Investigation of Wear Resistant Coatings Deposited on High Chromium Iron Applicable in Hot Rolling

Hardeep Singh[1*], Parvinder Singh Sandhu[2], Balwinder Singh[2]

[1*]M.Tech Scholar, ICL group of colleges, Haryana, India

[2] Faculty, Department of Mechanical Engineering, ICL group of colleges, Haryana, India.

Email : [1*]erhsingh21@gmail.com, [2]ersandhu@gmail.com, [3]balwinder.singh@iclhitech.com

Abstract

The rolling process is one of the oldest and most widely used processes in metal forming industry. Productive Lifetime of Rolls used in the rolling process is an important factor for product quality and cost of the product. Surface Wear is one of the major factors which decide the product quality, life of the roll. Surface coating is way to extending the lifetime of the rolls used in rolling mill. In current research WC- 12%Co + 10% Ni, Cr_2O_3 + 10% Ni and Al_2O_3+ 3%TiO_2 surface coatings were developed on high chromium iron roll material using detonation spray technology. Coatings were evaluated on high temperature tribometer using application parameters coatings were characterized before and after experimentation. Drastic control in wear was observed by application of coating in comparison to uncoated specimens. Cr_2O_3 + 10% Ni coating showed lowest specific wear rate and found suitable.

Keywords: Surface coating, high chromium iron, WC- 12%Co + 10% Ni, Cr_2O_3 + 10% Ni, Al_2O_3+ 3%TiO_2, detonation spray, high temperature tribometer.

1. Introduction

The rolling process is one of the oldest and most widely used processes in metal forming industry. It accounts for about 70 percent of all metals produced by a metal working process. The process holds potential for close control of the final product shape and properties. Productive Lifetime of Rolls used in the rolling process is an important factor for product quality and cost of the product. The lifetime of roll is limited by wear, thermal and mechanical fatigue, and plastic deformation. Surface Wear is the major way, which lowers the lifetime of roll. Surface wear damage of roll depends on various factors like roll material property, manufactured component material property, applied forces and environment. In hot rolling surface wear becomes more complex due to formation of tribochemical layers, oxidation and thermal softening.

Surface engineering is way to extending the lifetime of the rolls used in rolling mill. Among various surface engineering techniques surface coating is recent and shown good results in wear and corrosion resistance. Surface coating involves various factors like surface coating methods, surface coating feedstock, properties, microstructure of coatings, working conditions etc. Therefore in the present work, development and investigation of surface coatings was performed on High chromium iron (HiCr) material used in finishing roll in hot rolling process. Three different coating powders were deposited using detonation spray technology. In depth Characterization of the powder and as-sprayed coating was done by X-ray Diffraction (XRD) and Scanning electron microscope/Energy Dispersive X-Ray Spectroscopy (SEM/EDS). The study of friction and wear characteristics was performed at room temperature and elevated temperature of uncoated and surface coated specimens of HiCr on Pin-on-disc tribometer. Coefficient of friction and weight loss was evaluated.

2. Experimental work

2.1 Preparation of Substrate material

Substrate material in present work is commercially available HiCr iron, used for making finishing roll in many hot rolling industries in India. The nominal composition of HiCr iron is given in table 1.1. The HiCr iron was machined into cylindrical pins having circular cross-section of 7 mm diameter and length 50 mm to perform wear test on pin-on-disc tribometer. The faces of the pins were grinded followed by polishing with emery papers to make surface flat. Sketch of pin specimen is shown in figure 1.1 (a) & pictorial view of prepared sample is shown in figure 1.1 (b)

a)

Figure 1 a) Sketch of Pin sample for wear test on pin-on-disc tribometer
b) Pictorial view of prepared samples

Table 1 Nominal composition of HiCr

Material	C	Ni	Cr	Fe
HiCr	2.5-3.0	0.3-0.8	15-18	Bal.

2.2 Development of coating

2.2.1 Formulation of coating powder and its characterization

Following coating powders were explored for their potential in hot forming conditions.

