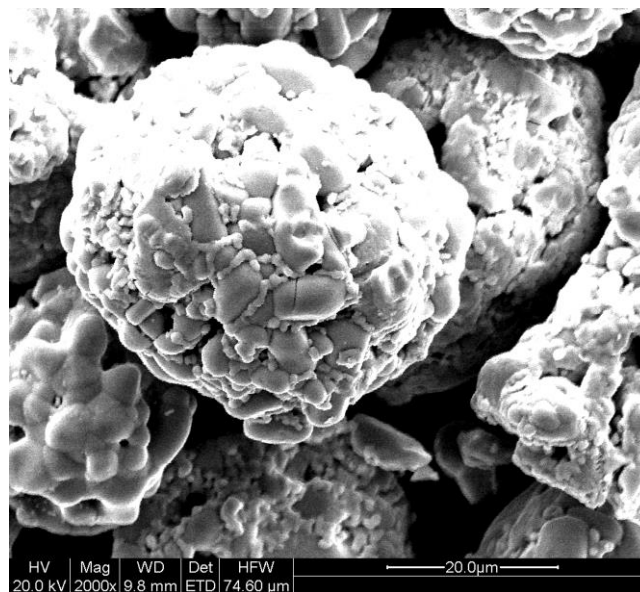


Fig. 1. SEM image of Cr₃C₂-NiCr powder.

Preparation of specimens

The specimens of dimension approximately 20 × 15 × 5 mm were cut from the hot rolled annealed sheet. Prior to cut the thickness of sheet was reduced to 5mm from 8 mm by surface grinding. The specimens were polished using 180, 220, 320, 600 and 800 grit size emery paper respectively followed by cloth polishing using alumina powder of 0.3 micron to obtain the mirror like surface finish. The specimens were degreased using acetone and grid blasted using alumina powder prior to coating by D-gun process. The parameters considered for spraying coating are shown in **Table 2**.

Oxidation study

The oxidation study was performed under cyclic conditions for 100 cycles. The cyclic oxidation was carried out at 900°C in silicon carbide tubular furnace for 100 cycles. One cycle consists of 1 h heating followed by 30 minutes cooling at room temperature. The uncoated sample was polished with emery paper followed by the cloth polishing before being subjected to oxidation test.

The specimens were washed properly with acetone and dried to remove the moisture. The weight of each sample was recorded and put in alumina boat. The alumina boats used for the studies were preheated at 1100 °C for 10 h. The samples were again weighed (with boats) before exposing it to high temperature oxidation.

Weight changes after each cycle was recorded using weighing balance having sensitivity 0.1 mg. After different number of cycles visual observation was recorded. The weight gain per unit area was plotted against number of cycles and parabolic rate constant was calculated.

Table 2. Spraying conditions adopted for D-gun spraying technique.

Process Parameters	Cr ₃ C ₂ -NiCr Coating
O ₂ /Acetylene Volume Ratio	1:1.21
Carrier gas flow rate, N ₂ (m ³ /h)	0.96
Frequency of shots (shots/s)	3
Diameter of spot (spot size)	20
Spraying distance from nozzle(m)	165
Powder flow rate (g/shot)	1-2

Characterization of specimens

After oxidation study, characterization of specimen was done using Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectroscopy (EDX), X-ray Diffraction Analysis, Cross Sectional analysis and X-ray Mapping.

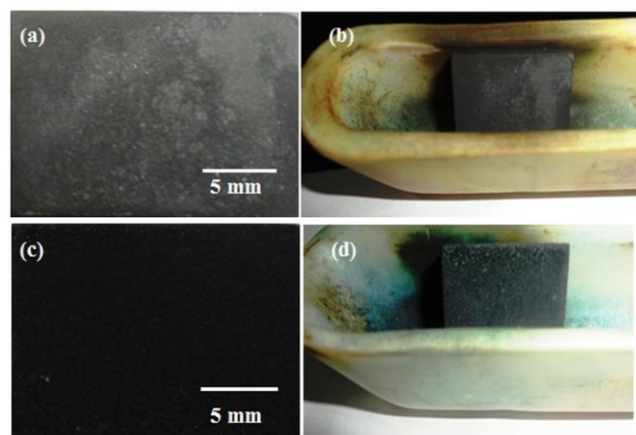


Fig. 2. Weight change measurement of bare SN 600 (a and b) and SN 600 Cr₃C₂-NiCr coated (c and d) after subjected to cyclic oxidation in air for 100 cycles at 900°C respectively.

