

TRIBOLOGICAL INVESTIGATION OF TUNGSTEN CARBIDE AND HARD CHROME COATINGS ON EN19 FOR COLD ROLLING APPLICATION

Sajjan Kumar Lal^a, Vivek Gaval^{a*}, Sachin Solanke^a, Ravindra Garmode^b

^aInstitute of Chemical Technology, Mumbai, Maharashtra 400019, India

^bSt. Francis Institute of Technology, Mumbai, Maharashtra 400103, India

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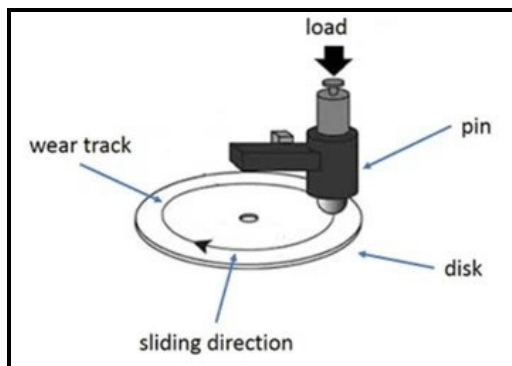
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*Corresponding author
vr.gaval@ictmumbai.edu.in

Graphical abstract



Abstract

In the heavy sheet metal industry, rolls are generally made up of EN19. To enhance the life of the cold rolling mills it can be coated. This paper presents an experimental study to check the effect of alloy coatings such as Hard Chrome and Tungsten carbide on wear resistance and other characteristics of roller base materials EN19 which is used in rolling process of heavy metal industry. In this paper a comparative tribological study of Hard Chrome, Tungsten carbide, EN19 through hardness testing, surface roughness analysis, SEM, and EDS, XRD, sliding wear experiments and statistical study through Design of Experiment has been performed to find out the best material which can increase the life of rolls. From the experiments carried out it has been found that hardchrome has 15.19 times the longevity of the substrate EN19. Tungsten carbide has 19 times the life of EN19 and 1.25 times the life of hardchrome. Tungsten carbide coating gives more hardness and wear resistance than the Hard Chrome and EN19 base material of the roll. Thus, using Tungsten carbide coating on EN19 rolls will improve the life and enhance the longevity of the roll used in cold rolling process in heavy steel industry.

Keywords: Coating; Hard chrome; Tungsten Carbide; Wear; COF (Coefficient of friction)

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1.0 INTRODUCTION

Rolling has a wide industrial application in manufacturing sheets, I-channels, bars, strips etc. In cold rolling process (which usually takes place at room temperature), the thickness of workpiece is reduced and moreover its length increases. In the rolling process rolls exert compressive force on the workpiece to change its cross section. In the rolling process rolls get worn out due to this severe loading and pressure condition.

In modern steel plants automated lines of production are used. The life of a simple roll without coating is approximately 2-3 months. After that it has to be reground in order to reuse but that decreases the dimension of the roll which isn't feasible after a certain extent. Replacing the roll hampers the line of production, which raises the overall price of production. So, there is a need to enhance the life of the rolls.

Different types of coatings/electroplating are used for aesthetics, functional, and industrial applications, resulting in

protection against wear, corrosion, and high temperature oxidation, as well as improved chemical resistance and lubricity [1,2]. Coatings with high hardness, such as nickel, chromium, cobalt, and phosphorus, are widely used in a variety of industrial applications to achieve optimum surface conditions, which improves the material's resistance to wear, erosion, and abrasion [3].

Winarto et al. found that at preheated temperature of 300°C the hardness of H13 steel can be enhanced with tungsten carbide coating. With this coating a strength of 1541 HVN was obtained which led to enhanced wear resistance [4]. Kiilakoski et al. investigated the case of high-speed slurry abrasion and cavitation erosion and found that the coating of WC18NiMoCrFeCo gave a better result in terms of wear resistance [5]. Integran Technologies USA, INC. explored that Hard chromium coating is frequently utilized in both military and industrial applications because it improves workpiece erosion and wear resistance. This hardchrome coating can have

a thickness between 0.25 to 10 mil. This can be done through electroplating method which gives lower COF and higher hardness, improved corrosion and thermal properties etc. [6]. Eriksson discovered that the wear properties of hard chromium and electroless nickel-phosphorus coatings were nearly identical at ambient temperature. On rough surface hard chromium displayed better result whereas on smooth surface electroless nickel-phosphorus showed better result. Substrate bearing capacity has considerable effect on coating performance [7]. Nascimento et al. investigated that Fatigue strength for substrate standard steel AISI 4340 reduced more in chrome electroplating than in WC specimen. The results of WC coating were better than chrome electroplating in the case of wear weight loss tests [8]. Khallaf et al. found that the hardness of $(\text{CrFeCoNi})_{1-x}(\text{WC})_x$ HEAs increased when WC concentration was increased, from 336.41 HV of $(\text{CrFeCoNi})_{1.0}(\text{WC})_{0.0}$ to 632.48 HV of $(\text{CrFeCoNi})_{0.80}(\text{WC})_{0.20}$. The strengthening process is most likely caused by the Cr-rich carbides precipitation and WC particle which are harder in nature. [9]. Pichler et al. investigated that by abrasive wear resistance test maximum wear resistance was observed for tungsten carbide showing highest volumetric loss of 100 mm³ in 6000 rev. And for without tungsten carbide 350mm³ of volume loss was observed. 800 mm³ of volume loss was observed for fine tungsten carbide specimen [10]. Seger investigated that spherical carbides have the highest carbide hardness and highest wear resistance with a small margin to FTC and monocrystalline coatings. Increased carbide amount gives increased wear resistance [11]. Nagentrau et al. studied whether the following conclusions may be reached from analysis of (WC) hardfacing on a carbon steel blade at microstructure level. SEM scans of the hardfacing revealed the coating's carbide and non-carbide regions in great detail. Around the coating zone, various sizes of carbides which have non-uniform distribution can be found. EDS examination indicated that the carbide region includes a high percentage of W, but the noncarbide area has both Fe & W, indicating the presence of a carbide and a binder in proximity. The development of iron WC was proven by XRD analysis to be related to the distribution of melted tungsten carbide electrode amid Fe due to solidification. [12].

