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Wear Behavior of Boron Steel Coated Cerium Oxide Tungsten Carbide Cobalt Coating

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Abstract

The main aim of present work is to enhance effectively the working life of boron steel (medium carbon steel, 22BMn5) having application in agricultural implements. Process was carried out by using a protective layer i.e. providing a coating. Coating provided had the basic properties like high density and high wear resistance. Tungsten Carbide with 12% Cobalt (WC-Co) coating powder was selected, it shows improved wear resistance. Powder was mixed with cerium oxide, (5% and 10%) using ball milling with powder to ball ratio of 1:1 for time interval of 1 hour. Study is carried out in order to have knowledge about its phase and micro-structural characteristics and phase. Specimens were fabricated and wear test was carried out using pin-on-disc test rig. Wear test was carried out under three different load conditions with 7 cycles (to completely understand the wear behavior/pattern). SEM/EDAX and XRD analysis of worn out surface was performed, and results depicted that when lubrication is done with low value of applied load, powder with high percentage of cerium oxide shows the best result i.e. practically negligible wear loss.

Key Words: D-Gun, Pin-on-disc Test Rig, XRD, SEM/EDAX

1. Introduction

An engineering element or a manufactured part usually gets fractured and needs repair and maintenance periodically when its surface cannot adequately withstand the applied external force or the environmental conditions to which it is subjected. The choice of a surface material with the appropriate properties and resistance to wear, corrosion and degradation, is crucial for its proper functioning. Functional surface degrades, thus affecting the service life of the component, micro-cracks may occur, oxide layer may be formed on the worn out surface leading to corrosion. Therefore, engineering components must perform certain functions effectively, under numerous conditions in aggressive environments, that are normally complex, combining load with chemical and physical degradation to the surface of the component. The agricultural machinery uses the engineering component that operates under various conditions in resisting environments. Wear problem on medium carbon steel was selected as a case study in present work. Medium carbon steel has major application in agricultural implements like spades, shovels, earth movers, washers, etc. Coatings have been used for long period for protection of substrate in many ways usually providing shape, strength and stiffness.

- The various types of coatings are:

1. metallic: NiCrAlY, Cr_3C_2-NiCr, Triballoy etc.
2. carbide: TiC, SiC, WC, Cr_2C_3 etc.
3. oxide: Al_2O_3, Cr_2O_3, TiO_2, ZrO_2 etc.

Different types of thermal spray technique used are: plasma spray, detonation gun spray, wire arc spraying, flame spray, high velocity oxy-fuel coating (HVOF), warm spraying and cold spraying [1]. Among the commercially available thermal spray coating techniques, detonation spray and HVOF spray are the best known choices to get hard, dense and wear resistant coatings [2]. Detonation waves are also called as high pressure shock waves because they are produced as a result of combustion of the

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gas mixture which then propagates through the gas stream [3]. These waves are propagated through a water-cooled barrel as D-guns pray technique involving the entrainment of powdered materials with the high-velocity combustion products [4].

Satya Prakash, J. S. Grewal, Hazoor Singh Sidhu, Vikas Chawla and many others are the renowned researchers in this field. Numerous steel specimens and alloys have been tested for wear, erosion and corrosion in varied conditions and results have always been effective using thermal spray coating technique. Efficiency has increased and also the working life of component has increased manifold. Aim for selecting base material was initiated from the available literature only.