Results and discussion

Visual analysis

During the visual inspection the texture and color of the surface scale was examined and recorded. The sample of

which was of grey color in the beginning was covered with the green color scale on the dark grey background as shown in **Fig. 2(a and b)**. On the other hand the grey color coating turned to dark green after the completion of 100 cycles as shown in macrographs **Fig. 2(c and d)**.

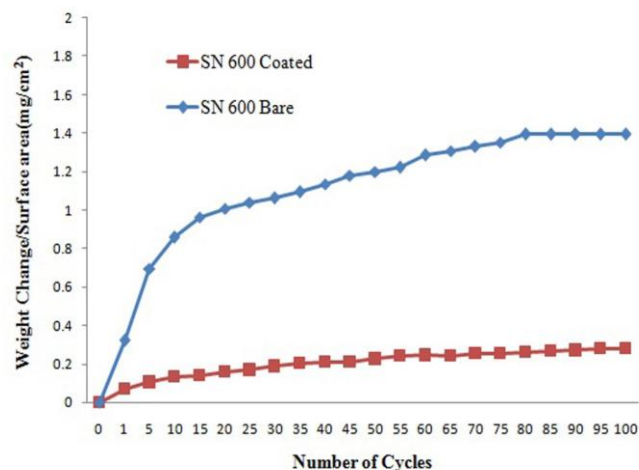


Fig. 3. Weight Change Measurement of bare SN 600 and Cr₃C₂-NiCr coated SN 600 specimen after subjected to cyclic oxidation in air for 100 cycles at 900°C.

Table 3. Parabolic rate constant.

Sample	Parabolic rate constant (g ² cm ⁻⁴ s ⁻¹)
SN 600 Bare	0.23 x 10 ⁻¹⁰
SN 600 Coated	0.008 x 10 ⁻¹⁰

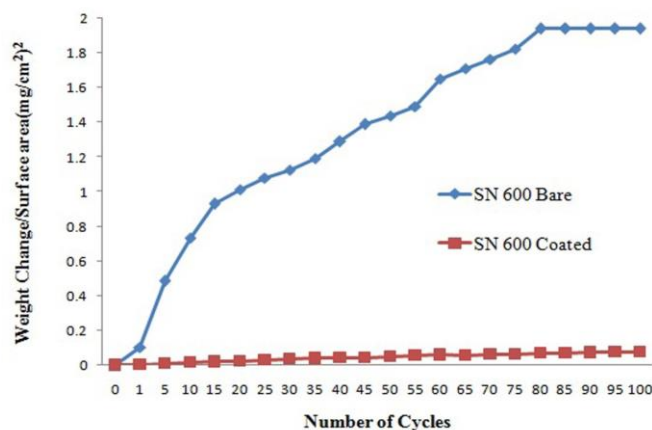


Fig. 4. Weight Change measurement of bare SN 600 and SN 600 Cr₃C₂-NiCr coated specimen after subjected to cyclic oxidation in air for 100 cycles at 900°C.

Weight change measurements

The weight changes (mg/cm²) vs. number of cycles has been plotted in **Fig. 3** and it was noticed that the weight gained by bare sample is more than the coated one after air oxidation at 900°C for 100 cycles. At the end of 100 cycles the weight gain by the bare and coated samples was 1.3933

mg/cm² and 0.2809 mg/cm² respectively. Graph for (wt changes/surface area)² Vs. No. of cycles has been plotted to calculate the parabolic rate constant (K_p) as shown in **Fig. 4**. Parabolic rate constant has been calculated and given in **Table 3**.