Raghav et al. examined the manufactured composites by using XRD, FESEM and EDAX. Co-25C-W compressive strength and microhardness. The compressive strength and microhardness were found to be higher for Co-25C-8W than Co-25C-2W. The compressive strength and microhardness for Co-25C-8W was found to be 92 MPa and 305 HV respectively. Also, Co-25C-8W had higher wear resistance than other trial alloys which was found through tribological study. Then Field Emission SEM revealed the worn-out areas of composites [13]. Phiri et al. investigated that the properties and thin film microstructure of WC-Co largely depended on factors such as post-processing treatment, substrate conditions, process parameters etc. There is a wide application of WC-Co coating such as aerospace bearings where high rate of wear take place [14]. Mekicha et al. found through scratch tests that comparing with uncoated pins, pins which were hard chrome plated displayed lesser material transfer, decreased value of friction and minimized amount of iron fines. These decreased values were due to tribochemistry of the chromium layer [15]. Valentina et al. investigated that the life of steel rod decreases due to corrosion, high temperature application, wear as per its

application. These can be overcome by applying a chromium layer which has high hardness. This hardness level goes on decreasing as penetrated below the surface layer i.e., as the depth of penetration of chrome layer goes on increasing [16].

1.1 Novelty of the Study

Based on the literature review it has been found that tungsten carbide and hardchrome coatings have shown very good results in different applications [4]-[8]. Since hardly any research work has been done earlier on rollers base material EN19 used in heavy steel industry to increase the life of the roll, so main objective of this research is to increase the life of roll used in heavy steel industry by employing tungsten carbide and hardchrome coating materials on base material EN19, thus increasing the productivity of rolling process.

2.0 EXPERIMENTAL PROCEDURE

Coating materials were chosen based on their physical and chemical qualities discovered through a review of the literature. As shown in Figure 1, hard chrome is plated using the Electro Deposition Coating (EDM) method. It is sometimes referred to as the E-coating process. It is comparable to the electroplating procedure. The surface is first cleansed (ultrasonic washing, degreasing, de-rusting, hand cleaning, and so on). Following this, water rinsing is used to eliminate any chemicals utilized during the cleaning process. The surface of the substrate is then activated with chrome salt, allowing the substrate's nature to respond to chrome plating. The workpiece now gets ready to be chrome plated after the surface has been activated. The workpiece is joined to the negatively charged cathode, and Chromium is joined to the positively charged anode. When we apply current to the solution, it is transformed into ions, with chromium ions becoming positive and the substrate becoming negative. As a result, chromium ions are drawn to negatively charged substrates. Chromium is deposited on the substrate, and a coating of chromium is created. Chromic acid or chrome sulfate is used as a solution. After the hard chrome plating is completed, it is thoroughly washed with water. The final inspection follows. Tungsten Carbide Coating is done using Electro-Spark Deposition Method (EDS) as shown in Figure 2.

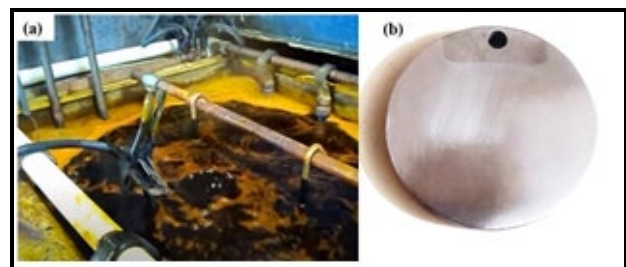


Figure 1 (a) EDM Method, (b) Hard Chrome coating.



Figure 2 Tungsten Carbide coating done on substrate by Electro-Spark Deposition Method.

Surface Roughness of the workpiece and coatings was checked by TR200 which is Surface roughness Tester as per ASTM D3359-09 as shown in Figure 3. Chemical composition of coatings is obtained using EDS process.



Figure 3 Surface roughness checking for substrate and coatings.

The hardness of substrates and coatings were checked with Rockwell Hardness tester as per ASTM E18-07 standards.

Tribological property of coatings are obtained using pin on disc tribometer as per ASTM G99-17 standard as shown in Figure 4. Tribological property wear test (dry test) has been done [20]. As shown in Table 1, samples were manufactured of EN19 substrate with a diameter of 40 mm and a thickness of 6 mm. The substrates have been grinded on grinding machine. One was coated with Hardchrome with a thickness of 100 microns using the Electro Deposition Coating Method described above. The other substrate Tungsten carbide coating was done by Electro-Spark Deposition Method with a thickness of 100 microns. Pin on disc wear test machine was utilized for the dry wear test. This machine was DUCOM, Bangalore, India Make.

