**Journal of Manufacturing Engineering, December 2014, Vol. 9, Issue. 4, pp 202-211**

# EFFECT ON THE PROPERTIES OF CERAMIC COATINGS WHEN BLENDED AND SPRAYED USING HIGH VELOCITY OXYGEN FUEL PROCESS

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## ABSTRACT

The aim of the present work is the investigation of sliding wear behaviour of two different coatings and study of the effect on the properties when they are blended. The mild steel samples were coated with the tungsten carbide/cobalt (WC-Co), nickel chromium boron silicon (NiCrBSi) and a blend of 30 % WC-Co and 70% NiCrBSi. The powders were sprayed using HVOF thermal spray process. The coatings were characterised with regard to porosity, adhesion tensile strength, micro hardness and microstructure. The sliding wear test of the coatings was performed by pin-on-disc apparatus and was analysed by Response Surface Methodology (RSM). To develop a wear model of coatings three factors temperature, load, sliding distance were used**.** ANOVA was carried out to determine the significant factors and interactions. The porosity of the specimens was determined by using the optical microscope. The coating morphology was studied by Scanning Electron Microscopy (SEM) and EDAX. The experiments have been subsequently analysed and assessed that the properties of WC-Co + NiCrBSi (blend) obtained are superior in all aspects to the other two basic coatings.

***Keywords****: RSM, protective coatings, thermal spray, HVOF, WC-Co/NiCrBSi.*

## 1. Introduction

One of the most important issues in the recent automotive industry is replacement for hard chrome plating as it is health hazardous [1], In addition to this the wear and frictional force at room and elevated temperatures at different loads is a serious problem for many machine components used in aircraft, marine, automobile and metallurgical industries. Though much research and experimentation is going on and there is a lot to be known. It is recognized that mechanical and chemical properties of HVOF sprayed coatings such as wear resistance; corrosion resistance; micro hardness and bond strength are controllable by selecting proper materials [3].

This problem can be overcome with the use of thermally sprayed coatings as it can be considered as an alternative for the replacement of hard chrome plating [2, 3]. Thermal spray coatings are increasingly employed in many applications to reduce wear and friction thereby increasing the efficiency, decreasing the downtime, component repair and replacement [4]. There are many types of thermal spraying techniques like plasma spraying, Flame spraying and HVOF spraying, among these coatings HVOF has often been used to form dense, and homogeneous coatings to protect essential components from wear, corrosion and oxidation in many industrial applications. [5,6, 7].

Tungsten carbide (WC) and chromium carbide ($Cr_3C_2$) and between are the materials which are often used for such a purpose [8-13]. Recently nickel alloy NiCrBSi also is found to be of considerable interest to the researchers. Chromium in it increases resistance to corrosion and oxidation at high temperature. Boron helps in decreasing the melting temperature and is one of the components of the hard compounds formed during the protective coating synthesis process. Silicon is added to maximize quality of melting process (14). Much research has been carried out by many researchers on different types of wears like abrasive wear (18), erosive wear (7), computational mechanics-based approach was discussed in ref (19). But a very few works are done on the blends and on sliding wear properties. In the present work the analysis of sliding wear behaviour for WC-Co, NiCrBSi and blend of WC-

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Co and NiCrBSi (30% WC-Co+ 70% NiCrBSi) was carried out using design expert software.

## 2. Experimentation

### 2.1 Materials

As substrate has very little effect on the evaluation of the coatings, mild steel is used for various coating depositions. The Commercially available powders from two different companies were used in this study. The composition of the powders WC-Co (88 % WC, 12 % Co) and NiCrBSi (Ni–17Cr–2.5Fe–2.5Si–2.5B–0.15C). The details of these powders are as given in table 1 and for the third coating the powder is prepared by mixing WC-Co and NiCrBSi powders mechanically blended in the ratio of 3:7.