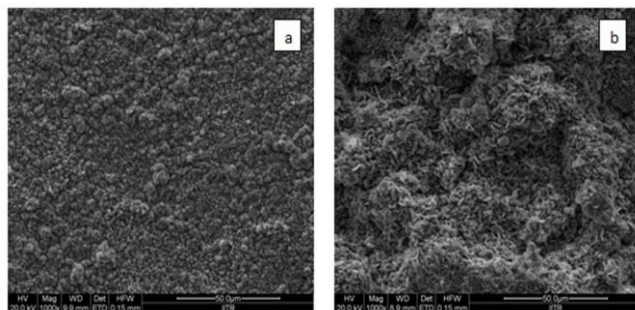


Fig. 5. SEM images of (a) bare SN 600 and (b) SN 600 Cr₃C₂-NiCr coated specimen after subjected to cyclic oxidation in air for 100 cycles at 900°C respectively.

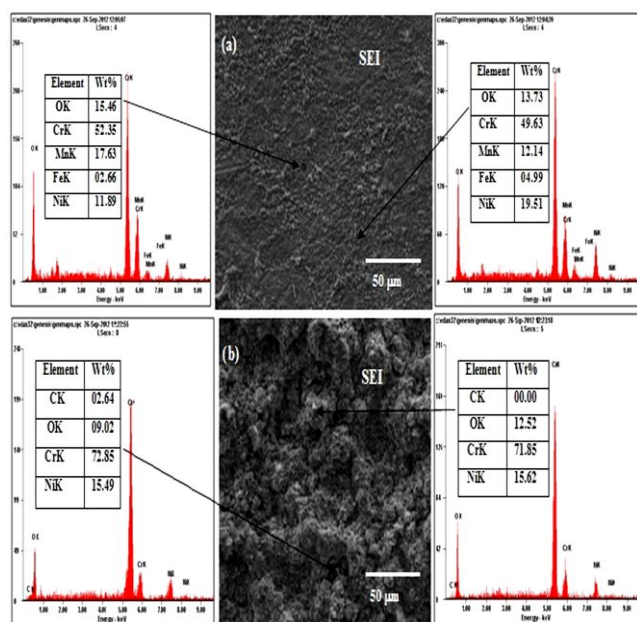


Fig. 6. EDX analysis of (a) bare SN 600 and (b) SN 600 Cr₃C₂-NiCr coated at 500x after subjected to cyclic oxidation in air for 100 cycles at 900°C respectively.

FESEM/EDX

Surface morphology of the samples subjected to cyclic oxidation in air at 900°C for 100 cycles has been shown in **Fig. 5**. In bare SN 600 and Cr₃C₂-NiCr coated SN 600, compact, non porous and adherent oxide layer has formed. During the EDX observation of bare SN 600, it was reveal that the Cr is present in the large amount by wt% with considerable amount of Nickel, Manganese, and Oxygen along with a small amount of iron as shown in **Fig. 6(a)**. For the Cr₃C₂-NiCr coated SN 600, Cr is also present in the large amount with substantial amount of Nickel and Oxygen along with a small amount of carbon as shown in **Fig. 6(b)**.

The microstructure of oxide layer formed on the bare and coated substrates are *Rhombohedral* and *Rhombohedral & columnar* as it can be seen in **Fig. 7 (a)** and **Fig. 7(b)** respectively. Jow-Lay Huang et al. [15] and J. Mougin et al. [16] also reported in their study that the microstructure of the scale consists of *Rhombohedral* and *columnar* crystals which may be Cr₂O₃ as reported by. Cr₂O₃ also exists inform of columnar grains which are of very small size [17].

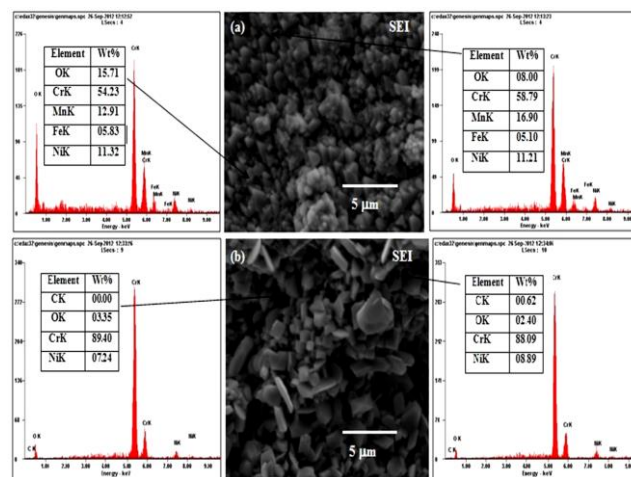


Fig. 7. EDX analysis of (a) bare SN 600 and (b) SN 600 Cr₃C₂-NiCr coated at 5000x after subjected to cyclic oxidation in air for 100 cycles at 900°C respectively.