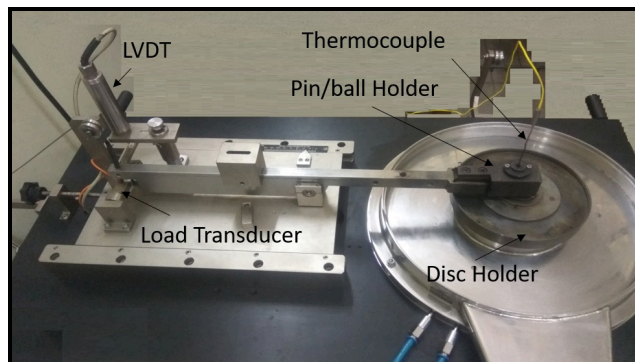


Figure 4 Wear test was performed on a pin on disc wear test equipment.

A load is attached to this machine. The track diameter that will be formed on the specimen by material removal is specified. The velocity rpm setting determines which disc will rotate on which substrate. When the machine is turned on, the disk does rotate at the predetermined velocity rpm on which the specimen is fixed, causing the specimen to move in a circular motion. An extremely hard ball (indenter) of Silicon Nitride Si₃N₄ which of 8 mm diameter is fixed on the lever, and the ball rubs and scratches the workpiece specimen while attempting to scratch and remove the coating material at the predetermined diameter track. As a result, a track is generated which is circular in form and can be seen on the specimen, as illustrated in Figure 5.

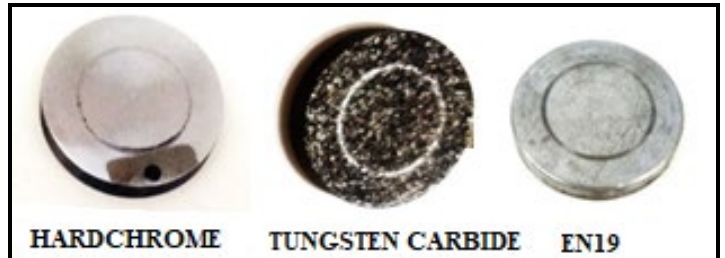


Figure 5 Track formed on specimen after wear test (Hardchrome, Tungsten Carbide & EN19).

Contech Instruments Ltd.'s High Precision Laboratory Balance is used to weigh the workpiece before and after the wear test. Weight change (difference) is calculated by subtracting the final weight after wear test from the baseline weight of the workpiece before wear test. The following expression is used to calculate the mass wear rate:

$$\text{Mass wear rate} = \frac{\text{Change in weight}}{\text{Sliding distance}}$$

Table 1 Wear test parameters.

Material	EN19 (Substrate), Hardchrome, WC coating
Dimension of test specimen	Diameter = 40 mm, Thickness = 6 mm
Surface finish	0.5 to 0.7 μm for Hardchrome & EN19 and approx. 100 μm for WC
Sliding Velocity (V)	1 m/s
Normal Load (P)	30 N
Sliding distance	500 m
Track dia.	30 mm
RPM	600
Time (t)	8.33 min

Figure 6 shows X-ray diffraction on various objects using a Bruker D8 DISCOVER machine. XRD was performed on a copper tube with 1.5418 Å at a voltage of 40kV, a current of 40mA, and a power of 1600W. The XRD graph was created with Origin software utilizing data from the XRD machine for various coatings done on specimen.

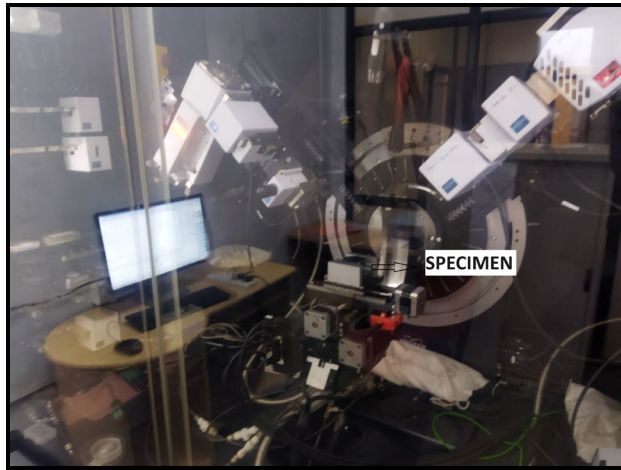


Figure 6 XRD performed for various materials.

FESEM and EDS for workpiece EN19 and Tungsten Carbide was done on Quanta 200F Netherlands make machine. Figure 7 shows FE SEM & EDS experimented for Hardchrome coating on ZEISS make Gemini SEM 300 machine. EDS i.e., Energy Dispersive Spectroscopy was performed on different samples on same machine.



Figure 7 FESEM & EDS for substrate EN19 and Tungsten Carbide done on Quanta 200F machine.

3.0 RESULTS AND DISCUSSION

In actual practice [21] the diameter of roll used is 60 mm to 400 mm and its length is 1400-1800 mm approximately. The roll

speed is 0.2 – 10 m/s. The thickness of the rolled workpiece which is usually low to medium carbon steel is 1.5 mm to 4 mm approximately and its width is 700-1500 mm approximately. The static and dynamic friction developed is 0.3 and 0.15 respectively approximately. Maximum Hertzian contact pressure developed between the rolled workpiece and EN19 roll is 500-2500 MPa.