**Table 1: Details of thermal spray powders**

| Coating | Powder type | Powder size (µm) | Manufacturer |
|---|---|---|---|
| WC-Co | Agglomerated and Sintered | -38+10 | H.C.Starck |
| NiCrBSi | Gas atomized | -53+15 | Wallcolomy |
| 30%WC-Co+70%NiCrBSi | Combination of above two | | |

### 2.2 Methods

Powders were sprayed onto the substrate specimens using the HVOF process. Prior to the coating deposition the substrate preparation was done by pre-cleaning the specimens in isopropyl alcohol for 5min. All the coatings were deposited on grit blasted (coarse Al2o3-24mesh) before spraying to provide a high degree of roughness that promotes mechanical interlocking for enhanced adhesion. The parameters employed during spraying are as given in table 2.

**Table 2: Spray parameters**

| Gas | Operating Pressure | Flow (SLPM) |
|---|---|---|
| Air | 07.0 Kg/Cm2 | 450 -600 |
| Oxygen | 10.0 Kg/Cm2 | 200-300 |
| LPG | 07.0 Kg/Cm2 | 60 - 80 |

The powders were sprayed by using a DJ 2700 HVOF gun (MEC), Jodhpur, located at VTC, Visakhapatnam. The operating conditions selected are: Flame temperature: min. 2500°C -3160°C. Flame velocity 2200 m/s, Particle velocity 650 m/s, Spray rate/ powder feed rate 25-38 gm/min, Spray distance 6 to 8inches, Deposition Efficiency 60-70% , Critical orifice No. Flow meter 01, Traverse rate (m/sec): 0.0028-0.0030m/sec.

## 3. Characterization of Coatings

### 3.1 Microstructural evaluation

The most common way of analyzing the porosity of the coatings is to prepare a metallography cross-section sample of each coating and the method employed to determine the porosity from the micrographs similar to the one as discussed in ref (15). In this study the coating porosity was measured by the micrographs taken by optical microscope (Olympus, Japan, located at GITAM University). Some reports claim that image analysis can reproducibly detect and measure microstructural features (pores, cracks, etc.) to a 95% confidence level within thermal spray coatings (16). The samples were polished using standard metallographic procedure and etched with Nital Solution (a chemical mixture of Nitric acid and ethyl alcohol). The average of 6 areas of each coating has been used for porosity measurement. The structures of the coated surfaces were evaluated by microstructural characterization using SEM and the microhardness of the coated samples at their cross sections was measured using a calibrated Vickers micro indentation hardness indenter. The parameters which are used during micro hardness testing are dwell time 15s, indenter speed of 60µm/sec, and the angle between two faces was maintained at $136^0$ under a test load of 50 grams. The reported values were averages of 5 measurements. These tests were conducted in Defense Metallurgical Research laboratories, Hyderabad, India.

### 3.2 Experimental Set Up for Wear Test:

The specimens taken for wear test were the coated pins having cylindrical shape of diameter 10mm. An EN31 disc of diameter 120mm and thickness of 9mm is taken as a standard counterpart for conducting the wear test. A sliding wear test was conducted on a pin-on-disk tribometer (Dotcom Tribometer) as per the ASTM G99 standard. The sliding wear caused by the coated pins which are held stationary under certain load in contact with the rotating disc, while the disc rotates beneath it at a constant velocity similar to as discussed in ref (19). The wear and friction experiments were conducted at loads of 20N and 30N. This machine is capable of providing wear and friction force data at different sliding speeds and loads with respect to time. The wear and friction data were collected at regular intervals of time and data reported in this study are the average of at least three runs. The results were model analysed using design expert software.

## 4. Design of Experiment (DOE)

In literature the wear has been studied by varying a single factor. In the present study an approach that takes the interaction effects of the all factors that influence the wear into consideration are evaluated. For this purpose Design of Experiments and Response surface Methodology has been used to find the interaction effects of factors on sliding wear of various coatings.