Grewal et al. studied detonation-gun spray technique which can be used for developing protective coatings of almost any material like oxides, carbides, metals, hard alloys and composite material powders onto mild steel and other EN series. It has optimized various process parameters like fuel ratio, carrier gas flow rate, frequency of detonations, and spray distance over the last few years [5]. Tungsten carbide with cobalt (WC-Co) is the best known powder available for wear resistant coating along with CrC-NiCr. Murthy et al. investigated the effect of grinding on the erosion behavior of a WC-Co-Cr coating deposited by both HVOF and D-gun spray process. He also compared the set techniques and found that the surface grinding improved the erosion resistance [6]. Singh et al. also made the efforts to reduce the wear rate of braking disc rotor with surface modifications techniques i.e. using D-Gun method and compared the wear properties grade of cast iron GI250 coated by WC-12CO and Stellite-6. Results have shown that WC-12CO on GI250 grey cast iron performs slightly better than the Stellite-6 [7]. Thakur and Arora studied the slurry and dry erosion behavior of HVOF sprayed WC-CoCr cermet coatings. For experimental work they used air-jet erosion test rigatan impact angle of 60 m/s [8]. Oxidation resistance of alloy coating failure was also observed and it was caused by the stresses generated in protective oxide scales. It was revealed from the study that scale cracking and spalling are the key factor to influence the lifetime of coatings. Grewal et al. [9] proposed that D-Gun spray can be used for developing protective coatings of almost any material like oxides, carbides, metals, hard alloys and composite material, onto mildsteel and other EN series. Rare earth metals have also proved their advantages in enhancing the service life of a mechanical component. These can be used in different percentages along with wear resistant powders for best effects. Kamal et al. [10] investigated the microstructure and mechanical properties of detonation gun sprayed NiCrAlY + CeO_2 alloy coatings deposited on superalloys. Dendritic structure formation is depicted by the coating and the microstructural refinement in the coating was due to process on T-22 boiler steel (ASTM-SA213-T-22) and studied the cyclic oxidation in air which was conducted at 900 °C temperature in the laboratory using silicon carbide furnace. Shakoor (2015) compared the properties of Ni-B and Ni-B-ZrO_2 coatings in their as deposited state to elucidate the effect of ZrO_2 addition on structural, surface, thermal and electrochemical properties of binary Ni-B coatings. It is noticed that Ni-B coatings are amorphous in their as deposited state while addition of ZrO_2 significantly improves the crystallinity [11]. Girisha (2016) reported that the conventional boron carbide (B_4C) powder particles of grain size 105 micrometer were blended with 1%, 2%, 3% of nanosized (44-55 × 10-9) boron carbide powder particles and were sprayed on commercially available, grit blasted 410 grade steel using HVOF. Results have shown that increasing the blending of % of nanopowders, enhanced hardness and wear resistance compared to micro B_4C coated and un-coated steel substrates [12]. Rao (2016) studied the conventional boron carbide powder(grainsize 105 μm) blended with 1%, 2% and 3% of nanosized boron carbide powder particles. The blended powder was sprayed on commercially available, grit blasted 410 grade steel using HVOF. Results indicate that blend increases hardness and wear resistance [13].

The present work is done on boron steel. Cerium oxide (rare earth metal) was added to coating powder so as to see its effect on efficiency and effectiveness of work piece material i.e. how it affect the service life of a component. Samples were coated with tungsten carbide cobalt coating (WC-12Co). The process was carried out by detonation gun technique which is a thermal spray process. Powders were mixed using ball milling machine and wear tests were carried out on pin on disc machine. Samples were then characterized to study and analyze their microstructure and worn out areas. Final consequence of the experiments reveals that the addition of cerium oxide to wear resistant powder (WC-12%Co), shows the least wear loss when applied load is less. Results also proves that the service life of base material increases effectively, thus reducing the repair cost and time.

1.1 Limitation of This Process

Thermal spraying is a line of sight process and the bond mechanism is primarily mechanical. Thermal spray application is not compatible with the substrate if the area to which it is applied is complex or blocked by other bodies.

