

Characterisation of 80% Cr₃C₂ - 20 % (Ni-20Cr) Coating and Erosion Behaviour

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Abstract - 80 % Cr₃C₂ - 20 % (Ni-20Cr) powder composition was used to generate coating on T-91 boiler tube steel using high velocity oxy fuel (HVOF) process. This paper focuses on characterization of the coating and will also investigate the mechanism of erosion at different operating temperatures. Microstructure, porosity, surface roughness, coating thickness and microhardness properties of the coating were characterized using the optical microscopy, X-ray diffraction, scanning electron microscopy/energy-dispersive x-ray analysis and electron probe micro analysis techniques. The 80% Cr₃C₂-20% (Ni-20Cr) coating possesses thickness 325 μm, surface roughness value 5.206 (Ra), average microhardness value 862 Hv and 1.8% porosity. The uncoated and coated samples were tested in air jet erosion test rig for enhancing the life of boiler tubes and improving the performance of the power plants.

Keywords - Characterisation, Erosion, Microhardness, Porosity and Surface roughness

I. INTRODUCTION

Hot corrosion and erosion is a serious problem in boiler tubes, internal combustion engines, fluidized bed Degradation of components in hot sections of gas turbine, boilers, industrial waste incinerators are mainly due to the high temperature oxidation, hot corrosion and erosion. Superalloys have been developed for high temperature applications but it is not possible for a single material to have different properties to meet the demand of today's industry [1]. These materials are not able to meet the requirements of both the high-temperature strength and the high-temperature erosion-corrosion resistance simultaneously. Coatings provide a way of extending the limits of use of materials at the upper end of their performance capabilities, by allowing the mechanical properties of the substrate materials to be maintained while protecting them against wear or corrosion [2].

Coating can be deposited by thermal spraying (flame spraying, vacuum plasma spray, low pressure plasma spray, high velocity oxy fuel), by sputtering or by evaporation. However thermal spray coatings are generally preferred for industrial applications [3, 4]. High velocity oxyfuel (HVOF) process is a relatively new and rapidly developing technology, which can produce dense coatings with high

hardness and adhesion values, and good erosion, corrosion, and wear resistance properties [5]. HVOF is reported to be a versatile technology and has been adopted by many industries due to its flexibility, cost effectiveness and superior quality of coating produced. This spraying process has been designed to retain the characteristics of the coating.

HVOF has been often applied to deposit high-chromium, nickel coatings onto the outer surface of various parts of the boilers, e.g. tubes to prevent penetration of hot gases, molten ashes and liquids to the boilers tube steels as the preferred erosion protection on the surface of heat exchanger tubes in combustion boilers [6, 7]. Cr₃C₂-NiCr coatings offer greater corrosion and oxidation resistance, also having a high melting point and maintaining high hardness, strength and wear resistance up to a maximum operating temperature of 9000°C [8–13]. So, Cr₃C₂-based coatings have been applied to a wide range of industrial components, including components used in steam and gas turbines [14–23]. The corrosion resistance is provided by NiCr matrix while the wear resistance is mainly due to the carbide ceramic phase [24].

There are a lot of applications where materials work under both erosive and corrosive conditions, such as ball valves, turbine blades in power generation installations, impellers of pumps, etc. In the literature it is possible to find wear-corrosion studies of thermal spray cermet coatings [25, 26–33] where synergism of wear and corrosion is studied in different working conditions (abrasive, erosive and friction combined with saline, alkaline or acid solutions). In all cases, thermal spray coatings showed better protection against wear and corrosion when compared with noncoated substrate (pure metallic surface). HVOF sprayed Cr₃C₂-NiCr coatings are one of the most important candidates for protection of materials from high temperature erosion and have been successfully used to protect pulverized coal fired boiler tubes [34, 35–36]. Cr₃C₂-NiCr can also be used in corrosive environment at service temperatures up to 800°C; its hardness decreases by increasing the temperature above 600°C but exhibits good erosion resistance up to 800°C [35]. Hence, Cr₃C₂-NiCr coatings are ideally suited as protective layers in corrosive environment at elevated temperatures [37]; coatings on steam turbine blades and boiler tubes against erosive wear and corrosion attack [35, 38–39].