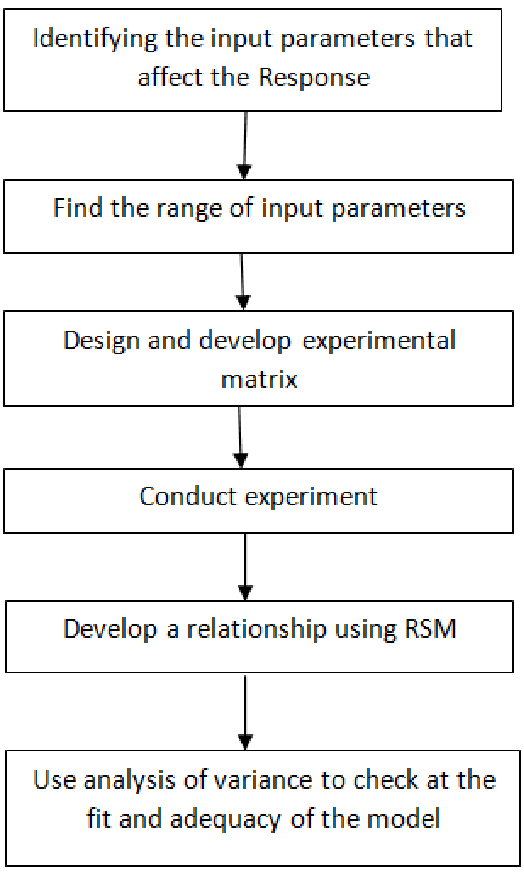



**Fig. 1 Sequence of investigation**

As claimed by the author J. Trpcevska e.tal in reference (20) the most significant factors that affect wear are grain size, hardness, sliding distance, temperature (21) spray parameters and spray systems (22) etc. The factors considered in the present work are Temperature, Sliding Distance, and Load. The parameter distance is taken as continuous variable, temperature and load as discrete variables. The experimental design matrix for different runs is shown in table 3. A user defined quadratic model is designed using DOE and RSM is chosen to find the relationship between the response function and variables using the statistical software package Design Expert Software 9 (Stat Ease, Inc., and Minneapolis, USA). The interaction between the variables and the ANNOVA has been studied using RSM. The quality of the fit of this model is expressed with the coefficient of determination R2. The scheme of investigation was conducted in the following sequence to develop empirical relationships and to predict specific wear rate of the coatings as shown in figure1.

**Table 3: The design table along with response for coatings WC-Co, NiCrBSi, WC-Co+ NiCrBSi coatings**

| Std | Run | Distance(A) m | Load(B) N | Temperature(C) $^0$C | Response( Wear ) µm WC-Co | NiCrBSi | WC-Co+NiC |
|---|---|---|---|---|---|---|---|
| 12 | 1 | 5[-1] | 20[-1] | 350[1] | 2.28 | 205.83 | 7.98 |
| 3 | 2 | 5[-1] | 20[-1] | 40[-1] | 28.74 | 9.63 | 2.84 |
| 4 | 3 | 5[-1] | 30[1] | 350[1] | 68.49 | 58.72 | 14.89 |
| 7 | 4 | 5[-1] | 30[1] | 40[-1] | 26.19 | 5.98 | 28.65 |
| 15 | 5 | 3753.75 [-0.33] | 20[-1] | 350[1] | 60 | 1051.98 | 201.34 |
| 19 | 6 | 3753.75 [-0.33] | 20[-1] | 40[-1] | 51.23 | 19.25 | 32.95 |
| 8 | 7 | 3753.75 [-0.33] | 30[1] | 350[1] | 220.8 | 295.62 | 47.77 |
| 1 | 8 | 3753.75 [-0.33] | 30[1] | 40[-1] | 60.3 | 16.45 | 51.33 |
| 9 | 9 | 7502.5[0] | 20[-1] | 350[1] | 340 | 1057.29 | 190.13 |
| 13 | 10 | 7502.5[0] | 20[-1] | 40[-1] | 56.59 | 19.61 | 52.19 |
| 16 | 11 | 7502.5[0] | 30[1] | 40[-1] | 61.32 | 18.57 | 54.9 |
| 10 | 12 | 7502.5[0] | 30[1] | 350[1] | 358.94 | 930.6 | 186.54 |
| 14 | 13 | 11251.25 [0.33] | 20[-1] | 350[1] | 340 | 1113.57 | 221.74 |
| 17 | 14 | 11251.25 [0.33] | 20[-1] | 40[-1] | 57.33 | 20.15 | 57.5 |
| 5 | 15 | 11251.25 [0.33] | 30[1] | 350[1] | 358.94 | 1193.99 | 405.36 |
| 11 | 16 | 11251.25 [0.33] | 30[1] | 40[-1] | 61.5 | 19.78 | 56.11 |
| 2 | 17 | 15000[1] | 20[-1] | 40[-1] | 57.77 | 21.46 | 59.62 |
| 20 | 18 | 15000[1] | 20[-1] | 350[1] | 500.68 | 1152.9 | 232.33 |
| 18 | 19 | 15000[1] | 30[1] | 350[1] | 549.68 | 1192.22 | 406.12 |
| 6 | 20 | 15000[1] | 30[1] | 40[-1] | 61.38 | 20.54 | 56.54 |