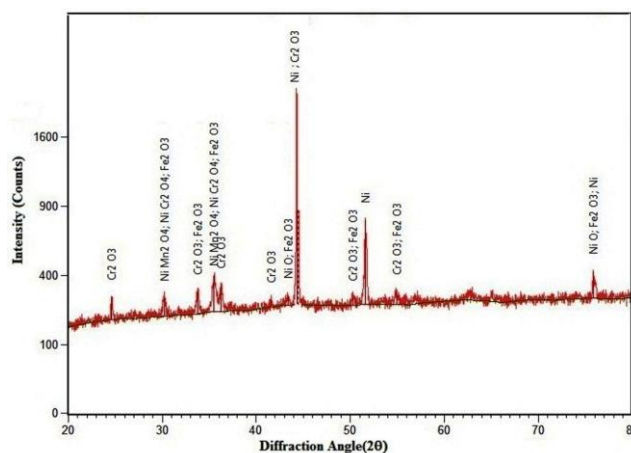


Fig. 8. XRD pattern of SN 600 bare specimen after subjected to cyclic oxidation in air for 100 cycles at 900°C.

X-ray diffraction

X-Ray diffraction analysis for bare SN 600 and Cr₃C₂-NiCr coated SN 600 subjected to cyclic oxidation in air for 100 cycles at 900°C is shown in **Fig. 8** and **Fig. 9** respectively. Major phases observed in bare SN 600 were Cr₂O₃, NiCr₂O₄, and minor phase observed were Fe₂O₃ and NiMn₂O₄. The presence of NiMn₂O₄ peak on bare SN 600 sample shows the diffusion of manganese from the

substrate. While the major phases formed on Cr_3C_2 -NiCr coated SN 600 are Cr_2O_3 , NiCr_2O_4 , Cr_{23}C_6 and Cr_7C_3 along with small peak of Ni.

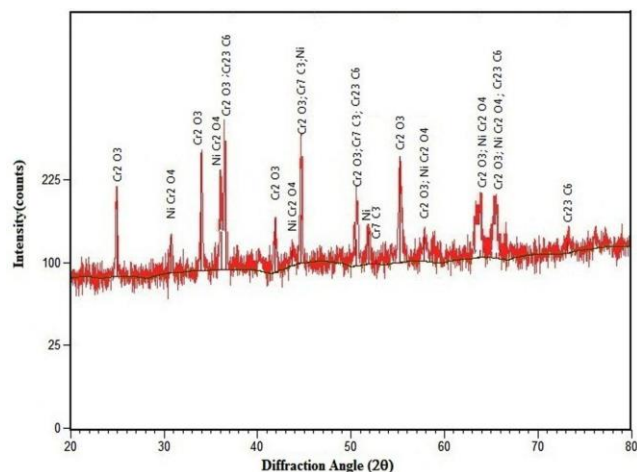


Fig. 9. XRD pattern of SN 600 Cr_3C_2 -NiCr coated specimen after subjected to cyclic oxidation in air for 100 cycles at 900°C.

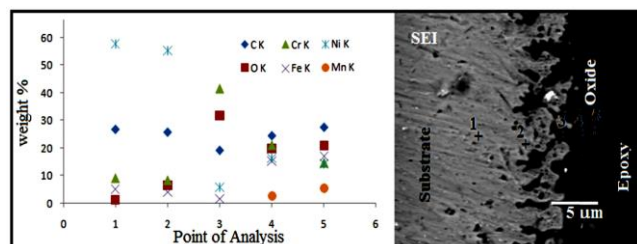


Fig. 10. Cross-sectional analysis of bare SN 600 after subjected to cyclic oxidation in air for 100 cycles at 900°C.