1. **WC-12%Co + 10% Ni (Nickel):** WC-12%Co powder cemented carbides are widely used by various thermal spray processes to deposit protective coatings where abrasion, erosion and other forms of wear exist with high density. WC+ 12 % Co exhibits low corrosion resistance. 10% Ni is added to enhance corrosion resistance in addition to increasing the flow ability of coating during deposition, and increase adhesion strength of coating. The powder was prepared by mixing commercially available WC-12%Co powder with 10% Ni powder by weight and mixing is done using ball milling for 8 hours.
2. **Cr_2O_3 (Chromium oxide) + 10% Ni (Nickel).** Cr_2O_3 is the hardest oxide that also exhibits low friction coefficient, high wear and corrosion resistance, and good optical and

adiabatic characteristics. The powder was prepared by mixing commercially available Cr_2O_3 Powder with 10% Ni powder by weight and mixing is done using ball milling for 8 hours.

3. **Al_2O_3-3%TiO_2:** Powder when sprayed using the Detonation Spray Process it produce coating which are very hard, Dense and exhibit good abrasive wear resistance under normal rpm and dynamic load. The powder was available commercially.

The powders were characterized by X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM) was done to study the phases and morphology of powder particles.

2.2.2 Coating Deposition

The three coating powders were successfully deposited using detonation gun process at SVX Powder M Surface Engineering Private Limited, H-14C Surajpur, Greater Noida, UP, India.

Prior to the coating deposition, the sample pins were grit blasted by alumina grit using industrial methodology to thinly remove the metal surfaces and create a rough contour with the surface roughness of about 8-10 µm Ra on the surface necessary for the adhesion of the coating followed by cleaning and preheating. The process parameter employed during deposition of coating is tabulated in table 2.

Table 2 Spray parameters as employed during Detonation spraying

Fuel Gas	Oxygen and Acetylene
Carrier gas	Nitrogen
Pressure of fuel gas (Oxygen)	0.2MPa
Pressure of fuel gas (Acetylene)	0.14 MPa
Pressure of Carrier gas (Nitrogen)	0.4 MPa
Pressure of Carrier gas (Air)	0.4 MPa
Consumption of gas per shot (oxygen)	$27x10^{-5}m_3$
Consumption of gas per shot (Acetylene)	$25x10^{-}5m_3$
Consumption of gas per shot (Nitrogen)	$5x10^{-5}m_3$
Consumption of gas per shot (Air)	$5x10^{-}5m_3$
No. of shot per second	2
Water Consumption rate	25 LPM
Consumption of powder per shot	0.05-0.02 GPS
Stand-off distance	150-188mm
Firing rate	1-10 Hz
Diameter of accelerating portion of barrel (coating coverage)	0.025 m
Coating thickness per shot	15-20 µm
Power supply from mains	430 V (50-60HZ)
Maximum substrate temperature	150^{o}C
Name of D-Gun	Awaaz detonation spray coating system

Manufacturer	International Advance Research centre for powder metallurgy and new materials, Hyderabad

2.3 Characterization of As-sprayed coatings

The as-sprayed coatings were characterized by X-Ray Diffraction (XRD), Field Emission-Scanning Electron Microscopy (FE-SEM) and Energy Dispersive Spectroscopy (EDS). Also porosity, hardness, bond strength and surface roughness were evaluated for as-sprayed coatings.

2.4 Wear Test

Wear test were performed under dry sliding conditions using a high-temperature pin-on-disc machine equipped with a computerised data acquisition and control [Model: Wear and Friction Monitor Tester TR-20 LE PHM 400] shown in figure 2. The uncoated, heat treated and coated pins were held stationary against the counter face of a Rotating Disc made of EN-31. EN-31 steel is a plain carbon iron; case hardened 62 to 65 HRC. Composition of the disc material is given in Table 3.

The samples were polished with emery papers for roughness value less than 1 µm. Pin sample were cleaned and weight was measured before and after experimentation with a precision of 0.1 mg using microbalance before mounting in pin holder on pin-on-disc tribometer. High temperature experimentation was performed after desired disc temperature was attainted

Table 3 Composition of EN-31

Material	C	Si	Mn	P	S	Cr	Fe
EN 31	0.9-1.20 %	0.1.-0.35%	0.30-0.75%	0.04 0%	0.040 %	1-.60%	Bal.

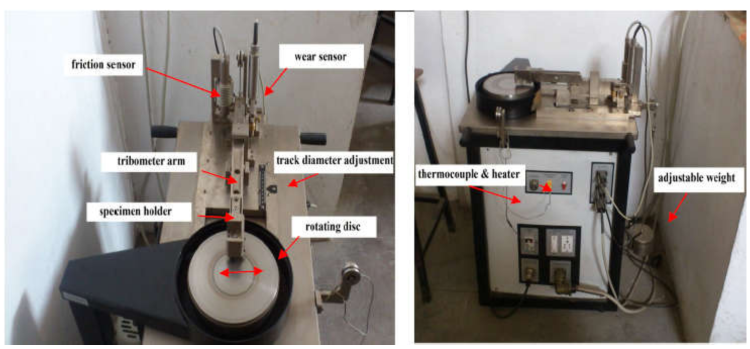


Figure 2 Pictorial views for High temperature pin–on-disc tribometer rig.