### 4.1 Response surface methodology

Response Surface Methodology (RSM) with three levels of each factor were used in the present study. The factors were designated as A (distance traversed, m), B (load, N) and C (temperature, $^0$C) respectively. The coded values of upper, middle, and lower levels of each factor are designated by +1, 0, and -1 respectively. The coded values of various factors and response wear for 3 coatings under consideration used in the present study are shown in Table 3 and the factors and their levels considered under this study are tabulated in table 4

**Table 4: Various factors and their levels used in Sliding wear behaviour study.**

| Factor | Name | Units | Type | Subtype | Lower Level | Upper Level | Std. Dev. |
|---|---|---|---|---|---|---|---|
| A | Distance Traversed | m | | Continuous | 5 (-1) | 15000 (1) | 5346.273 |
| B | Load | N | Numeric | Discrete | 2 (-1) | 3 (1) | 0.504219 |
| C | Temp | $^0$C | | Discrete | 40 (-1) | 350 (1) | 156.308 |

**Table 5: Mechanical properties**

| Coating | Coating thickness (μm) | Bondstrength (N/mm$^2$) | Porosity (%) | Microhardness ($HV_{50}$) |
|---|---|---|---|---|
| WC-Co | 140-160 | 79.22 | 0.855 | 1293 |
| NiCrBSi | 200-230 | 58.15 | 0.922 | 997 |
| WC-Co+NiCrBSi | 140-170 | 84.25 | 1.788 | 1388 |

## 5. Experimental Results and Discussions

The results obtained from the above experimentation are as tabulated in table 5. It is observed that the bond strength of the blended coating is superior to the other two coatings and the insight of the coating into the substrate was observed to be very secure from the figures 2 (c). It can be noticed that the blended coating showed higher micro hardness than the other two. The increase in the micro harness is due to the presence to hard phases in the coating.

The adherence between the coating and substrate is high as no cracks or voids were observed at interface which is evident from figures 2 (a) and 2 (c). But in case of figure 2 (b) the bonding is not so good. Lower hardness in NiCrBSi might be due to the absence of the hard phases in the materials. The three coatings have uniform structure.