In this experiment the same roll material EN19 is selected having Poisson's ratio 0.3, Elastic Modulus 200 GPa and hardness 23 HRC. It was rubbed against a Silicon Nitride ball having Poisson's ratio 0.23, Elastic Modulus 166 GPa. The experimental velocity was set at 1m/s. Maximum Hertzian contact pressure developed between the Silicon Nitride ball and EN19 through 30 N load was 1500 MPa. In actual practice rolled workpiece is low to medium carbon steel and in this experiment Silicon Nitride is selected as rubbing material which is much harder than low to medium carbon steel. Moreover, the actual conditions were tried to simulate in experimental set up.

The surface roughness of EN19, hardchrome and tungsten carbide was found to be 0.722, 0.576 and 100 Ra microns in order.

The hardness of substrates checked with Rockwell Hardness tester were found to be 65 HRC, 55 HRC & 23 HRC for Tungsten carbide, Hardchrome and EN19 respectively. Adhesion Strength Test result (as per ASTM C633-2017) were found to be 12580 psh for Hardchrome and 10850 psh for Tungsten carbide.

Five readings were taken for each material and averaged out. Wear test result from experiments is depicted in Table 2. The COF is obtained in the form of an excel sheet from the WINDUCOM 2010 machine software. The COF of WC, EN19, Hardchrome was investigated to be 0.66, 0.66 & 0.554 respectively as shown in Figure 8. The difference in COF value is due to different roughness levels of substrates.

Mass wear rate of Hardchrome, WC, substrate EN19 were revealed to be 1.66667×10^{-07} , 1.333×10^{-07} & 2.53333×10^{-06} (gm/Nm) respectively as shown in Figure 9. It means workpiece EN19 wore out at a higher rate followed by Hardchrome and WC. WC was investigated to be the best coating with the least wear among these.

Table 2 Experimental Result from Wear test machine.

Material	Load (N)	V (m/s)	Sliding distance (m)	Time (min)	RPM	Track diameter (mm)	Weight before (gm)	Weight After (gm)	Change in weight (gm)	Mass Wear rate (gm/Nm)	COF
Hard Chrome	30	1	500	8.333	600	30	64.349	64.3465	0.0025	1.66667×10^{-07}	0.554
WC	30	1	500	8.333	600	30	65.464	65.462	0.002	1.333×10^{-07}	0.66
EN19	30	1	500	8.333	600	30	65.502	65.464	0.038	2.53333×10^{-06}	0.66

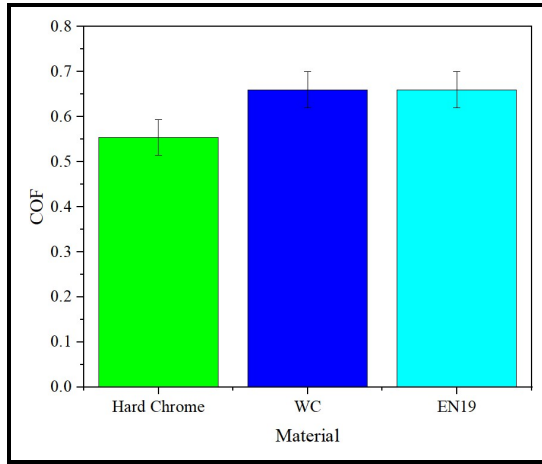


Figure 8 COF for various materials.

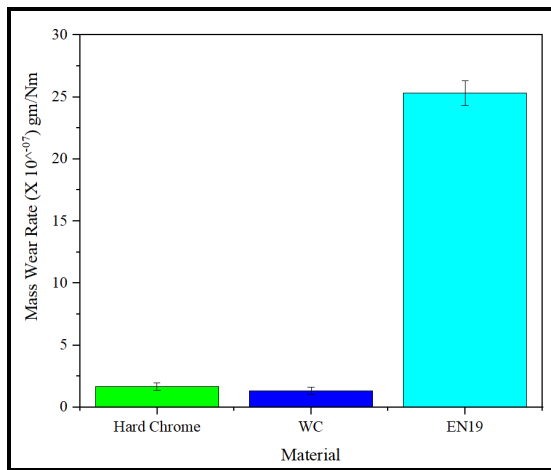


Figure 9 Mass Wear rate for various materials.

As demonstrated in Figure 10, the EDS Graph figure supports the existence of Hardchrome coating by 100% by weight.

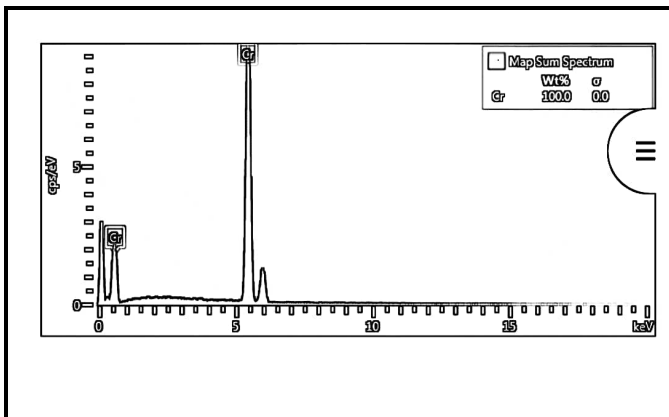


Figure 10 EDS for Hardchrome coating.