2.4.1Parameters for experimentation

The test parameters in high temperature pin-on-disc tribometer wear studies were selected on the basis of typical values encountered in forging industry. Various input and output parameters in pin-on-disc experimentation are shown in table 3. The final parameters used in wear testing are given in table 4.

Table 3 Input and output parameters

Input parameters	Output parameters
Load (N)	Wear
Sliding Distance (m)	coefficient of friction
Sliding Velocity (RPM)	Friction force (N)
Wear track diameter (m)	
Pin Diameter	

Temperature (ºC)

Table 4 Test parameters

Test Parameter	Values
Load	40N
Temperature	400C, 4000C and 6000C
Duration	30 minutes
Sliding distance	1000 m

The samples after wear test were inspected to understand the wear behavior, the worn out samples were studied with the help of field emission-scanning electron microscope.

3. Results

3.1 Characterization of coating powders

Figure 3shows the SEM morphology of the coating powders. The morphology of WC-12%Co + 10% Ni powder was found to be spherical (Figure 3 (a)). Particles were of irregular size and shape. While the morphology of the Cr_2O_3 + 10% Ni and Al_2O_3-3%TiO_2 was found to be irregular and sharp edged (Fig. 3 b & c). The XRD diffractograms for the coating powders were shown in Figure 4. XRD analysis of WC-12%Co + 10% Ni powder WC, Co were found in strong intensity phases and Ni was present in medium intensity. In Cr_2O_3 + 10% Ni powder diffractogram Cr_2O_3 was found in string intensity and Ni in medium intensity phase. In Al_2O_3-3%TiO_2 powder Al_2O_3 was found to be in strong intensity phase and TiO_2 in low intensity phase.

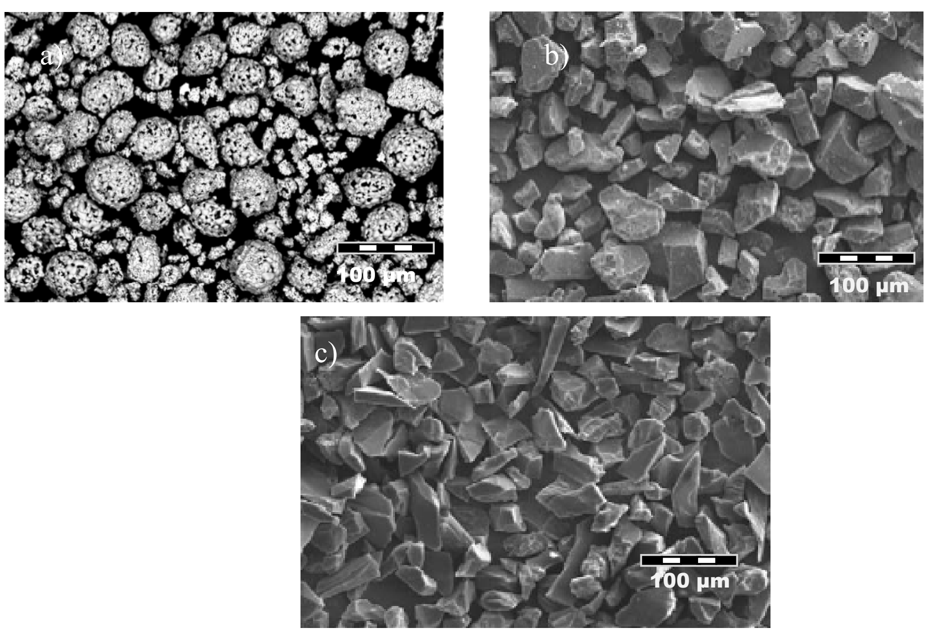


Figure 3SEM morphology of composite Coating powder (a) WC-12%Co + 10% Ni (b) Cr_2O_3 + 10% Ni (c) Al_2O_3-3%TiO_2