2. Experimental Work

2.1 Material and Methodology

Boron steel (medium carbon steel) has been extensively used in agricultural areas from long time but only hardening processes have been done on it. Thermal spray coating process have not been yet tried and tested, but the present work includes the application of uncoated and coated specimens mixed with cerium oxide. Literature has been added on the work done on this base material but addition of rare earth metal has not been reported till now. In the present study tungsten carbide with addition of 12% cobalt (WC-12Co) was selected as the coating material on medium carbon steel i.e. boron steel 22BMn5. This powder is carefully chosen as it has high wear resistant characteristics. Coating powder was mixed with 5% and 10% Cerium (rare earth metal). Detonation gun (developed in U.S), is a high-velocity thermal spray coating technology that provides an excellent resistance against wear and corrosion. It is a variant of thermal spraying in which powder feedstock of the coating material is heated and accelerated by high-pressure, high-temperature gaseous products resulting from controlled explosive combustion of a carefully measured mixture of gases (usually oxygen and acetylene), to impact a substrate at very high velocities to yield a well-adherent layer. Steps carried are as follows:

1. Base sample was picked from the industrial scrap.
2. Sample were cleaned to remove any dust or dirt particle and finished to required specification dimensions.
3. Samples were then coated using D-Gun process. Powders were mixed with cerium oxide and two different compositions were prepared.
4. Fabricated samples were then tested on pin-on-disk machine and 3 different load conditions with and without lubrication.
5. Wear rate and volume loss was then calculated and graphs were plotted accordingly.
6. SEM/EDS and XRD analysis were carried out to check the microstructure of worn out surfaces and phase detection was done.
7. Results were compiled after that.

Table 1 lists the major components of boron steel. It is a medium carbon steel. Medium carbon steel is the most common form of steel, balances ductility with strength and has good wear resistance characteristics. From this chemical composition, samples of suitable size i.e. pins of length 30 mm having diameter 10 mm were fabricated. Samples were cleaned and finished to remove any dirt, dust and scale particle. Base material is used in many agricultural implement like washers (disc shaped thin plate with hole), shovel and spade. It has good wear resistance and literature has shown that modification has been done by using heat treatment processes like quenching. Agricultural implement shovel blades have been targeted for the present work. Coatings were then developed on the substrate material.

D-Gun thermal spray process was selected for the coating deposition. Deposition of coating powders involved the parameters as listed in Table 2.

Cost analysis for coating can be done as detonation gun: -300/-per sample (approximately). Sample used for the experimental purpose were taken from scrap of industry. (M/S Chari Wires, Faridabad) and coating pow-

Table 2. The process parameters employed during detonation gun spray process

Parameters	WC-12Co (5% and 10% Cerium)
Fuel gas	Oxygen and Acetylene
Carrier gas	Nitrogen
Pressure of fuel gas (Oxygen)	2–3 bar
Pressure of fuel gas (Acetylene)	1–1.5 bar
Pressure carrier gas (Nitrogen)	2–4 bar
Flow rate of fuel gas (Oxygen)	2800–5120 LPM
Flow rate of fuel gas (Acetylene)	2240–2420 LPM
Flow rate of carrier gas (Nitrogen)	720–960 LPM
No of shots per second	3
Stand-off distance	150–180 mm
Maximum substrate temperature	110 °C

Table 1. Chemical composition of boron steel (substrate material)

C	Si	Mn	S	P	B	Cr
0.19-0.26	0.40 max	1.10–1.40	0.025 max	0.025 max	0.0008–0.0050	0.15–0.35

ders were purchased from Ador Fontech Limited, Gurgaon at 3000/-per kg.