Figure 11 shows EDS Graph which was executed on Netherlands make machine Quanta 200F and it validates the presence of tungsten carbide coating. This coating has Tungsten 56.0%, Carbon 22.5%, Iron 15.6%, Cobalt 3.4% and Oxygen and Chromium in minor proportion by weight.

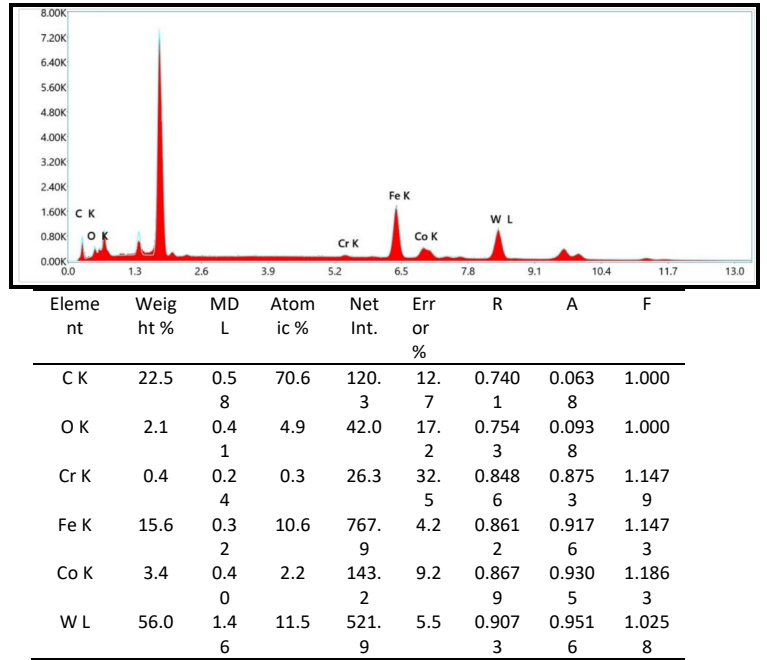


Figure 11 EDS result for Tungsten Carbide coating.

Figure 12 shows an EDS graph that indicates the presence of Iron (Fe) - 72.9%, Carbon (C) - 25.3%, and Silicon, Sulphur, Chromium, Manganese in minor amounts - 0.2 to 0.65 by weight for EN19. In the heavy steel industries roll is made of material EN19.

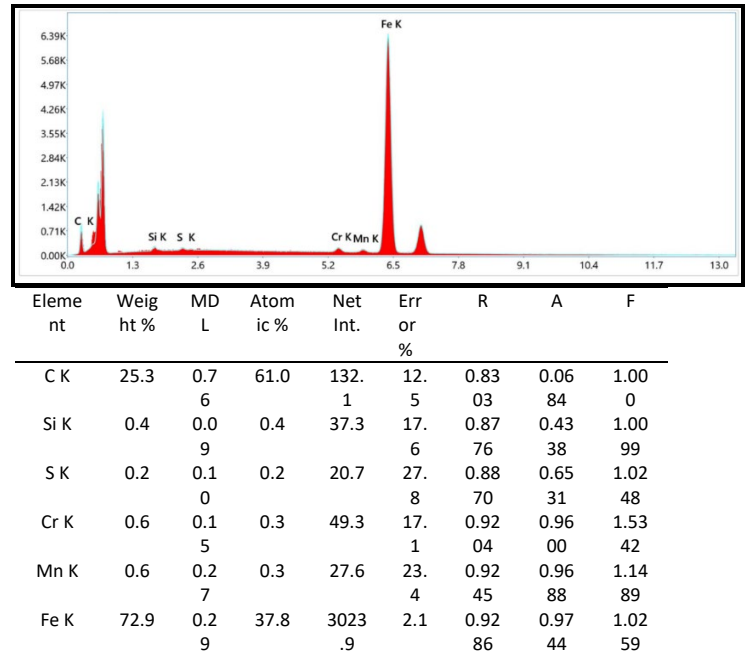


Figure 12 EDS result for substrate EN19

The FE SEM image in Figure 13 at 100X shows the wear track created in substrate EN19 following a wear test, which includes pits and craters. The photos reveal that abrasive and adhesive wear has occurred. SEM photos demonstrate that EN19 has the most wear (pits and craters form) as compared to Hardchrome and WC.

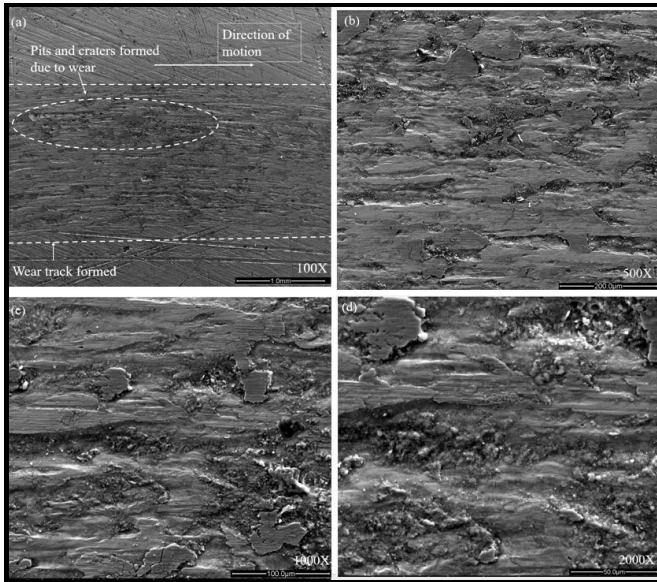


Figure 13. FESEM of EN19 within the wear track at various magnifications [a) 100X, b) 500X, c) 1000X, d) 2000X].