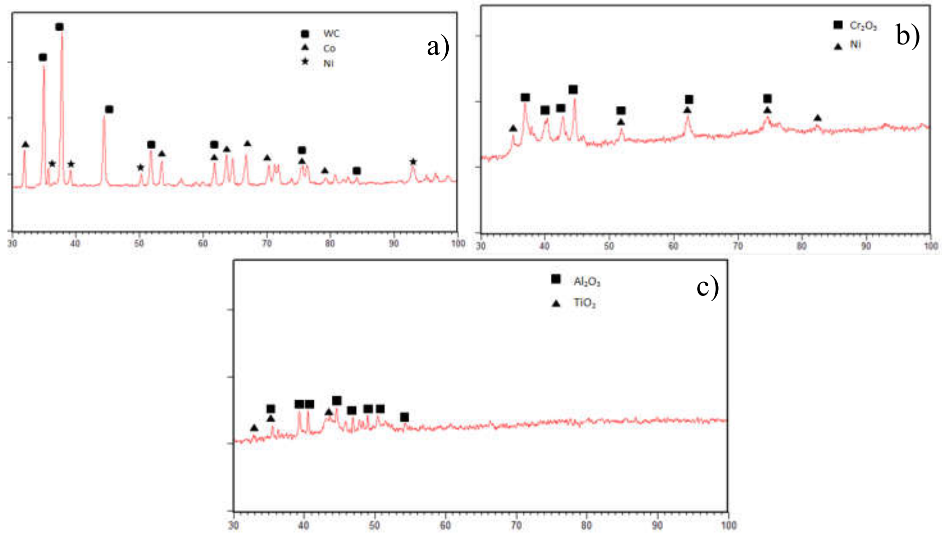


Figure 4 XRD diffratograms of the feedstock (a) WC-12%Co + 10% Ni (b) Cr_2O_3 + 10% Ni (c) Al_2O_3-3%TiO_2

3.2 Characterization of As-sprayed coatings

Table 5 shows the value the Coating thickness, bond strength, porosity and surface roughness. The value of surface hardness is reported in table 6.

Table 5 Characteristics value for coated specimen

Characteristics	WC-12%Co + 10% Ni (C1)	Cr_2O_3 + 10% Ni (C2)	Al_2O_3-3%TiO_2 (C3)
Average Coating thickness (µm)	100-150	100-150	100-150
Bond Strength (MPa)	80.1	60.20	72.21
Porosity (%)	1.8	1.6	2.1
Surface Roughness (µm)	5.6	6.2	6.12

Table 6 Surface Hardness values

Characteristics	WC-12%Co + 10% Ni (C1)	Cr_2O_3 + 10% Ni (C2)	Al_2O_3-3%TiO_2 (C3)	Uncoated
Coating Hardness (vicker hardness HV 0.3 @VHN)	1110	950	785	300

3.2.1 XRD of As-Sprayed

The X-Ray diffractograms for detonation sprayed WC-12%Co + 10% Ni, Cr_2O_3 + 10% Ni and Al_2O_3-3%TiO_2 powders in high chromium iron are shown in figure 5. In WC-12%Co + 10% Ni sprayed coating WC, W_2C and WCoC, phases were observed in strong intensity phase. Also formation of NiO_2 phase shows oxidation of Ni powder during coating process. In Cr_2O_3 + 10% Ni coating powder Cr_2O_3 and NiO_2 phases were in strong intensity. In

Al_2O_3-3%TiO_2 sprayed coating Al_2O_3 and TiO_2 were observed in strong intensity phase while Al_2TiO_5 was observed in medium intensity phase.