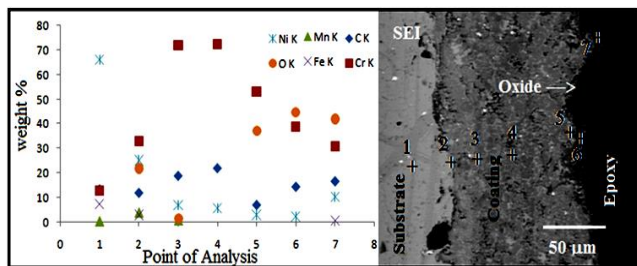


Fig. 11. Cross-sectional analysis of Cr_3C_2 -NiCr coated SN 600 after subjected to cyclic oxidation in air for 100 cycles at 900°C.

Cross-sectional analysis

Cross section image of bare and Cr_3C_2 -NiCr coated SN 600 has been shown in **Fig. 10** and **Fig. 11** respectively. Prior to the analysis the samples were cut along the cross-section and polished down using emery paper followed by cloth polishing using alumina powder. Samples were mounted using cold setting resin powder and gold coated and silver lined in order to make the conductivity of samples and characterize them using FESEM/EDX. It was observed that the oxide layer formed on bare SN 600 was approximately 6-10 μm thick after the cyclic oxidation in air for 100 cycles at 900°C. Whereas in the case of Cr_3C_2 -

NiCr coated SN 600 the thickness of oxide layer formed was 3-6 μm approximately under the same conditions.

During the cross-section analysis of oxidized bare SN 600 (**Fig. 10**) shows that at point 1 and 2 which are in the substrate just below the oxide, the amount of chromium present is very less whereas at point 3 which lies at the interface of oxide layer and substrate the amount of Cr is large. It can be inferred that Cr must have diffused towards the oxide layer from the substrate and formed Cr_2O_3 layer. At point 4 Mn also starts to appear and its concentration increases at point 5 which shows the outward diffusion of Mn. Since Mn is diffused in good amount towards oxide layer which lead to the formation of spinel of Mn and Ni as confirmed by XRD. Fe_2O_3 is also present in the scale.

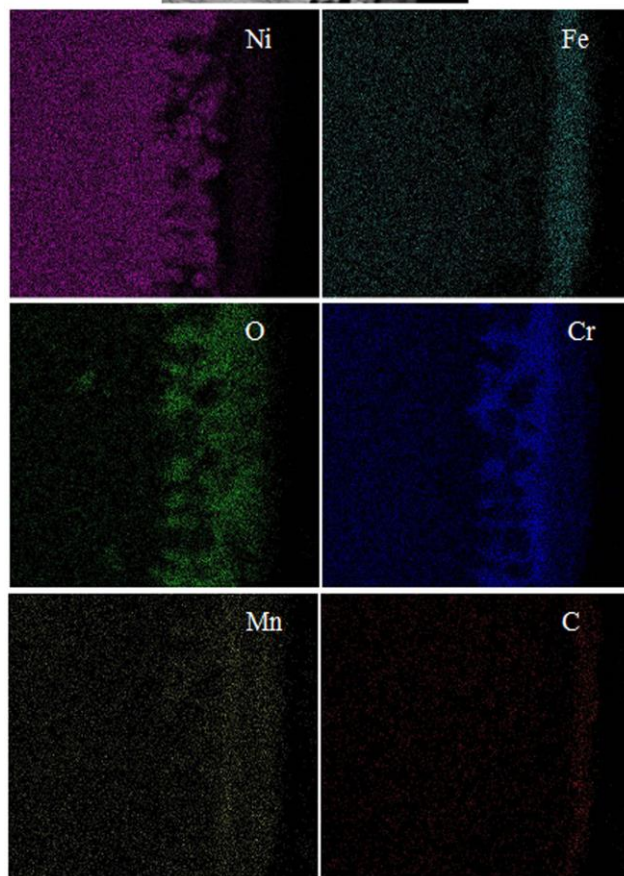
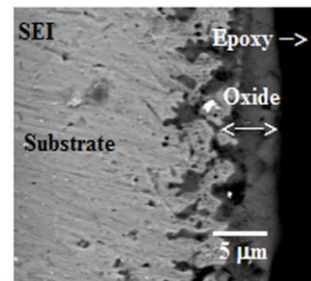


Fig. 12. Mapping of bare SN 600 after subjected to cyclic oxidation in air for 100 cycles at 900°C.