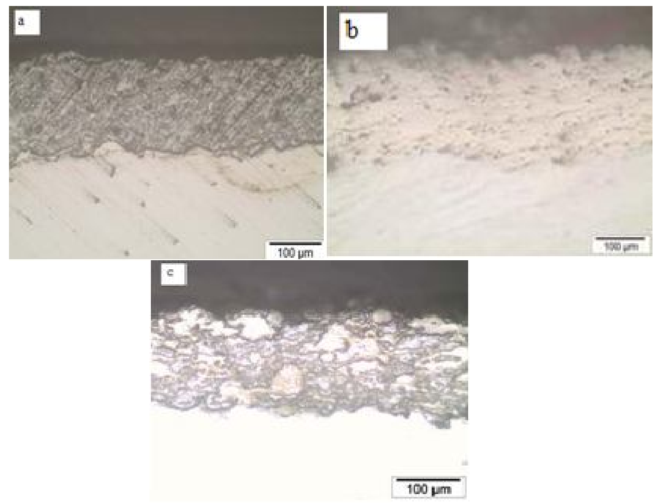



**Fig. 2 Microstructure of Coatings (a) WC-Co (b) NiCrBSi (c) WC-Co+NiCrBSi**

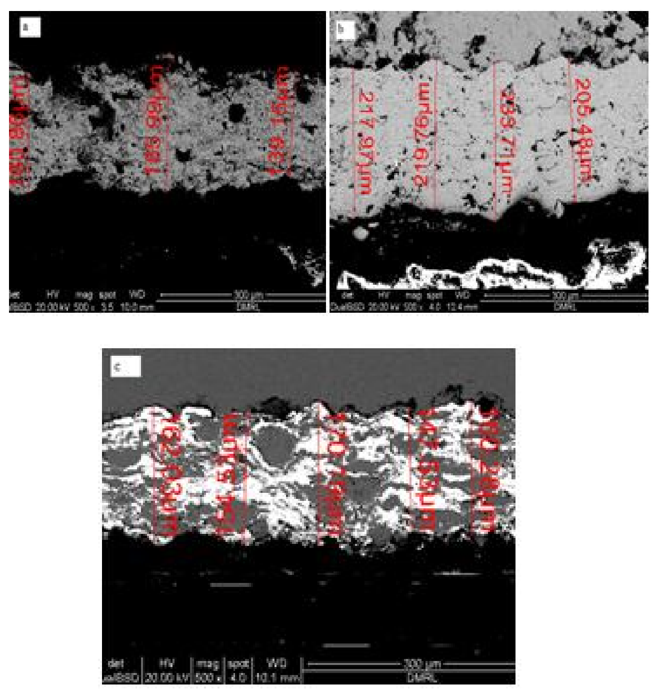



**Fig. 3 Thicknesses of the Coatings (a) WC-Co (b) NiCrBSi (c) WC-Co+NiCrBSi**

The thickness of the coatings was measured and tabulated and it can also be observed from the optical micrographs that the porosity is < 2% as shown in figure 3. The microhardness of the blend is high.