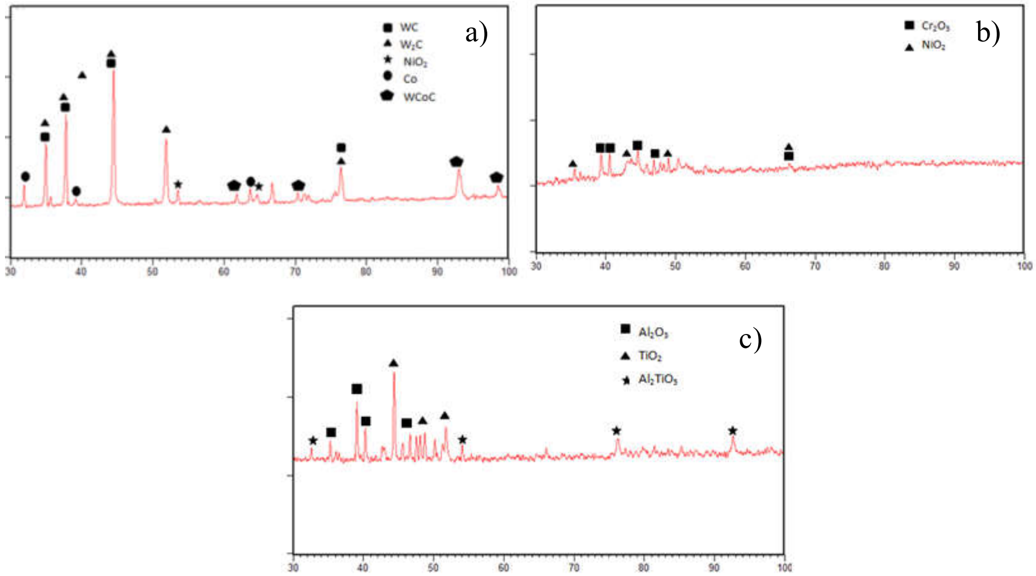
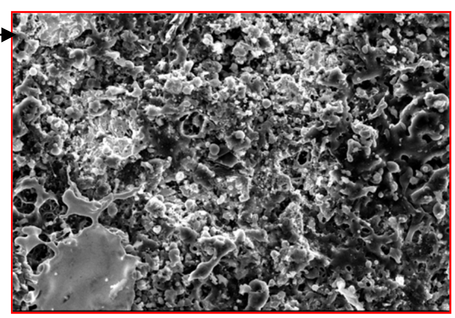


Figure 5 XRD Diffractograms (a) WC-12%Co + 10% Ni (b) Cr_2O_3 + 10% Ni (c) Al_2O_3-3%TiO_2

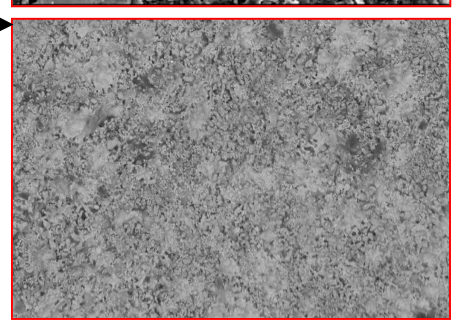
3.2.2 SEM –EDS analysis as-sprayed coating

The SEM micrographs as well as Energy Dispersive Spectrum with elemental composition for detonation sprayed WC-12%Co + 10% Ni, Cr_2O_3 + 10% Ni and Al_2O_3-3%TiO_2 powders in high chromium iron are shown in figure 6. The SEM images shows splat morphology formed due to striking of molten droplets on substrate surface. In WC-12%Co + 10% Ni coating few un-melted particles was seen. Also the coating was observed with black patches which are commonly seen in carbide coatings. Similar morphology was observed in Cr_2O_3 + 10% Ni and Al_2O_3-3%TiO_2 coatings. The coatings were greyish colour, seen commonly in oxide coatings.

Elements	Weight %
W K	38
C K	41
Co K	11
Ni K	06
O K	04
Totals	100%



Elements	Weight %
Cr K	51
O K	39
Ni K	08
Fe K	02
Totals	100%



Elements	Weight %
Al K	39
O K	47
Ti K	14
Totals	100%

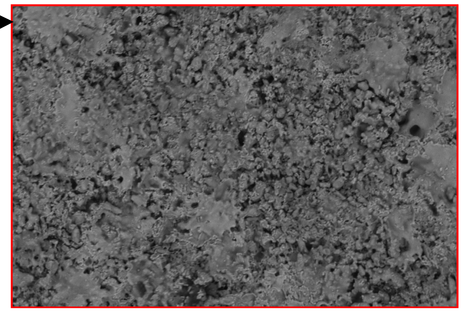


Figure 6 SEM & EDS analysis of As-Sprayed Coatings (a) WC-12%Co + 10% Ni (b) Cr_2O_3 + 10% Ni (c) Al_2O_3-3%TiO_2

3.3 Characterization of Worn out samples

3.3.1 SEM analysis for worn out samples

The SEM morphology of the worn surfaces is shown in figure 7-9, SEM morphology clearly shows the wear tracks formed in experimentation, pullouts, pits, wear debris, oxidation patches formation of edges. SEM morphology indicates more adhesive wear at 40°C as more pull out and pits can be seen while both abrasive wear and adhesive wear at higher temperature it is also observed with increase in temperature abrasive wear rises.