Similarly in case of oxidized Cr_3C_2 -NiCr coated SN 600, cross sectional analysis (**Fig. 11**) shows that the amount of chromium is about 12%wt at point 1 and 33%wt at point 2 which lies on substrate, further

concentration of Cr increases on moving towards coating and is large at point 3 and 4 which lies on coating. At point 5 and 6 which lies in oxide region Cr and oxygen coexist thereby indicates the formation of Cr_2O_3 . The oxide scale formed on the surface of coated SN 600 was found to be very thin

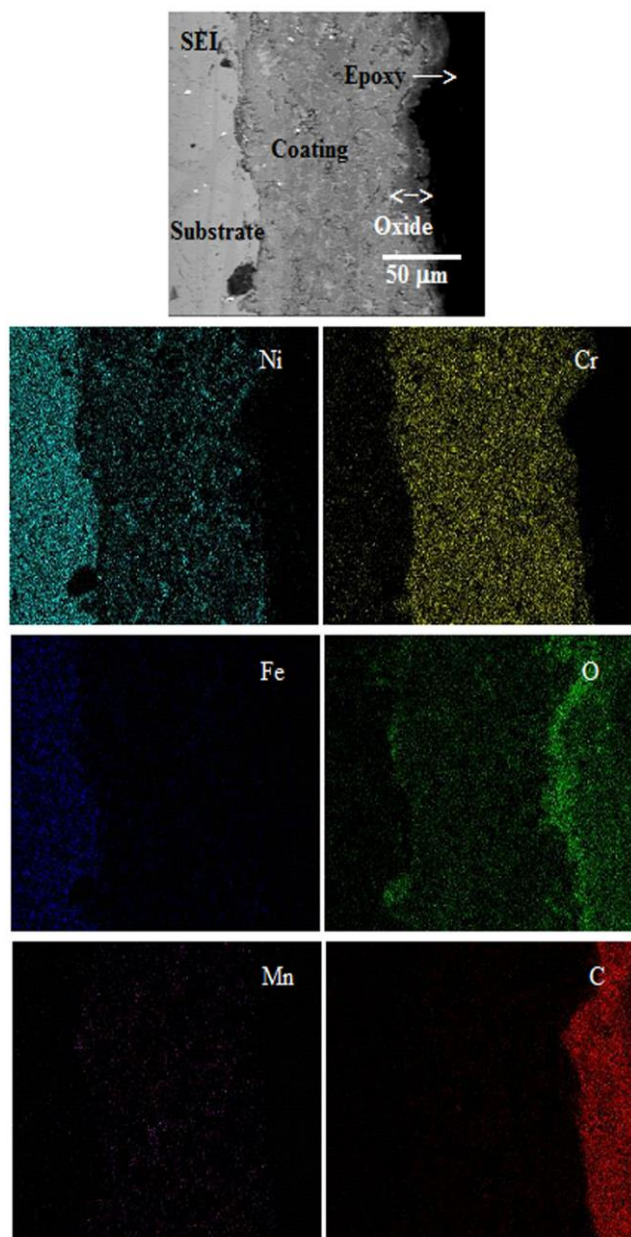


Fig. 13. Mapping of Cr_3C_2 -NiCr coated SN 600 after subjected to cyclic oxidation in air for 100 cycles at 900°C .

X-ray mapping

X-Ray mapping of bare SN 600 and Cr_3C_2 -NiCr coated SN 600 subjected to cyclic oxidation in air for 100 cycles at 900°C are shown in **Fig. 12** and **Fig. 13** respectively. X-ray mapping of SN 600 bare (**Fig. 12**) reveals that Cr and O are coexisting and indicating presence of Cr_2O_3 . Similarly the Fe and Ni are also present in higher concentration in the top

layer of scale forming iron-oxide and spinel of Ni with Mn and Cr.

The mapping of oxidized Cr_3C_2 -NiCr coated SN 600 is shown in **Fig. 13**, which shows that a thin and dense layer of chromium and oxygen is formed on the top of the coating thereby confirming formation of Cr_2O_3 .