Carbides, oxides and cermets have received the majority of interest. In particular, nickel–chromium based coatings containing chromium carbide particle dispersions (hard phase), due to their excellent erosion –corrosion resistant properties. There are several papers in the literature which shows that the absence of pores and cracks (micro and macro) is very important when erosion resistance is required. It is also reported that the wear resistance increases with increase in Carbide content and carbide hardness, the erosion resistance can also be improved by similar approaches [40]. So the present research work is aimed to characterize the HVOF sprayed 80%Cr₃C₂ -20% (Ni-20Cr) (wt. %) coating on T-91 boiler tube steel. The substrate material selected for this study have been provided by Guru Nanak Dev Thermal Power Plant , Bathinda, Punjab (India) and are used for fabricating boilers tube. The coating has been characterized with respect to microstructure, porosity, micro hardness, and phase formation using the techniques of x-ray diffraction (XRD), scanning electron microscopy/energy-dispersive analysis (SEM/EDAX).

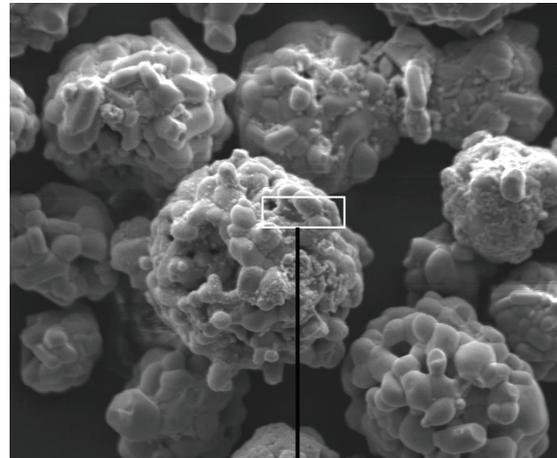
II. DEPOSITION AND CHARACTERIZATION OF THE COATINGS

A. Substrate Material

T-91 boiler tube steel was selected as substrate material. The substrate material selected for this study has been provided by Guru Nanak Dev Thermal Power Plant, Bathinda, Punjab (India) and is used for fabricating boiler tubes This substrate material is used in steam boilers in the chemical industry, power plants, fertilizer units and paper mills etc.. Strips were cut from the boiler tube using milling machine and specimens with dimensions of approximately 22mm × 15mm × 3 mm were prepared from these strips using surface grinder and were cut using slitting wheels. The specimens were polished with SiC papers down to 180 grit, and subsequently were grit-blasted with alumina powders (Grit 45) before development of the coatings by the HVOF process.

B. Coating Formulation

The coating was developed at M/S Metallizing Equipment Co. Pvt. Ltd. (Jodhpur, India) by using commercial HVOF thermal spray systems. Commercially available 80% Cr₃C₂-20 % (Ni-20Cr) coating powder (Agglomerated Sintered, Amperit 586.054, H.C.Starck GmbH, Made in Germany) was used to develop the coating. SEM micrograph showed in Fig. 1 shows that the powder particles have spherical shape. The EDAX analysis validates the coating powder composition. The microstructure observed in the present study is analogous with the findings reported in the studies of S. Wirojanupatump *et al.* [19], J.K.N. Murthy *et al.* [38] and Manpreet Kaur *et al.* [39].



Ck	07.60	Ok	02.92
Crk	81.01	Nik	08.47

Fig. 1 Micrograph and EDAX analysis of 80%Cr₃C₂-20 % (Ni-20Cr) powder

The Hipojet-2100 HVOF system was used for powder spraying. Liquefied petroleum gas (LPG) was used as the fuel. All of the process parameters were kept constant throughout the coating process. The specimens were cooled with compressed air jets during and after spraying. The spray parameters used for the Hipojet-2100 system were an oxygen flow rate of 250 LPM, a fuel (LPG) flow rate of 60 LPM, an airflow rate of 900 LPM, a spray distance of about 20 cm, a fuel pressure of 8 kg/cm², an oxygen pressure of 9 kg/cm², and air pressure of 5 kg/cm² and powder feed rate of 28 g/min.