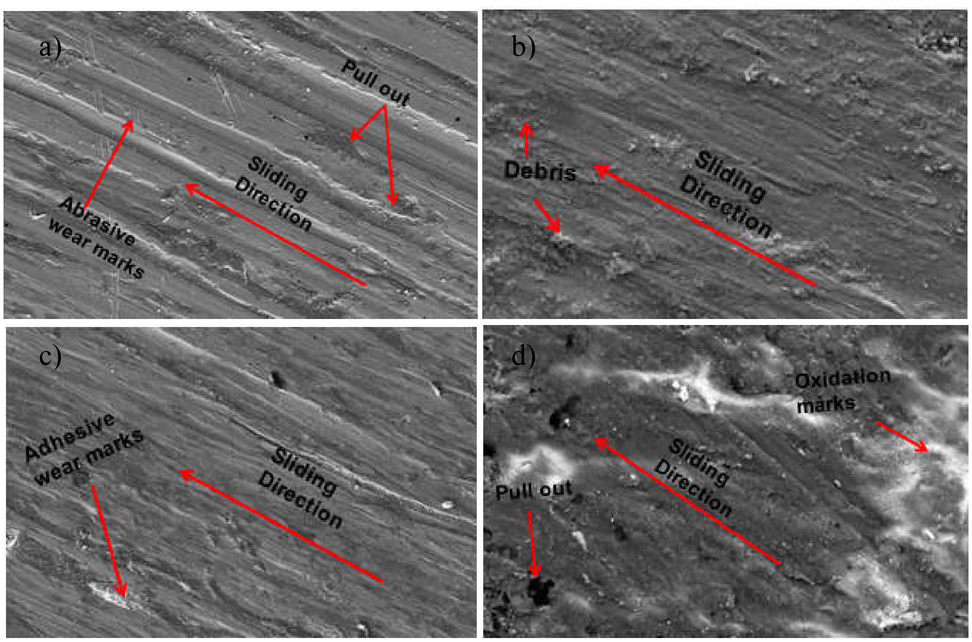


Figure 7 SEM analysis of worn out samples at 40°C (a) Uncoated specimen (b) WC-12%Co + 10% Ni (c) Cr_2O_3 + 10% Ni d) Al_2O_3-3%TiO_2

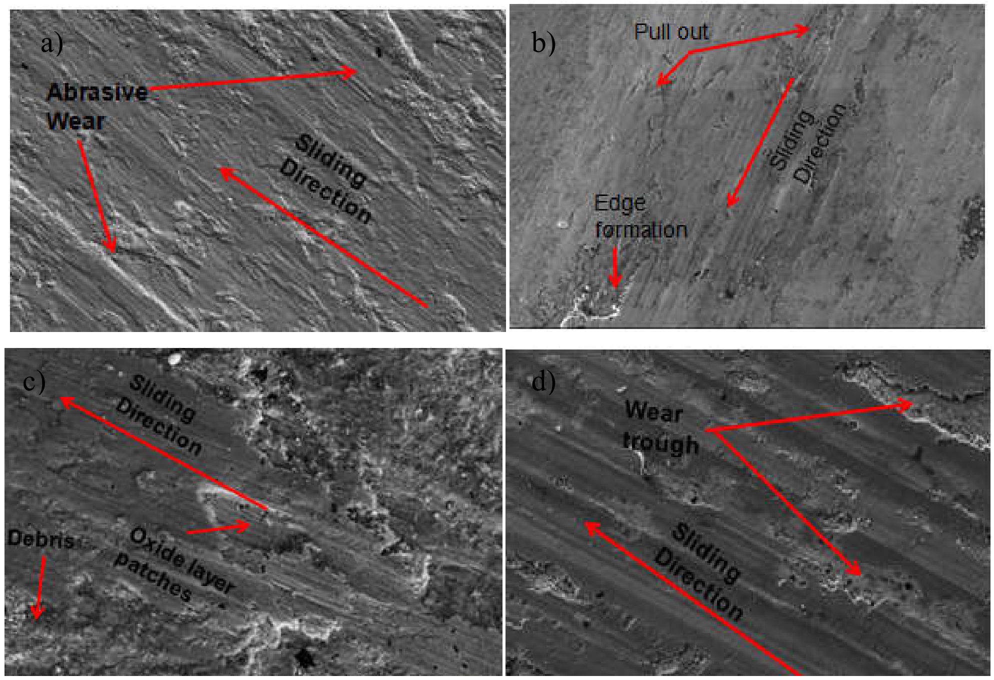


Figure 8 SEM analysis of worn out samples at 400°C (a) Uncoated specimen (b) WC-12%Co + 10% Ni (c) Cr_2O_3 + 10% Ni d) Al_2O_3-3%TiO_2