Figure 14 depicts a FESEM image of Hardchrome inside a wear track created during a wear test at a magnification of 200X. Craters & pits are produced as a result of wear, indicating that abrasive and adhesive wear has occurred. In addition, compared to EN19 pictures, this SEM image reveals reduced deterioration in Hardchrome.

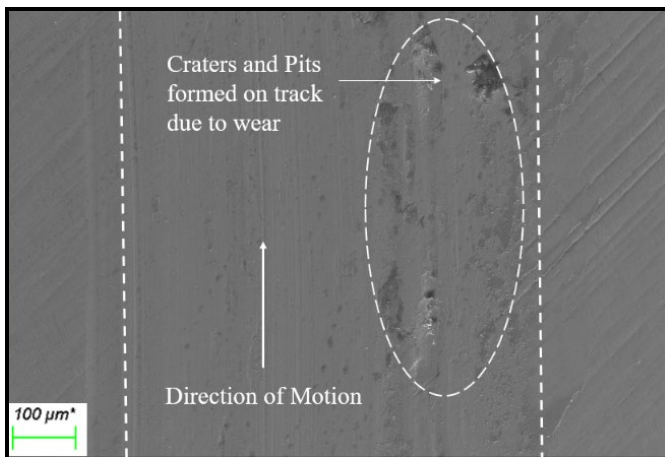


Figure 14 FESEM of Hardchrome wear track at 200X.

Figure 15 reveals the wear track obtained for tungsten carbide at magnification 200X, 1000X, 4000X after performing wear test. The images depict some wear has taken place and the reason for it could be due to adhesive and abrasive nature. But wear in WC is less than that of substrate EN19.

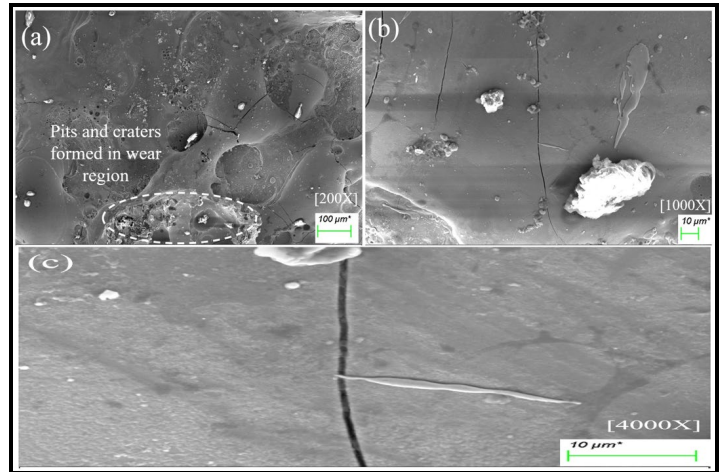


Figure 15 FESEM of tungsten carbide at the wear track originated after performing wear test at different magnification [a) 200X, b) 1000X, c) 4000X].

Figure 16 represents X-ray diffraction graph for EN19 was performed on the D8 DISCOVER Bruker make machine. Peaks for EN19 are seen at $2\theta=44.48$ (intensity 6582), which corresponds to the Fe (110) plane, $2\theta=64.73$ (2908.41), which corresponds to the Fe (200) plane, and $2\theta=82.14$ (3099.87), which corresponds to the Fe (211) plane, and it coincides with the standard files [19]. Because this graph features peaks which are sharp and narrow in nature and no broad peaks, EN19 is crystalline in nature.

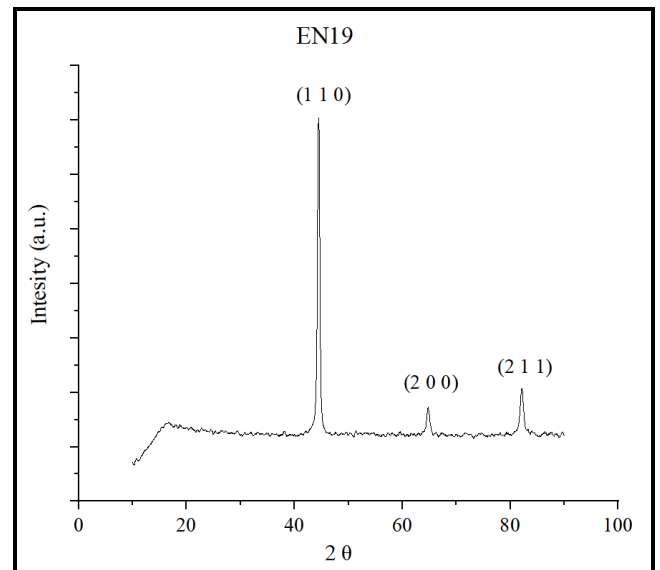


Figure 16 XRD graph for EN19.