Bare SN 600 and D-gun sprayed Cr_3C_2 -NiCr coated SN 600 shows good oxidation resistance at 900°C under cyclic condition after 100 cycles. In bare SN 600, light brown color appears on sample after 1st cycle which changes to dark brown after 2nd cycle. After 5th cycles grey spots also appears on the sample. After 10th cycle, the greenish color also starts appearing. And finally after 100 cycles the grey color oxide forms all over the surface of the specimen as shown in **Fig. 2(a and b)**). Similarly in Cr_3C_2 -NiCr coated SN 600, dark grey oxide appears all over the surface which becomes denser till 5th cycle. In 10th cycle, green color oxide also starts appearing on the surface of the specimen. Finally at the end of 100 cycles, entire specimen was covered with dark green oxide scale as shown in **Fig. 2(c and d)**. The formation of dark oxide i.e Cr_2O_3 may be attributed to the decarburization of the carbides particles [18].

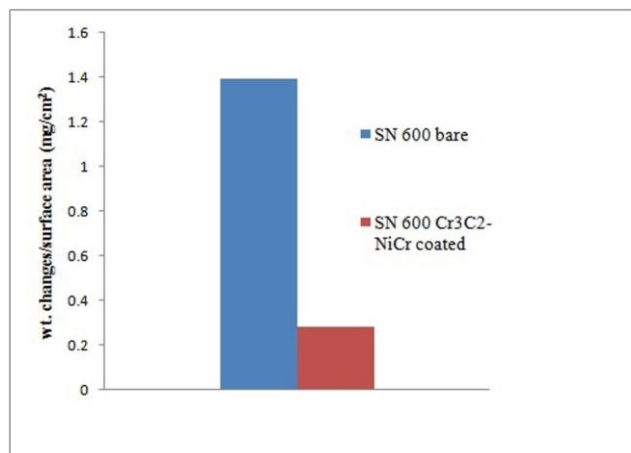


Fig. 14. Weight changes/surface area for bare SN 600 and Cr_3C_2 -NiCr coated SN 600 specimen after subjected to cyclic oxidation in air for 100 cycles at 900°C .

From weight change plot as shown in **Fig. 3**, it was found that the weight gain by Cr_3C_2 -NiCr coated SN 600 is very small as compared to bare SN 600. Weight gain/area after 100 cycles for bare SN 600 was found to be $1.4\text{mg}/\text{cm}^2$ whereas for Cr_3C_2 -NiCr coated SN 600 it was around $0.3\text{mg}/\text{cm}^2$ as shown in **Fig. 14**. Parabolic rate constant (K_p) was calculated using the (weight change/area)² vs. number of cycles plot as shown in **Fig. 4**. The K_p value for bare SN 600 and coated SN 600 was found to be $0.23 \times 10^{-10} \text{ g}^2\text{cm}^{-4}\text{s}^{-1}$ and $0.008 \times 10^{-10} \text{ g}^2\text{cm}^{-4}\text{s}^{-1}$ respectively. K_p value of the coated sample is very less as compared to that of bare one which shows that coating was successful to enhance the corrosion resistance. In a study reported by H. Singh et al. [19], the mass gain in case of plasma sprayed Ni-20Cr coated Superni 600 subjected to cyclic oxidation at 900°C after 50 cycles was found to be $13.98 \text{ mg}/\text{cm}^2$ and parabolic rate constant was found to be $4.10 \times 10^{-10} \text{ g}^2\text{cm}^{-4}\text{s}^{-1}$ which is much higher as compare to D-gun sprayed SN

600. It was found in literature that mass gain by the D-gun sprayed Cr_3C_2 -NiCr coated DIN12CrMo44 steel specimen under isothermal oxidation in air at 850°C is 100 times less than the uncoated specimen after 70 h [6].

It was observed that the oxidation rate was higher during initial cycles while it becomes constant with increase in no. of cycles. Initial increase in oxidation rate may be due to the diffusion of elements such as Cr, Mn from the substrate towards the oxide scale which combines with oxygen to form oxide of these elements. The oxide form on the surface starts acting as a stronger diffusion barrier which stops the diffusion of metal ions with oxygen during remaining cycles [1]. P. Elliot has reported that Cr_2O_3 is stable up to the temperature of 950°C , beyond this at high temperatures Cr forms CrO_3 (g) which is volatile in nature, therefore CrO_3 layer will not protect the substrate anymore [20].