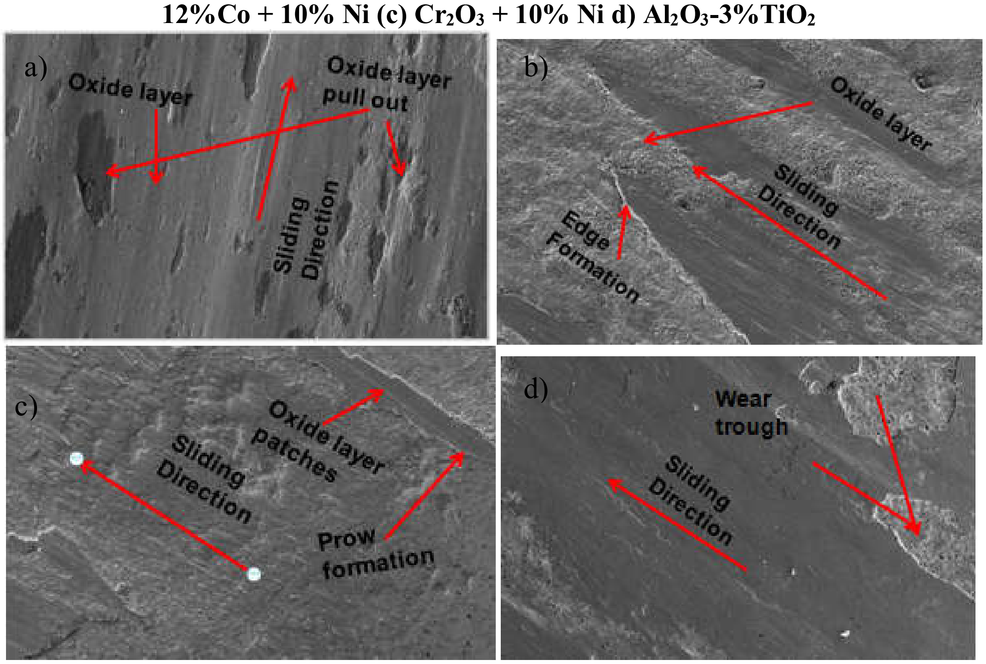


Figure 9 SEM analysis of worn out samples at 600°C (a) Uncoated specimen (b) WC-12%Co + 10% Ni (c) Cr_2O_3 + 10% Ni d) Al_2O_3-3%TiO_2

3.3.2 Friction and Wear Behaviour

Friction Behaviour

The average coefficient of friction plots for uncoated and coated specimens at 40^0C, 400^0C and 600^0C are shown in figure 10. It can be observed that the coated samples have higher coefficient of friction then uncoated specimens. The observation is supported by resistance to wear of coated samples and adhesive nature of coating elements. The high coefficient of friction is also due to hard carbide and oxide elements protruding from the coating surface which difficult to deform and causes resistance to sliding between pin and disc. Coefficient of friction falls with increase in temperature due to softening of disc and formation of oxide layer which act as gliding layer resulting in lowering of friction coefficient.