Figure 17 represents XRD graph for Hardchrome. It has been found through literature survey and comparing to standard diffraction peak for hardchrome [17] the peak for Hardchrome coating performed on substrate as shown in fig.17 is found at $2\theta=43.956$ corresponds to HC (110) plane, $2\theta=81.07$ corresponds to HC (211) plane. The tight, sharp peaks in this graph indicate that the hardchrome coating is crystalline in nature.

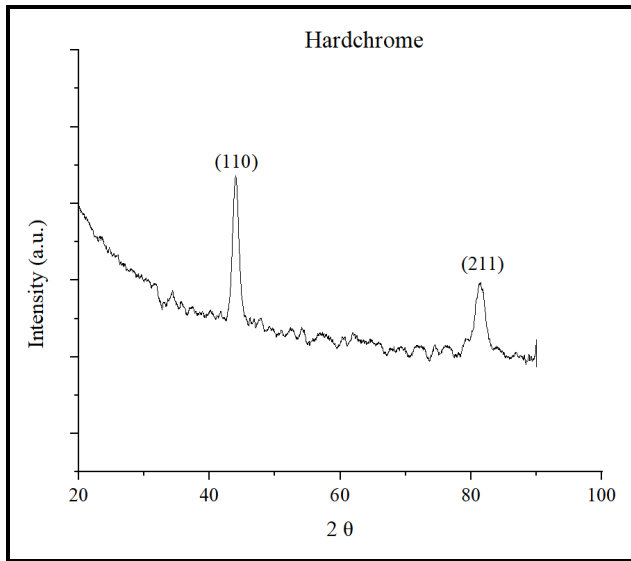


Figure 17 XRD graph observed or Hardchrome.

Figure 18 represents XRD graph for Tungsten Carbide. It has been found through literature survey and comparing to standard diffraction peak for WC [18], the peak for Tungsten Carbide (WC) coating applied on substrate as shown in fig.18 is found at $2\theta = 32.35$ (intensity 691) corresponds to WC (001) plane, $2\theta = 35.39$ (740) corresponds to WC (100) plane, $2\theta = 39.88$ (997) corresponds to Co (222) plane, $2\theta = 42.47$ (1171) corresponds to Fe (110) plane, $2\theta = 44.48$ (1025) corresponds to Co (400) plane, $2\theta = 46.64$ (939) corresponds to WC (101) plane, $2\theta = 50.05$ (1007) corresponds to Cr (024) plane, $2\theta = 64.77$ (1179) corresponds to WC (110) plane, $2\theta = 72.91$ (1375) corresponds to WC (111) plane, $2\theta = 82.28$ (1432) corresponds to WC (102) plane.

This graph displays narrow sharp peaks and no broad peaks, indicating that the WC coating is crystalline.

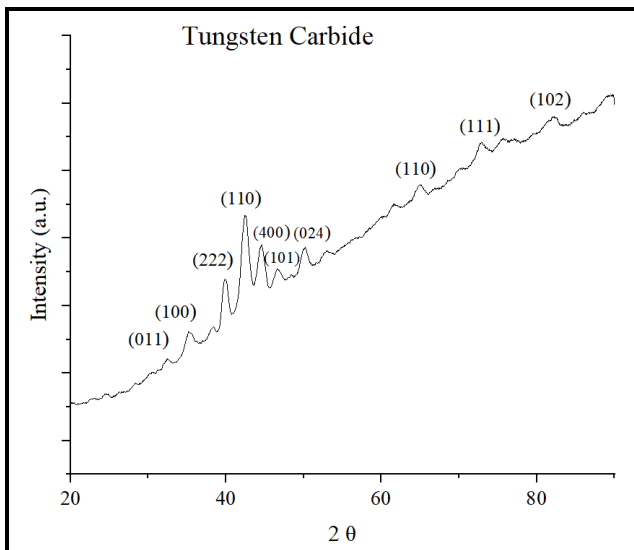


Figure 18 XRD graph for Tungsten Carbide.

From the XRD graph of all specimens EN19, Hardchrome, and Tungsten Carbide, it has been found that they have narrow sharp peak which represents that they have crystalline structure. The XRD graph results of the coated Tungsten Carbide, Hardchrome on the substrate EN19 agree with that of their EDS results. XRD also confirms the composition of coating on substrate. It is found $a=b \neq c$ and $\alpha = \beta = \gamma = 90^\circ$ ($a=4.221$, $b=4.09$, $c=3.791$, $\lambda= 1.5405$). It has been found that coatings are tetragonal, slightly monoclinic in symmetry.

3.1 Design of Experiment Analysis

A Design Experiment was carried out, focusing on different coating materials, in order to identify the key element contributing to roll wear and deterioration.

Table 3 Different levels and parameters selected for Taguchi DOE

Parameter	Level 1	Level 2	Level 3
Normal Load (N)	20	30	40
Speed (rpm)	600	700	800
TD (mm)	20	25	30
Material	Hardchrome	EN19	WC

The four factors that make up the wear test analysis are material, load, speed, and track diameter. Three different levels of analysis are conducted on each factor, as shown in Table 3. The experiment continued using the L9 orthogonal array. The results are shown in Table 4 for mass wear rate data for various parameter combinations at their respective levels. As shown in Table 5, the Signal to Noise Ratio was calculated using software Minitab 14. For the S/N ratio, the "Smaller is better" philosophy was used in order to achieve the least amount of coating wear. According to the Response Table for Signal to Noise Ratio, material has the greatest impact on roll wear life (Rank 1), followed by load (Rank 2), speed (Rank 3), and track (Rank 4).