2.2 Characterization

Below Table 3, details the chemical composition of EN-31 disc used in the test rig. It is made of stainless steel. It is an excellent high carbon alloy offering high hardness with compressive strength and abrasion resistance. It has its main application in wear resisting machine constituents. Pin on disc test rig was used to carry out the wear tests for both coated and uncoated samples in wet and dry condition (with and without lubrication) under different load conditions of 30N, 60N and 90N. Top surface was then characterized and examined using XRD & SEM/EDAX techniques. Pin on disk test rig used for the experimental purpose has compact design, friction and wear sensors with large number of testing capabilities. Parameters like load can be varied, track diameter as per the rpm can be varied. Tests may be done at different environmental conditions and with or without any gas chamber creating vacuum for sample. Also different shape of specimen can be tested like ball depending upon the die available to hold it. This simply states that depending upon the requirement adjustments in the test rig can be done easily as per the requirement. The present research was done at room temperature with and without lubrication. SEM and XRD techniques were used for analysis of microstructure and phase detection of worn out surface. JSM 6610LV, JEOL fitted with EDAX Genesis software attachment available at SEM lab, Amity University, Noida (India). SEM was done to identify inclusions, un-melted, partially melted particles and pores in the as sprayed coatings. EDAX genesis software indicates the elemental compositions (weight %) present at point/ area of interest. SEM provides details surface information by tracing a sample in a raster pattern with an electron beam. It has powerful magnification, high resolution and three-dimensional images produced by SEM provide topographical, morphological and compositional information [14]. XRD is a widely used method producing qualitative results. Measurements are made to understand the composition of the material on an atomic level, since all diffract the beam differently. Room temperature condition was selected for conducting wear tests.

Table 3. Chemical composition of EN-31 carbon steel disc (by wt%)

C	Si	Mn	S	P
0.42 max.	0.05–0.35	0.40–0.70	0.05 max.	0.05 max.

Figure 1 shows the top view of pin on disk test rig holding the specimen for wear test. Figure completely displays the parts of tribo-meter i.e. the base plate with load cell and friction cell. L-keys were used to tighten the pin shaped specimens. This tribo-meter can also be used for testing ball shaped specimens. Wear test were carried out for both coated and uncoated samples in two different conditions of dry and wet state. Load of 30N, 60N and 90N were selected at fixed velocity of 1 m/s i.e. 160 rpm with a slight variation of ±2 r.p.m. was observed. Track radii for pin was fixed to 80 mmand wear tests were carried out for a total sliding distance of 5400 m (7 cycles of 5 min, 5 min, 10 min, 10 min, 15 min, 15 min, 30 min duration), so that only top coated surface was exposed for wear test. 7 cycles were taken so as to clearly observe the wear pattern through graphs. Pins were cooled at room temperature and cleaned with brush to remove any dirt or debris particles. Samples were weighed and also height was measured before and after using weighing balance and vernier caliper. Significant changes were noticed and recorded after the wear tests. Using the same apparatus i.e. pin on disk machine many other tests can be carried out like load can be varied, environmental conditions can be changed (present tests were carried out at room temperature), rpm and track diameter can be varied as per the load conditions with or without gas chamber i.e. creating vacuum.

3. Result and Discussion

3.1 XRD Analysis

Figure 2 and 3 both show the XRD plots. Peaks in these plots depict the phases present on the top surface of susbtrate material. In Figure 2 when uncoated bare sam-

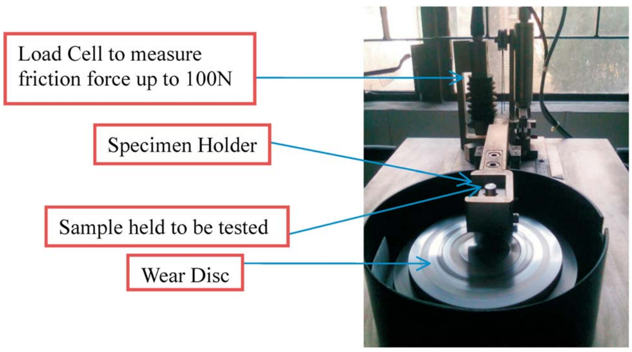

Figure 1. Pin on disc machine (major parts).