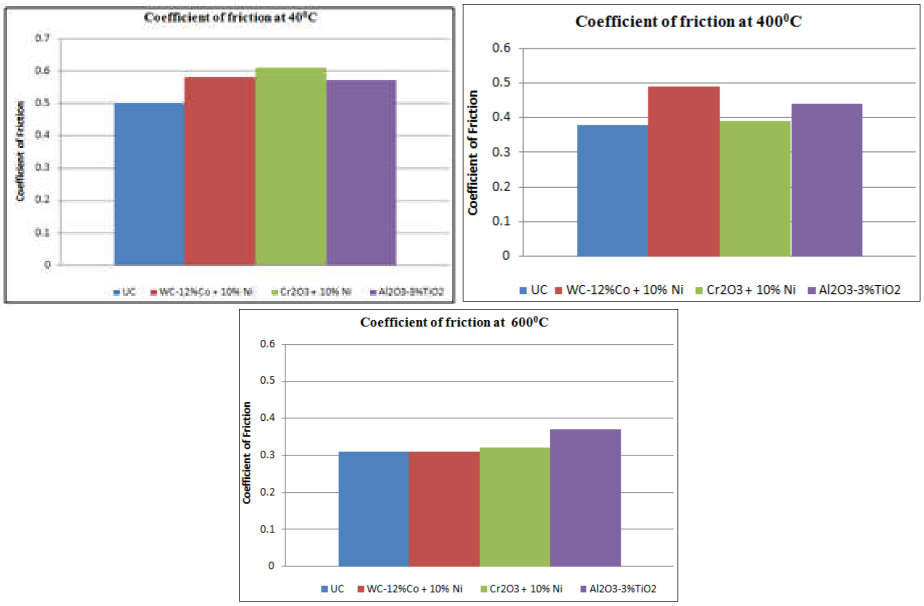


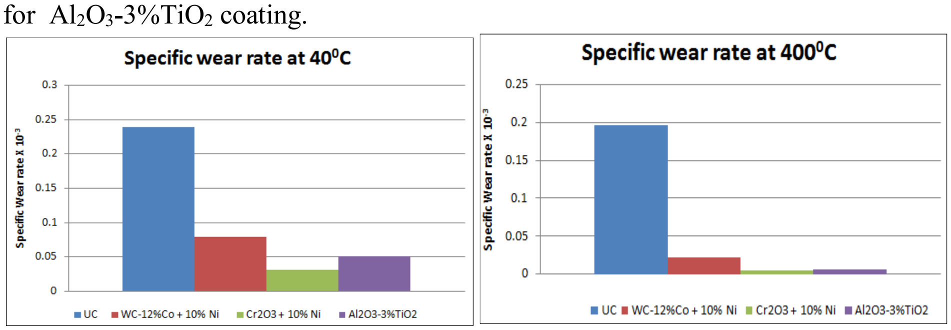
Figure 10 Average coefficient of friction plots for uncoated and coated samples. At (a) 40⁰C (b) 200⁰C(c) 600⁰C

Wear Behaviour

The specific wear rate leads to comparison of wear rate for coated; heat treated and coated sample at different temperature. Specific wear rate was calculated using relation:

$$\text{Specific wear rate} = \frac{\text{Weight loss}}{\text{Density x Distance x load applied}}$$

The plots of specific wear rate at different temperature are shown in figure 11. It is clearly observed that specific wear rate for coated sample is very low in comparison to uncoated and coated samples at all three temperature range. In uncoated specimen high specific wear rate was observed in uncoated specimens. At high temperature the specific wear rate falls due to softening of disc and formation of oxide layer. The oxide layer provides easy gliding between pin and disc. Similar phenomenon of decreasing specific wear rate with increase in temperature was observed among the coated specimens. At room temperature Cr_2O_3 +10%Ni shows the minimum specific wear rate. At 400^{0}C, Cr_2O_3 + 10% Ni and Al_2O_3-3%TiO_2 powder coatings shows minimum specific wear rate. At 600^{0}C WC-12%Co +10%Ni and Cr_2O_3 + 10% Ni coatings shows minimum specific wear rate while slight increase in specific wear rate was observed for Al_2O_3-3%TiO_2 coating.



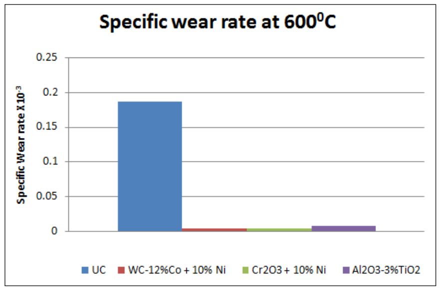


Figure 11 Specific Wear plots for uncoated and coated samples At (a) 40^{0}C (b) 200^{0}C(c) 600^{0}C

4. Conclusions

1. Detonation spray process provides the possibility of deposition of WC-12%Co + 10% Ni, Cr_2O_3 + 10% Ni and Al_2O_3-3%TiO_2 composite powders on high chromium iron.

2 Bond strength of WC-12%Co + 10% Ni was maximum Cr_2O_3 + 10% Ni coating bond strength was found minimum.

3. Specific Wear rate of all the coated specimens was lower than uncoated samples thus coating was found effective in lowering wear of HiCr iron in hot rolling process.

4. Minimum specific wear rate was observed for Cr_2O_3 + 10% Ni coating at all temperature conditions among all the coatings.

5. It is concluded that life of HiCr iron rolls can be enhanced using surface coating technology and the three coating powders used in the current research shows satisfactory results.

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