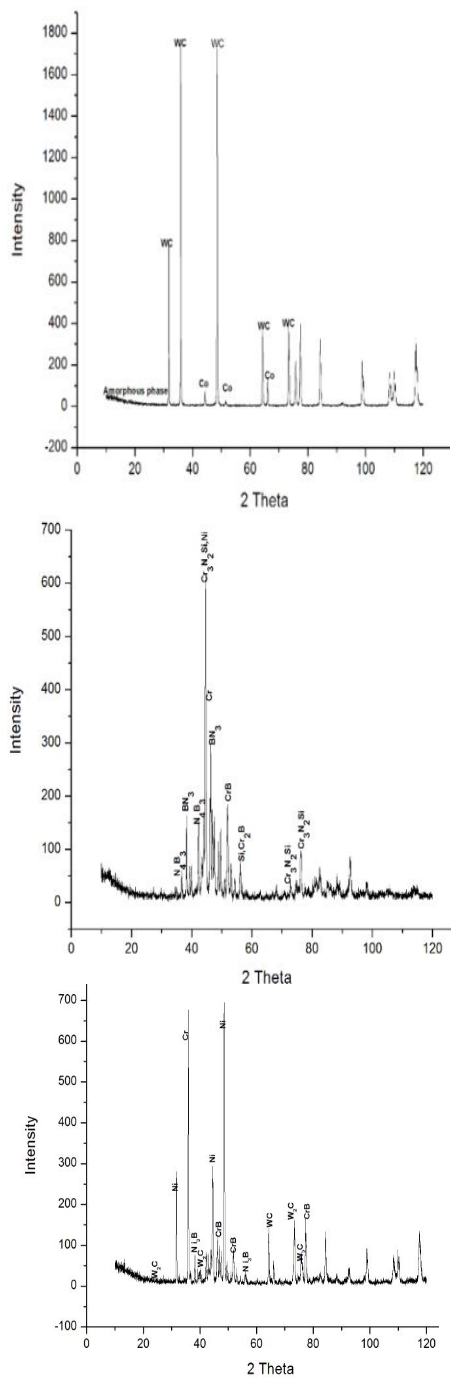
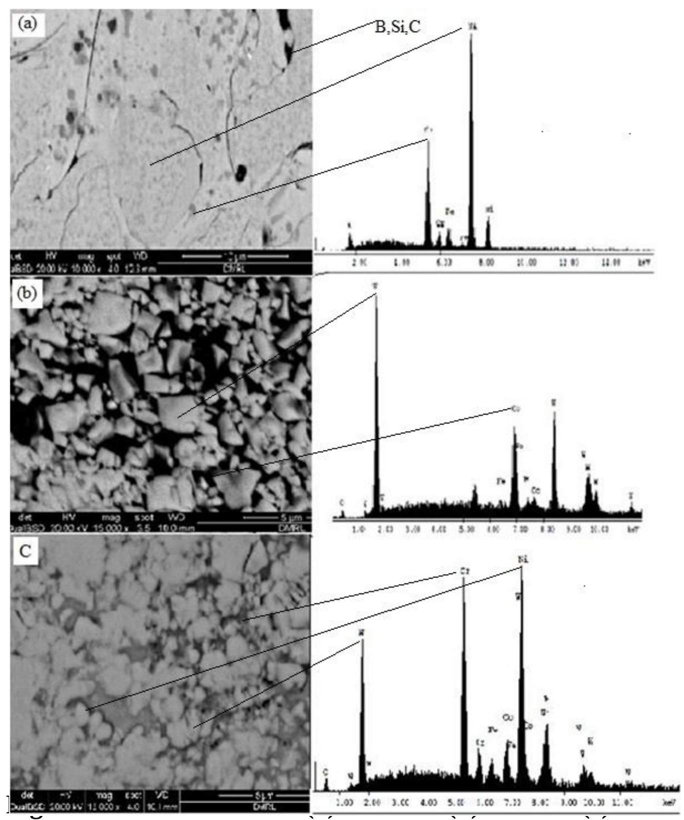



**Fig. 4 XRD of the Coatings (a) WC-Co (b) NiCrBSi (c) WC-Co+NiCrBSi**

Figure 4 shows the new phases formed in the powders selected. The new phases formed in 4(c) are CrB, Cr and Ni and these formed the basis for the improved properties.




**WC-Co+NiCrBSi**

Figure 5 shows the SEM and EDAX of the coatings. In Figure 5(a) the white phase is Nickel and the grey phase is chromium and the black spots specify the removal of borides and silicides which might have been wiped away while polishing of the samples before taking the SEM. In figure 5 (b) the white flakes correspond to tungsten and the dark phase to cobalt. In figure 5(C) the white phases with spherical atomic structure corresponds to Nickel, non spherical white flakes correspond to tungsten; the dark phase is the chromium, and very small amount of Cobalt. In the blend of WC-Co and NiCrBSi, the NiCrBSi serves as a good binder as the hard carbide particles have strong binding with coating which resulted in good wear resistance.

## 6. Empirical Modelling

### 6.1 Sliding wear

In current work RSM was applied to develop a mathematical model as a multiple regression equation. In RSM the response variable is a surface to which the model is fitted. The effect of Evaluation of various factors on the responses was modelled considering a second-order polynomial response surface mathematical model given by

$$Wear = b_{0+}\sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} Sq(x_i) + \sum_{i=1}^{k-1}\sum_{j=i+1}^{k} b_{ij} x_i x_j + \in r$$