C. Characterization of the Coatings

The coated sample was subjected to XRD, and SEM/EDAX analysis to characterize the surface and cross-sectional morphology of the coatings. The XRD analysis was carried out with a Diffraction patterns obtained by Bruker AXS D-8 Advance Diffractometer (Germany) with CuKα radiation. A scanning electron microscope (JSM-6610, Jeol, New York) with an EDAX attachment (Oxford, UK) was used for SEM/EDAX analysis. The porosity measurements were made with an image analyzer with Dewinter Material Plus 1.01 software based on ASTM B276. The image was obtained through the attached PMP3 inverted metallurgical microscope with stereographic imaging. To identify the cross-sectional details, the samples were cut across the cross section, mounted in transoptic powder, and subjected to mirror polishing. The coating thickness was measured with a scanning electron microscope. The microhardness of coatings was measured by the Omnitech Microhardness tester. A 300 g load was provided to the needle because the penetration and hardness value was based on the relation $Hv = 1854.4 \times F/d^2$ (where F is the load in grams and d is the mean penetrated diameter in micrometers). Surface roughness of the specimens was

measured by Surface roughness tester Mitutoyo Model SJ 201.

D. High Temperature Erosion Tests

The solid particle high temperature erosion tests were performed on air jet erosion test rig TR-471-M10, as per ASTM G76. The test method utilizes dry compressed air mixed with the erodent particles, which were fed at a constant rate from hopper through erodent feeding system in the mixing chamber and then accelerated by passing the mixture through a converging nozzle. These accelerated particles impacted the specimen kept in the furnace unit consisting of specimen heater and air heater. The specimen could be held at angles of 30° and 90° with respect to the impacting particles using an adjustable sample holder. The details about conditions of erosion testing are given in Table I. The uncoated as well as the coated specimens were polished down to 1µm alumina wheel cloth polishing to obtain similar condition on all the samples before being subjected to erosion run. The samples were cleaned in acetone,

dried, weighed to an accuracy of 1×10⁻⁵ g using an electronic balance, eroded in the test rig for 3 hours and then weighed again to determine weight loss. In the present study standard alumina powder of 50 micron was used as erodent. In general, Erosion resistance is measured using weight loss technique by measuring the weights before and after the test. But at high temperature, weight change measurements leads to flawed results due to oxidation of samples. In order to overcome the limitations of the weight change technique, a different technique was used for the present investigation. Erosion resistance was measured in terms of thickness loss after the erosion testing.

TABLE I EROSION TEST CONDITIONS

Erodent material	Alumina (Irregular shape)
Erodent Specifications	50 micron Al ₂ O ₃
Particle velocity (m/s)	35m/s
Erodent feed rate (g/min)	2 gm/min
Impact angle (°)	30, 90
Test temperature	Room temperature, 200°, 400° and 600°C
Nozzle diameter (mm)	4
Test time (Hrs)	3 Hours

The specimens subjected to erosive wear were analyzed for the characterization of erosion products using surface EDAX.

III. RESULTS AND DISCUSSION

A. Measurement of Coating Thickness

It is impossible to get coatings that are too thick without coating depletion and or crack propagation through the layer.

It is standardised that coating thickness is approximately 300–500 µm in the case of a cermet coating, while metallic alloys such as stainless steel could be deposited getting a final thickness of 2 mm [41].

The thickness was monitored during the process of depositing the coatings with a Minitest-2000 thin film thickness gauge (Elektro-Physik Koln Company, Germany; precision ±1 µm), to obtain the coatings with uniform thickness. The thickness of the coating for some randomly selected samples has been measured from the SEM micrographs along the cross section of the mounted samples. The SEM micrographs from cross-section showing the thickness of the coating has been shown in Fig. 2. The thickness of the 80%Cr₃C₂ -20% (Ni-20Cr) coatings is found to be 325 µm. The thickness of the coatings has been found to be in the desired range as reported by Jun wang *et al.*, T. S. Sidhu *et al.*, Manish Roy *et al.*, N. Espallargas *et al.* and Hazoor Singh *et al.* in their studies [15, 42-45].