ple is tested at higher load without lubrication, it results in highest wear rate loss. Figure 2 depicts the XRD Plot of bare substrate material that shows the peaks of its main constituents only. It includes mainly Manganese (Mn), Carbon (C), and Chromium (Cr) in highest percentage. Also the utmosttop peak shows the composition of Mn and boron. These peaks are results after testing the samples at the maximum applied load condition i.e. total worn out top layer. This implies that in field work bare boron steel if used without coating has very less efficiency and can't work for longer duration when applied load is high hence, needs immediate repair, maintenance which highly affects cost and is time consuming. In Figure 3, when the sample is coated and used in wet state, then the results show about negligible wear loss. Figure 3 reveals the top layer of worn out specimen. The top layer of specimen is coated and when it gets worn out, chemicals like Cobalt (Co), Manganese (Mn) and Carbon (C) were observed as major constituents. Also small percentage of cerium oxide was visible as the cerium oxide was thoroughly mixed with the coating powder using ball milling. Higher peaks are being seen in the XRD plot. This results that when wear is negligible, coating layer hasn't been distorted and different phases can be detected through it. It also proves that the boron steel if used in coated state, it results in higher efficiency and reduced cost.

3.2 SEM

Figure 4 shows the SEM images of different samples including coated specimen as well as uncoated bare sample at two variant loads and at different condition. It can be revealed from the Figure 4(a) image that when 30N load is applied on uncoated sample in dry state, wear rate is high and the top layer is divided into layers creating an uneven surface. Similarly, in Figure 4(b) when higher load is applied top layer of sample completely changes, uneven peaks appear on the surface making it rough. In Figure 4(c) samples have been coated with addition of rare earth metal, but as condition is dry therefore, wear takes place even when a load of 30N is applied. Abrasive wear takes place when surface is in contact with the disk, thus top layer gets distorted. This is adhesive wear. Figure 4(d) shows the best result when minimal load is applied and lubrication is their to prevent any wear loss. Cerium oxide at this instance shows its effectiveness and enhances the life of substrate material. Very minute or almost invisible peaks can be observed in this case. In Figure 4(e) i.e. dry state when higher load is applied, coating shows its effect but up to a certain limit, after that cracks appear on the surface thus microstructure of surface gets affected. This is micro-ploughing (the material is shifted to the sides of the wear groove. The material is not removed from the surface). Peaks and valleys were visible when magnification was about 500 times. In Figure 4(f) when lubrication is applied abrasive wear takes place as applied load is high thus peaks can also be seen, lumps show the worn out area. From all the above images it can be concluded that coatings are appropriately applied and they show their effect i.e. enhancing the life of bare material and increasing its efficiency.

Figure 5(a) and (b) above shows the presence of different elements on the top surface of specimens after coating. In Figure 5 'C' denotes the presence of $CaCO_3$ and 'O' for SiO_2. Maximum amount of ferrous (in both weight percentage and atomic percentage) is present on the top layer after specimen has been tested on tribo-meter. Simi-

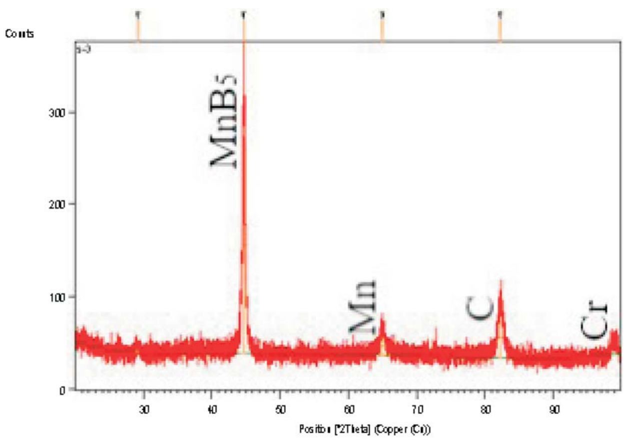

Figure 2. XRD Plot (DRY, Un-90N).