This equation of wear (assumed surface) contains zeroth, first and second order terms variables (A,B and C), where $b_0$ is the mean response over all the test conditions (intercept); bi is the slope or linear effect of the input factor $x_i$ (the first-order model coefficients); $b_{ii}$ the quadratic coefficients for the variable i (linear by linear interaction effect between the input factor $x_i$ and $x_i$); and $b_{ij}$ is the linear model coefficient for the interaction between factor i and j. Significance testing of the coefficients, adequacy of the model, and analysis of variance were carried out using the Design Expert Software to find out the significant factors, square terms, and interactions affecting the response. The analysis of variance (ANOVA) is shown in Table 6,7 & 8. The ANOVA shows the significance of various factors and their interactions at 95% confidence interval. ANOVA shows the ''Model'' as ''Significant'' while the ''Lack of fit'' is ''Not significant'' which are preferable from a model point of view. The probability values <0.05 in the ''Prob. > F'' column indicates the significant factors and interactions. The main factors and their interactions are included in the final wear model, while the insignificant interactions are excluded. The Model F-value of 67.37 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, AC are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

**Table 6: ANOVA of WC-Co Coating**

| | | | |
|---|---|---|---|
| Std. Dev. | 37.65 | R-Squared | 0.9688 |
| Mean | 167.95 | Adj R-Squared | 0.9545 |
| C.V.% | 22.41 | Pred R-Squared | 0.9434 |
| PRESS | 33467.80 | Adeq Precision | 24.829 |

The "Pred R-Squared" of 0.9434 is in reasonable agreement with the "Adj R-Squared" of 0.9545.

**Table 7: ANOVA of NiCrBSi Coating**

| | | | |
|---|---|---|---|
| Std. Dev. | 155.73 | R-Squared | 0.9429 |
| Mean | 421.21 | Adj R-Squared | 0.9096 |
| C.V.% | 36.97 | Pred R-Squared | 0.8257 |
| PRESS | 8.882E+005 | Adeq Precision | 14.396 |

The "Pred R-Squared" of 0.8257 is in reasonable agreement with the "Adj R-Squared" of 0.9096

**Table 8 ANNOVA of WC-Co+NiCrBSi**

| | | | |
|---|---|---|---|
| Std. Dev. | 54.10 | R-Squared | 0.8788 |
| Mean | 118.34 | Adj R-Squared | 0.8082 |
| C.V.% | 45.71 | Pred R-Squared | 0.780 |
| PRESS | $1.020\text{x}10^5$ | Adeq Precision | 11.772 |

The "Pred R-Squared" of 0.780 is in reasonable agreement with the "Adj R-Squared" of 0.8082; i.e. the difference is less than 0.2.

Wear Model for WC-Co: Coded form

$$Wear = 167.95 + 127.49A + 18.49B + 115.71C - 6.60AB + 113.92AC + 16.58BC + \in \quad \text{(Eq 1)}$$

Un coded form:

$$Wear = 17.05399 + 2.2928 \times 10^{-3}A + 8.46184B - 0.52385C - 1.76139 \times 10^{-3}AB + 9.8027 \times 10^{-5}AC + 0.21397BC + \in \quad \text{(Eq 2)}$$

Wear Model for NiCrBSi: Coded form

$$Wear = 508.6488 + 258.9055A - 45.96B + 404.065C + 60.8765AB + 253.2045AC - 45.082BC - 174.884A^2 + \in \quad \text{(Eq 3)}$$

Un coded Form:

$$Wear = 223.35420 - 0.048553A - 100.3220B + 2.42646C + 0.016239AB + 2.17883 \times 10^{-4}AC - 0.58170BC + \in \quad \text{(Eq 4)}$$

Wear Model for WC-Co+NiCrBSi: Coded form

$$Wear = 118.34 + 90.39A + 12.48B + 73.08C + 29.67AB + 70.52AC + 8.24BC + \in \quad \text{(Eq 5)}$$

Un coded form:

$$Wear = 16.59733 - 0.019564A - 55.1447B - 0.24952C + 0.007915AB + 6.07 \times 10^{-5}AC + 0.106277BC + \in \quad \text{(Eq 6)}$$