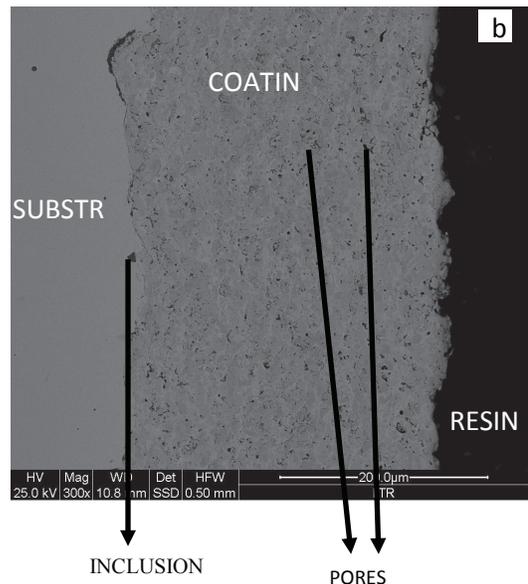


Fig.2 Cross-sectional microstructures of 80% Cr₃C₂-20 % (Ni-20Cr) sample

B. Porosity and Surface Roughness of the Coatings

Thermally sprayed coatings inherently contain porosity in the as-deposited condition. The relative amount of porosity and the average pore size depend strongly on the deposition device and processing parameters [46]. Porosity or voids in the coating microstructure is an important issue in thermal spraying, as due to this physical property corrosion resistance of different thermal spraying coatings differs [42]. The absence of pores and cracks (micro and macro) is very important when erosion or corrosion resistance is required. The coating deposited was found to be dense in nature. Dense coatings usually provide better corrosion resistance than the porous coatings. The porosity of the HVOF sprayed coatings has been reported to be very less. This may be due to

propulsion of high velocity powder particles out of the nozzle towards substrate. Porosity measurements for 80%Cr₃C₂-20% (Ni-20Cr) coating is found to be < 1.8%. The measured value of the porosity is in good agreement with the findings of J.K.N. Murthy *et al.*, T. S. Sidhu *et al.*, Hazoor Singh *et al.*, Michael *et al.* and L. Fedrizzi *et al.* [38,42,45,47-48].

Surface roughness is another important parameter contributing in higher erosion or corrosion resistance. Surface roughness (Ra) value of each coating has been evaluated by Surface roughness tester Mitutoyo Model SJ 201, the value of surface roughness for 80% Cr₃C₂-20% (Ni-20Cr) coating is 5.206 μm. Surface roughness values of Cr₃C₂-NiCr coatings has been found similar with the findings of N. Espallargas *et al.*, L. Fedrizzi *et al.*, Manish Roy *et al.* and Bin yin *et al.* [44, 48-50].

C. Microhardness of the Coatings

In many aggressive environments, protective coatings may have to encounter the problems of erosion-corrosion degradation. The softer coatings are more susceptible to erosion-corrosion mode of degradation (Sidhu *et al.* 2006). Microhardness of the coatings was measured by 300-gram load provided to the needle for penetration. The microhardness data of the coatings has been compiled in Fig. 3, which shows microhardness profiles along the cross section of the coatings as a function of distance from the coating- substrate interface. The microhardness of the substrate and 80% Cr₃C₂ -20% (Ni-20Cr) coating was found to be in the range of 233 – 399 Hv and 873-902 Hv respectively. As indicated by the profiles, the maximum value of the hardness has been achieved by 80%Cr₃C₂-20%(Ni-20Cr) coating of the order of 902 Hv. The microhardness of coating is much higher than the substrate. Further, an increase in the microhardness of the substrate has been observed near the coating-substrate interface in both cases which may be attributed to the work hardening by sand blasting prior to coating as suggested by Sunderajan *et al.* [51]. The partial effect of High velocity striking of powder particles may also have resulted in surface hardening Hidalgo *et al.* [52]. Hardness is the most frequently quoted mechanical property of the coatings. As evident from the profile shown in Fig. 3 the coating exhibited nearly uniform hardness values on all the substrates. The interface of substrate and coating shows higher value of microhardness, whereas the hardness of the coating is much higher than the substrate. The measured values of microhardness are in good agreement with the findings of Scrivani *et al.*, Wirojanupatump *et al.*, Mathews *et al.*, Murthy *et al.*, Manish Roy *et al.*, Bin Yin *et al.*, Zorawski *et al.*, Wang and Lee, Wang and Shui, Wang and Lee, Hawthorne *et al.*, Uusitalo *et al.* and Sidhu *et al.* [16,19,32,38,49-50,53-59]