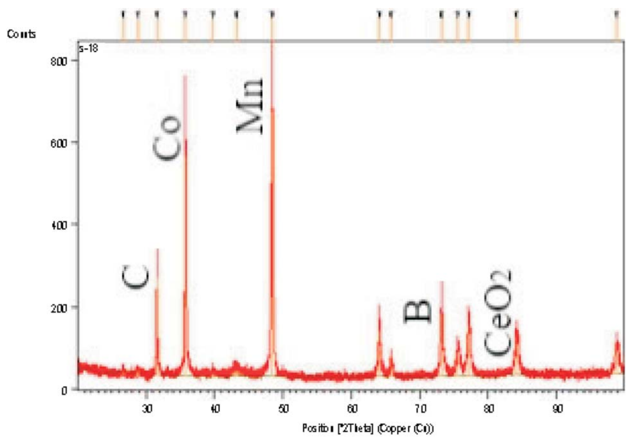

Figure 3. XRD Plot (WET, WC-Co + 10% Ce-30N).

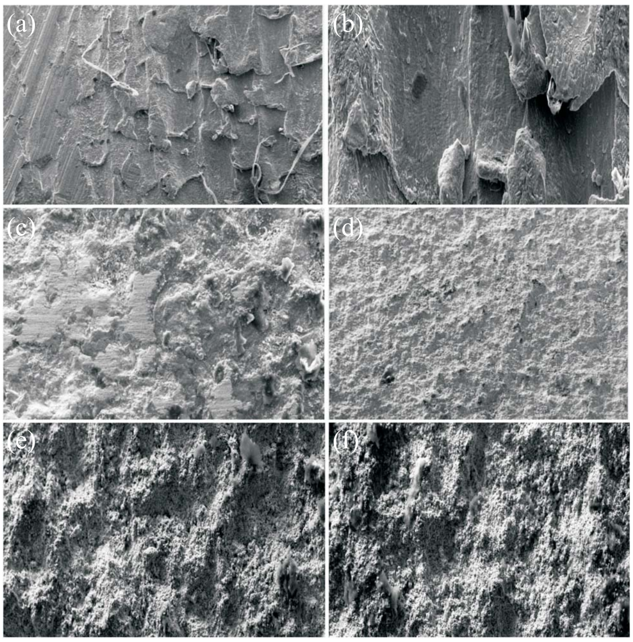

Figure 4. SEM images of different samples. (a) DRY, Uncoated 30N. (b) DRY, Uncoated, 90N. (c) DRY, WC-Co + 10% Ce, 30N. (d) WET, WC-Co + 10% Ce, 30N. (e) DRY, WC-Co + 10% Ce, 90N. (f) WET, WC-Co + 10% Ce, 90N.

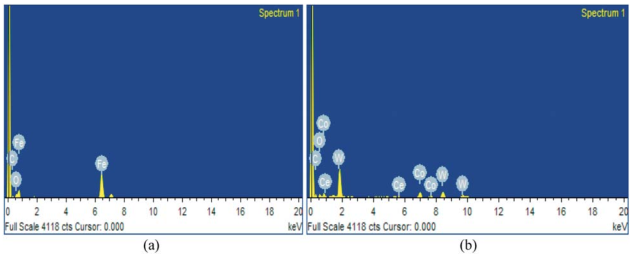

Figure 5. EDAX images (combined with SEM results). (a) Uncoated sample at 90N. (b) WC-Co + 10% Ce sample at 30N.