Table 4 L9 Orthogonal Array

Sr. No.	Disc	Counter material (ball)	Load (N)	Speed (rpm)	Track diameter (mm)	sliding distance (m)	Weight before (gm)	Weight After (gm)	Change in weight (gm)	Mass Wear rate (gm/Nm) × 10 ⁰⁷
1	Hard chrome	Si ₃ N ₄	20	600	20	500	65.546	65.544	0.002	2.000
2	Hard chrome	Si ₃ N ₄	30	700	25	500	65.541	65.537	0.004	2.667
3	Hard chrome	Si ₃ N ₄	40	800	30	500	64.345	64.339	0.006	3.000
4	EN19	Si ₃ N ₄	20	700	30	500	65.500	65.475	0.025	25.000
5	EN19	Si ₃ N ₄	30	800	20	500	65.502	65.464	0.038	25.333
6	EN19	Si ₃ N ₄	40	600	25	500	65.521	65.469	0.052	26.000
7	WC	Si ₃ N ₄	20	800	25	500	65.457	65.456	0.001	1.000
8	WC	Si ₃ N ₄	30	600	30	500	65.446	65.444	0.002	1.333
9	WC	Si ₃ N ₄	40	700	20	500	65.464	65.461	0.003	1.500
10_CT	WC	Si ₃ N ₄	20	600	20	500	65.464	65.463	0.001	1.000

Table 5 Response Table for S/N Ratio

Level	Material	Load	Speed	Track dia.
1	-8.028	-11.326	-12.272	-12.538
2	-28.110	-13.030	-13.334	-12.273
3	-2.006	-13.788	-12.538	-13.333
Delta	26.104	2.461	1.061	1.059
Rank	1	2	3	4

The highest S/N ratio is shown by Material-3, notably tungsten carbide, in Figure 19, demonstrating its superiority as the best material option. Nickel and hard chrome are the next choices. According to the main effect plot's optimal values, a load of 20 N, a speed of 600 rpm, and a track diameter of 25 m are judged ideal. The experimental approach is then used to corroborate these results. The ideal conditions for the Tungsten Carbide material were load at 20N, speed at 600 rpm, and track diameter at 25m, according to a confirmation experiment. The experiment's conclusions were corroborated by the results, which are presented in Table 4 under experiment number 10_CT. They reveal a significant decrease in mass wear rate to 1.0×10^{-07} gm/Nm.

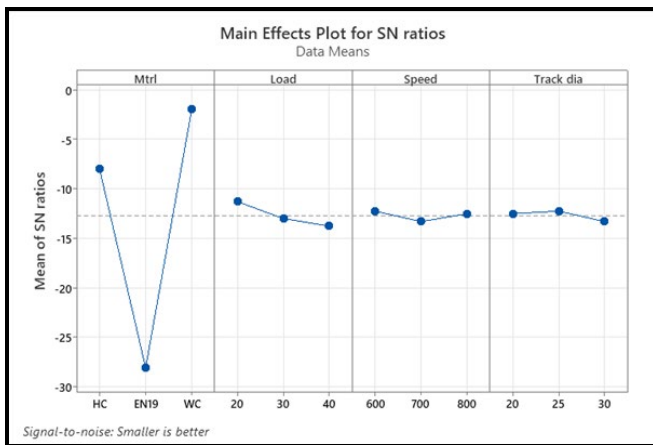


Figure 19 The Main Effect plot for Signal to Noise ratio.

4.0 CONCLUSIONS

To maximize the efficiency of the rolling process, the rolls, which are the basic and most significant instrument, should have a long life. However, because of the high load dynamic rolling application, roll wear and tear happens regularly, reducing productivity and efficiency of the process and resulting in a significant capital loss. In this paper, coatings of tungsten carbide, and hardchrome are done on the substrate EN19 which were verified by EDS results. The XRD graph revealed that coatings were crystalline in nature. The coatings were tetragonal slightly monoclinic in symmetry. XRD results are in line with the EDS materials composition results. From the experiments on pin on disk wear machine, mass wear rate of hardchrome, tungsten carbide, substrate EN19 was found to be 1.66667×10^{-07} , 1.333×10^{-07} & 2.53333×10^{-06} (gm/Nm) respectively. It can be concluded that substrate EN19 wore out at rapid rate followed by hardchrome and tungsten carbide was investigated to be the best coating with least wear amongst

these. Hardchrome gives 15.19 times higher life than substrate EN19. Tungsten carbide gives 19 times higher life than substrate EN19 and 1.25 times higher life than Hardchrome. SEM pictures demonstrated the occurrence of wear on the specimen due to adhesive and abrasive wear, which is the cause of roll wear out. According to Taguchi's Design of Experiments, material has the greatest impact on roll longevity, followed by load, speed, and track diameter. Tungsten Carbide was confirmed to be the best coating material by the main effect plot of S/N ratio. Taguchi's Design of Experiments results are aligned with wear test results. Experimental and analytical methods both showed that tungsten carbide outperformed other materials in terms of wear rate life.

Hence, it can be concluded that Tungsten carbide is one of the best materials which if coated to rolls can increase its life substantially and thus can minimize capital and time loss due to wear out of rolls. Also, Tungsten carbide has low friction, and good corrosion resistance.

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