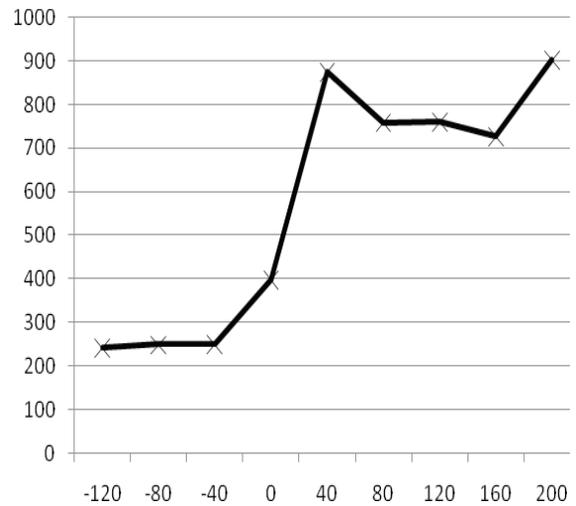


Fig. 3 Microhardness of the coatings and substrate

D. X-Ray Diffraction Analysis

The HVOF sprayed as coated specimens were subjected to XRD analysis obtained by Bruker AXS D-8 Advance Diffractometer (Germany) with CuK α radiation. The specimens were scanned in 2 θ range of 10 to 110° and the intensities were recorded at a chart speed of 1 cm/min and with Goniometer speed 1o/min. The diffractometer interfaced with software provides 'd' values directly on the diffraction pattern. The XRD of powders indicated presence of Ni, Cr, C, NiC, Cr₃C₂ phases. The as coated samples prepared by HVOF spraying technique indicated Cr₂₃C₆, Cr₇C₃ and Cr₃Ni₂ phases along with Ni, Cr, C, NiC, Cr₃C₂ phases. This is expected in case of thermal spray coating deposition, which involves rapid cooling of the molten particles onto the relatively cooler substrate material and results in the formation of non-equilibrium and metastable phases in the as-sprayed coating. Similar phases have also been reported by Sidhu *et al.*, Kamal S. *et al.*, M. Kaur *et al.* and Chatha *et al.* [39, 60-62].

E. Surface SEM/EDAX Analysis of the coatings

Figure 4 depicts the SEM morphology and EDAX of the surface of the as sprayed coatings. It can be observed from the microstructures of the Cr₃C₂-NiCr coatings that they have almost uniformly distributed irregularly shaped fine grained microstructure. Surface SEM of coatings also indicated presence of melted, partially melted and unmelted particles, which are identified in the coating by their size and surface morphology as suggested by Dent *et al.* [63]. The coating-substrate interface shows no gaps or cracks, which is a characteristic feature of good adhesion between the coating and the substrate. Micro-structures observed in present study are almost analogous to those reported by Kaur *et al.*, Sundararajan *et al.*, Sidhu *et al.*, Kamal *et al.*, Dent *et al.* and Mahesh *et al.* in their studies of HVOF-sprayed coatings [39, 51, 60-61, 63-64]. The dark phase contains a little higher amount of Cr than white phase. However both the phases

have been found rich in chromium. The same was confirmed by the EDAX analysis of the surface of Cr₃C₂-NiCr coatings shown in Fig.4.