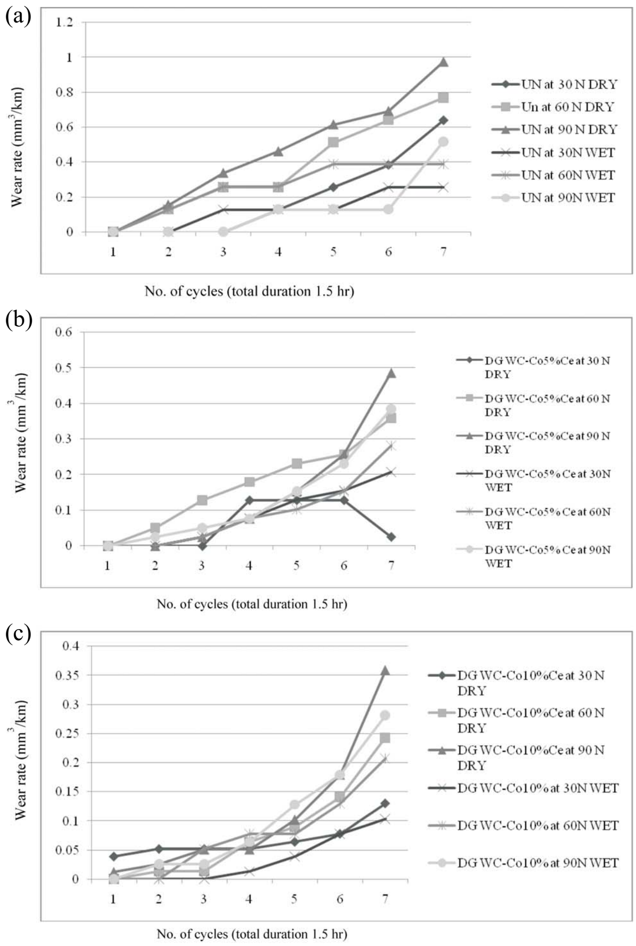

Figure 6. (a, b, c) Line graphs of wear rate vs. no. of cycles.

larly in Figure 5(b) i.e. in coated sample when it has been tested and wear has taken place, pieces of tungsten carbide and cerium can be clearly observed. On the top worn out layer tungsten is present in highest percentage (almost 50%). This is because the specimen is coated with it, hence this proves that coating has been applied properly without any failure and applied load is less therefore, less wear has taken place. As the applied load increases wear rate increases and top layer gets off completely.

3.3 Wear Rate

Figure 6(a) shows the difference between dry and wet state when bare material is being tested at different loads for 7 cycles. These 7 cycles have been selected so as to completely observe the wear pattern when time increases. It can be seen that uncoated sample in dry condition shows the maximum loss in the wear rate whereas uncoated sample in lubrication state shows the best result when minimum load of 30N is applied amongst them. Figure 6(b) shows the difference between dry and wet samples when coated with WC-Co+5%Ce. Addition of cerium oxide increases the wear resistance of coating powder. It can be observed that when maximum load is applied wear loss is highest in dry conditions. Similarly when applied weight is less in lubricated state wear rate loss increases linearly as time increases. Figure 6(c) shows the difference between dry and wet samples when coated with WC-Co+10%Ce. Higher amount of addition of cerium affects the working life of sample. Thus, there is much saving in cost and time. In this case also when applied weight is less wear loss rate is also low. These graphs show the different points forming a wear pattern. Form

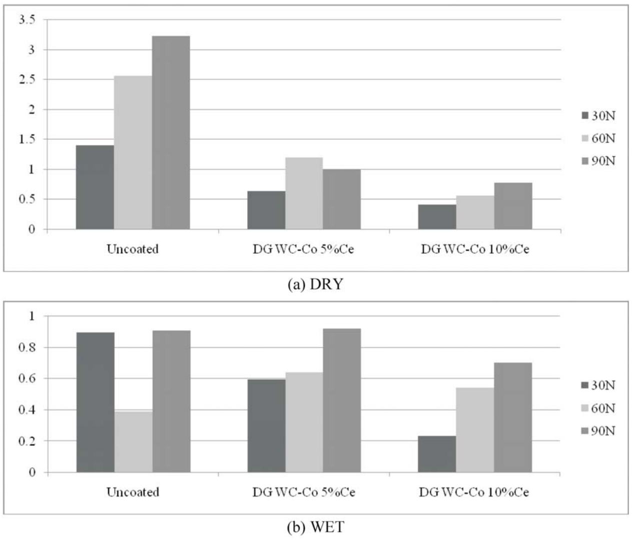

Figure 7. Comparison of uncoated and coated sample in dry and wet state.

the above graphs it can also be concluded that wear rate is affected by the addition of Ce in wear resistant coating powder. Greater amount of addition of cerium results in more effective results, thus enhancing the efficiency of specimens. Also in wet state wear rate has dropped down gradually in all the cases.