During FESEM analysis it was revealed that a continuous, adherent and coherent oxide was formed. This effectively reduces the rate of oxidation [15]. EDX analysis shows that the scale of bare SN 600 contains Cr in large amount along with Ni, Mn, O and small amount of Fe which led to the formation of oxide of Cr, Ni and spinels of Ni-Mn, Ni-Cr whereas the scale of Cr_3C_2 -NiCr coated SN 600 contains Cr in large amount along with Ni, O and small amount of C which led to the formation of Cr_2O_3 , Cr_{23}C_6 , Cr_7C_3 and spinel of Ni-Cr. These results were further supported by XRD (Fig. 8 and Fig. 9) and hence confirmed the presence of Cr_2O_3 , NiCr_2O_4 , NiMn_2O_4 , NiO and Fe_2O_3 in bare SN 600 and Cr_{23}C_6 , Cr_2O_3 , Cr_7C_3 and NiCr_2O_4 in Cr_3C_2 -NiCr coated SN 600 respectively. Cross-sectional analysis of bare SN 600 shows the outward diffusion of Mn. It was also observed that Cr and O having higher concentration at the interface of oxide layer and substrate. This was further supported by the mapping (Fig. 12) which shows that the concentration of Cr and O is higher at the interface of the oxide and substrate and at the bottom of the oxide while on the top layer Mn, Fe and Ni along with oxygen are present which confirms the formation of XRD phase NiMn_2O_4 , Fe_2O_3 and NiCr_2O_4 which helps in providing the oxidation resistance. This is due to the formation of spinels having very less diffusion coefficients of cations and anions than those in their parent oxides [15]. In Cr_3C_2 -NiCr coated SN 600, formation of Cr_2O_3 , NiCr_2O_4 along with the carbide is indicated by the peaks of Cr_{23}C_6 and Cr_7C_3 as shown in XRD. Formation of carbides further led to reduction in content of Cr in the NiCr matrix. This was reported in literature that NiCr phase followed the classical oxidation mechanism to form NiO scale under Cr_2O_3 layer. But in case of Cr_3C_2 -NiCr coated SN 600, since the concentration of Cr and oxygen is higher throughout the oxide as shown in mapping (Fig. 13) therefore mainly Cr_2O_3 layer was formed. As it can be observed in cross-sectional analysis thin layer of oxide was observed in coated sample. In a study S. Matthews et al. reported that the carbide phase led to the formation of a thin continuous and adherent Cr_2O_3 scale [21]. Subhash kamal et al. reported in their study that Oxidation resistance of Cr_3C_2 -NiCr coating is solely based on Cr_2O_3 formation, which acts as diffusion barrier for corrosion species [22]. So it can be inferred that the Cr_2O_3 oxide layer was formed on both the bare and coated exposed alloys.

From the present study it can be concluded that Cr_3C_2 -NiCr coating has shown excellent resistance to high temperature oxidation. The formation of Cr_2O_3 and NiCr_2O_4 along with the formation of Cr_{23}C_6 and Cr_7C_3 in the scale on the surface of specimens may have contributed to the better corrosion resistance of the specimens.

Conclusion

1. The D-gun sprayed Cr_3C_2 -NiCr coating has proved to be sustainable at 900°C for 100 cycles of exposure. The weight gain of coated sample was found to be about one-fourth of that as for bare one.
2. The parabolic rate constant K_p for Cr_3C_2 -NiCr coated SN 600 specimen is around 28 times less than that for the bare alloy, which shows the better result by developing the Cr_3C_2 -NiCr coating on superalloy against oxidation.
3. The XRD analysis of coated sample revealed the presence of Cr_2O_3 , Cr_{23}C_6 , NiCr_2O_4 (spinel) and Cr_7C_3 in the scale formed after oxidation of which helped to improve the oxidation resistance.
4. The Cr_3C_2 -NiCr coating is widely used in wear resistant application but here D-gun sprayed Cr_3C_2 -NiCr coating is provided to protect the substrate alloy from corrosion at high temperature and has shown excellence result by sustaining under severe conditions of cyclic oxidation in air.
5. Further work is being carried on hot corrosion in Na_2SO_4 -25%NaCl environment at 900°C under cyclic conditions. This environment is closer to waste incinerator or bio-fuel fired boilers.

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