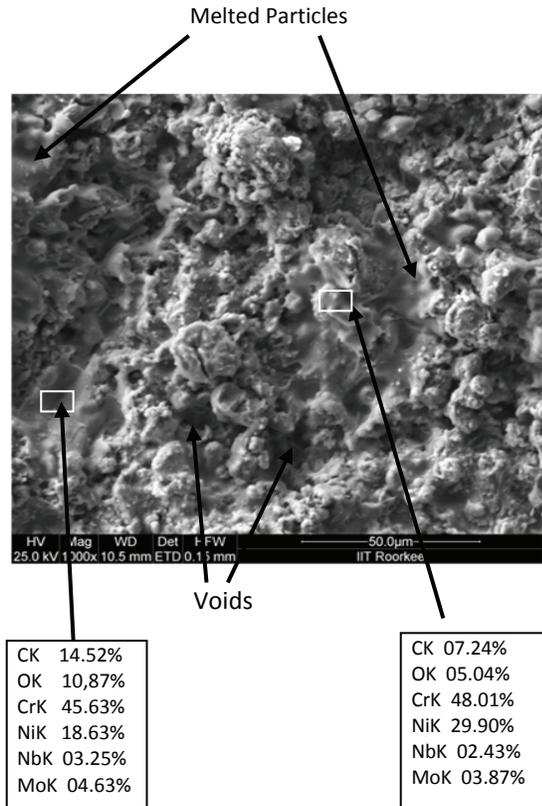


Fig. 4 SEM and EDAX of the surface of 80% Cr₃C₂ -20% (Ni-20Cr) applied on coated steels

F. Cross-Sectional SEM/EDAX Analysis of the Coatings

As sprayed coated samples were cut across the cross-section using a slow speed diamond cutter and mounted in transoptic mounting resin and were mirror polished by using different grades of SiC papers and then they are subjected to cloth polishing with 3µm diamond suspensions followed by a final step of 0.05µm alumina suspensions for metallurgical examination. SEM morphology and EDAX of the as sprayed coating surface shown in Figs. 5 (a) and (b), shows that the microstructure of the coating has almost uniformly distributed irregularly shaped fine grained microstructure.

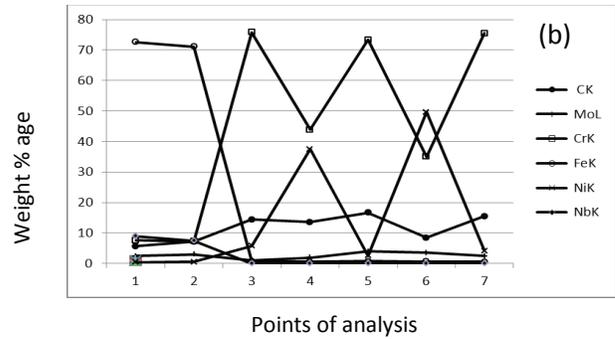
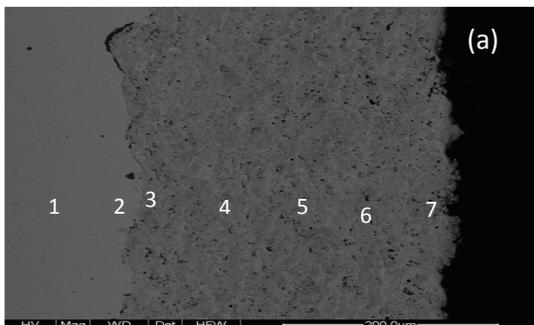


Fig. 5 (a) Cross-sectional microstructure and (b) point analysis of 80%Cr₃C₂ -20% (Ni-20Cr) of as coated sample

It can be inferred that the coating has uniform and dense microstructures and exhibits layered morphologies due to the deposition and resolidification of molten or semimolten droplets. Surface SEM of coating also indicated presence of melted, partially melted and unmelted particles, which are identified in the coating by their size and surface morphology. However, only a limited number of unmelted particles can be observed in the microstructures. The coating also possesses some voids and oxide inclusions that are typical characteristics of the HVOF sprayed coating. The dark phase contains little higher amount of Cr than white phase. However both the phases have been found rich in chromium. The same was confirmed by the EDAX analysis of the surface of coating shown in Fig. 6(b). The coatings have a dense structure with the porosity (less than 1.8%) randomly distributed in the coating. Cross-sectional image shows typical lamellar structures and flattened splats. The coating microstructure consists of metallic binder NiCr (bright area) and CrC (dark area). Similar results have been reported in the studies carried out by Manish Roy *et al.*, Fedrizzi *et al.*, Bin Yin, Zhang *et al.* and Li *et al.* [43, 48, 50, 65-66].

FE-SEM /EDAX analysis was carried out at different point of interest along the cross section of as coated samples of 80%Cr₃C₂ -20% (Ni-20Cr) as shown in Fig. 5 (a) and (b). The coatings indicated the uniform distribution of Cr and Ni.