Figure 7 shows the comparison between different coatings and the uncoated bare material at different loads. From this comparison it can be clearly said that at least applied load (30N), D-Gun sprayed WC-Co with 10%Ce shows the best result i.e. almost negligible wear loss as compared to other conditions. It can also be concluded that rate of wear loss is highest in case of uncoated sample when 90N load is applied in dry state. This proves that under lubricating condition substrate material performs well with and without coating at any load condition. Table 4 depicts the wear rate for different specimens.

The wear resistance for coating-substrate-condition combinations in their increasing order is given below:

WC-Co + 10% Ce (wet) > WC-Co + 10% Ce (dry) >
WC-Co + 5% Ce (wet) > WC-Co + 5% Ce (dry) >
Uncoated (wet) > Uncoated (dry)

From the above stated end result it is clear that as the applied load increases wear rate increases. This is because the specimen and the disc are in contact with each other with higher penetration rate and even the lubrication oil shows less effect in it. Whereas in similar case when the applied load is less there is a lesser amount of penetration and oil film easily creates the gap between the

Table 4. Results for various specimens with their conditions

Coating	Condition	Load (N)	Wear rate (mm^3/km)
Uncoated	DRY	30	0.137
		60	0.139
		90	0.097
	WET	30	0.040
		60	0.060
		90	0.090
WC-Co-5% Ce	DRY	30	0.111
		60	0.223
		90	0.202
	WET	30	0.067
		60	0.068
		90	0.007
WC-Co-10% Ce	DRY	30	0.016
		60	0.052
		90	0.333
	WET	30	0.068
		60	0.045
		90	0.135

mating surfaces thus reducing abrasive wear rate. When lubrication is not applied i.e. in dry case wear rate is high even when small load of 30N is applied. This is because the surfaces are in true contact with each other and gets worn out. Top layer of the microstructures gets completely damaged as the load increases. Coating layer having thickness of 200 microns gets worn out when the load increases. Wear rate for bare metal at 30N load in dry conditions was 0.1185 mm^3/km, which increased to a value of 0.1810 mm^3/km when load of 90N was applied under similar dry conditions. These values drop down to 0.0474 mm^3/km (30N) and 0.0949 mm^3/km (90N) when lubrication oil is applied. This drop in wear rate shows the effect of addition of lubrication oil. Coating is the only way to protect the surface from getting damaged as this medium carbon steel has applications in agricultural implements. Also addition of rare earth metal shows its effect as higher percentage of addition results in lesser wear rate loss. This can also be understood from the bar graphs (Figure 7). It shows the comparison between the different coatings under different load conditions in both wet and dry state.

4. Conclusion

- Coatings have been successfully deposited on the substrate material.
- Cumulative weight loss for coated and uncoated samples increases with increase in load.

- Wear rate loss for same conditions decreases in case of wet system whereas in case of dry system it increases.
- D-Gun is characterized by extremely high density, high micro hardness with low porosity and can be used for a variety of materials like metallic, carbides and oxides.
- Tungsten carbide with addition of cobalt (chosen as coating powder) is the best known wear resistant coating in literature. Wear rate for coated specimen is much less than that of bare specimen. Addition of cerium oxide enhances the service life of component.
- SEM analysis as depicted in Figure 6 above proves that the micro structure of the top layer of coated specimen gets less distorted and almost negligible wear can be observed.
- Cerium oxide shows it effectiveness when applied load is less and lubrication is done. Cracks and peaks valleys are not visible, surface is smooth enough i.e. working life of the component has been increased manifold.

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