G. X-Ray Mapping

HVOF sprayed 80%Cr₃C₂ -20% (Ni-20Cr) as coated samples was cut and mounted in transoptic mounting resin, mirror polished and silver pasted between samples and the stubs in order to have conductivity and thereafter gold coated to facilitate X-Ray mapping analysis shown in Fig. 6 shows different elements present. X ray mapping of as sprayed coated sample shows presence of Fe as main element in substrate and the uniform distribution of Ni and Cr particles in coating with Cr being the dominant element. In the coated sample Fe has been found to be restricted to the substrate steel only, which show that inter-diffusion of the base steel elements to the coating has not taken place, which is a favorable feature for a good coating-substrate system. Small amount of oxygen is found in the outer regions of the coating.

The coating is mostly composed of nickel and chromium as both co-exist throughout the thickness of the coating.

H. Thickness Loss Due To Erosion

It can be analyzed from Fig. 7 that the solid particle erosion of the uncoated T-91 substrate is higher at 30° impact angles, which was behaving in a ductile manner. Hence while considering the effect of impact angle it can be inferred that at all the temperatures the thickness loss of uncoated T-91 substrate at 90° impact angle is lower than that at 30°. However, while considering the effect of temperature, the thickness loss of substrate increases with increase in temperature. At 600° Celsius sample temperature the thickness loss of uncoated substrate is 1.9 times as compared with thickness loss at room temperature for 30° impingement angle, whereas for 90° impingement angle the thickness loss of the uncoated substrate at 600°C sample temperature is almost 2.4 times than that at room temperature.

It was observed from Fig. 8 that the thickness loss of the 80% Cr₃C₂ - 20% (Ni-20Cr) coated T-91 boiler tube steel was lower at 30° impact angle and increases with increase in temperature. The thickness loss for 30° impingement angle at 600°C is 1.3 times greater than that at room temperature, whereas for 90° impingement angle it is 1.5 times.

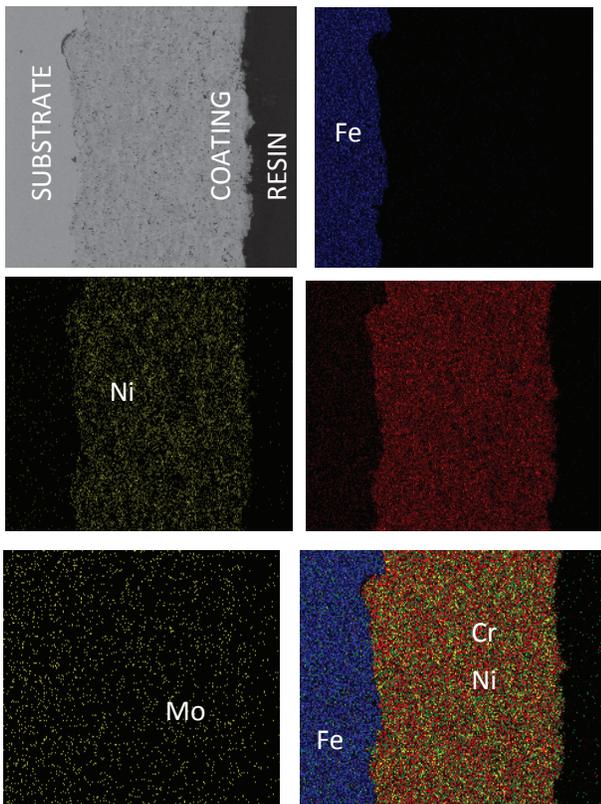


Fig. 6 X-Ray elemental mapping of as coated 80%Cr₃C₂-20% (Ni-20Cr) sample

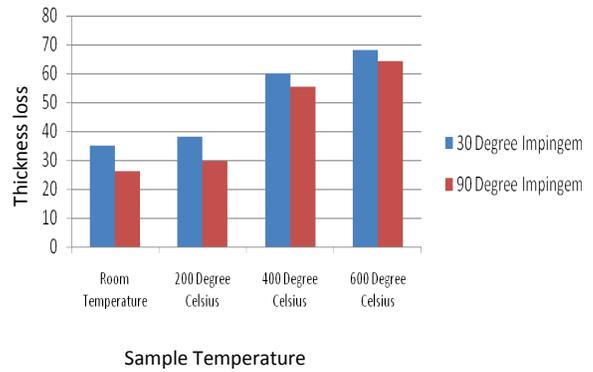


Fig.7 Thickness loss of uncoated T-91 at different sample temperature and impingement angles during erosion testing

Also it can be clearly observed that thickness loss for 90° impingement angle at 600°C sample temperature is comparatively much greater than for 30° impingement angle at same temperature, whereas at all other temperatures this difference is comparatively less.

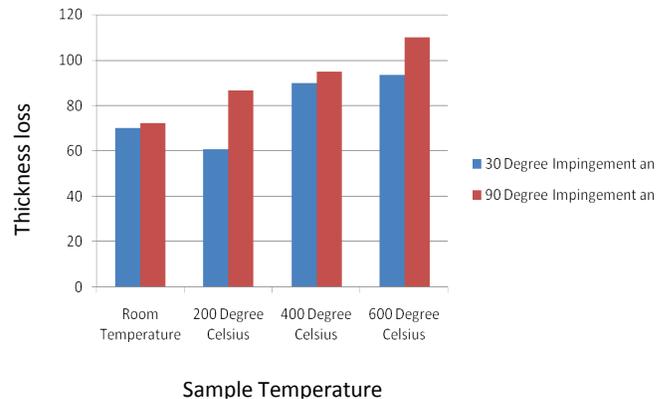


Fig. 8 Thickness loss of the 80% Cr₃C₂ - 20% (Ni-20Cr) coated T-91 boiler tube steel at different sample temperature and impingement angles during erosion testing

The solid particle erosion rate of the uncoated substrate steels as shown in Fig. 8 indicated maximum erosion took place for the samples tested at 30° impact angles, whereas thickness loss was lesser when the samples were tested at 90° impact angle. Such behavior is typical of a ductile material as proposed by Wang *et. al.*, Murthy *et al.*, Wang and Shui, Mishra *et.al.*, and Sidhu *et.al.* [35, 38, 55, 67-68]. The material subjected to erosion initially undergoes plastic deformation and is later removed by subsequent impacts of the erodent on the surface. The generalized behavior of the uncoated T-91 boiler tube steel was ductile in nature.

It has been observed in general that all the coated T-91 boiler tube steel samples showed higher thickness loss than that of uncoated T-91 steel. By contrast, the coatings exhibited greater thickness loss at a steep impact angle (90°) than at a shallow impact angle (30°), the coatings were deemed “brittle” materials as shown in Fig. 9. For HVOF coated 80%

Cr₃C₂ -20% (Ni-20Cr) coating, particle impact angle had a noticeable effect on thickness loss whereby thickness loss increased with increase in impingement angle. The 80% Cr₃C₂ -20% (Ni-20Cr) coatings at 300 impact angle have shown decrease in thickness loss when the sample temperature was increased from room temperature to 200°C, with continuous increase in the thickness loss values afterwards at 400°C and 600°C as shown in Fig. 9. The initial decrease in thickness loss between room temperature and 200°C might be due to an increase in the fracture toughness of the Cr₃C₂- NiCr coating. The increase in thickness loss between 400°C and 600°C was due to a decrease in flexural strength of the Cr₃C₂- NiCr coating as suggested by Wang and Luer [35].

IV. CONCLUSIONS

- 80% Cr₃C₂ -20% (Ni-20Cr) powder was successfully deposited by HVOF spray technique giving dense, uniform, and adherent coating free from surface and cross sectional cracks with porosity less than 1.8%.
- The presence of high carbide content may be the reason for the high microhardness values in coatings as compared with the substrate.
- For uncoated samples thickness loss at 30° is greater than that at 90° impingement angles. The overall ductile behavior has been revealed by uncoated T-91 boiler tube steel. Whereas the coating has shown brittle behaviour with greater losses at 90° degree impingement angle.
- The initial decrease in thickness loss between room temperature and 200°C might be due to an increase in the fracture toughness of the Cr₃C₂- NiCr coating. The increase in thickness loss between 400°C and 600°C was due to a decrease in flexural strength of the Cr₃C₂- NiCr coating.

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