### 6.2 Effect of Individual variables on wear

The sliding wear response model in terms of coded level of factors (A, B, C) and interactions (AB, BC, AC, CD, BD) of WC-Co, NiCrBSi, and a blend of WC-Co+NiCrBSi is shown in equations1 in 6. The equation 1, 3 & 5 in terms of coded factors is used to make predictions about the response for the equation in terms of coded factors (-1 for Low Level and 1 High Level). The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients The constants 508.04, 167.95,118.34 for WC-Co, NiCrBSi & WC-Co+NiCrBSi respectively in these model equations indicates the overall mean sliding wear for all test conditions. The coefficients associated with A, B, C indicates the extent of influence on the sliding wear by each of the factors. The constant terms 17.05, 135.67, 16.59 in equations 2,4 &6 are the intercepts and represents the average value of the response wear when all factors are at the middle level for WC-Co, NiCrBSi and WC-Co+NiCrBSi respectively. The coefficients of the terms distance, load, and temperature indicate the extent of influence the factors have on the response variable. Wear test is performed at the following parameters for model validation.

**Table 9: Wear test parameters to validate the model**

| Run | Distance (m) | Load (kg) | Temperature |
|---|---|---|---|
| 1 | -1 | -1 | 1 |

**Table 10: Validation of wear models of NiCrBSi, WC-Co, NiCrBSi+WC-Co coatings**

| | RUN1 | | | RUN2 | | |
|---|---|---|---|---|---|---|
| Coating | Model Wear | Experimental Wear | % Error | Model Wear | Experimental Wear | % Error |
| NiCrBSi | 1.49 | 1.62 | 8 | 1.49 | 1.56 | 4 |
| WC-Co | 22.55 | 23.64 | 4.6 | 22.55 | 23.88 | 5.5 |
| WC-Co +NiCrBSi | 76.26 | 78.59 | 3 | 76.26 | 78.33 | 2.6 |

The validity of the wear model is confirmed by conducting confirmation tests at the parameter levels shown in table 9. The experimental values and the modelled values are shown in table 10. It is observed that the variation between the experimental and theoretical values for the coatings is of the order 2-8%.

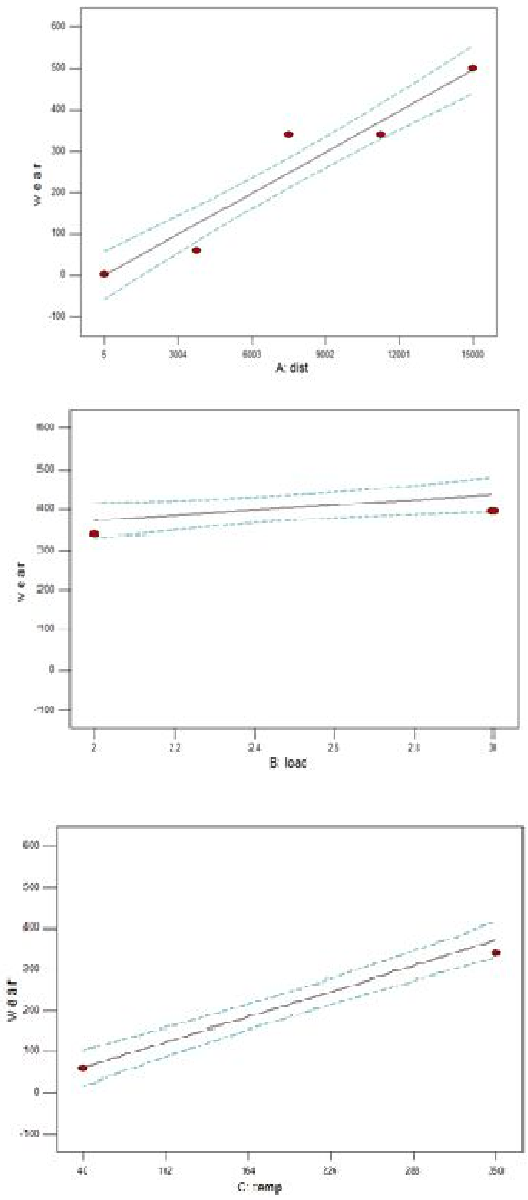
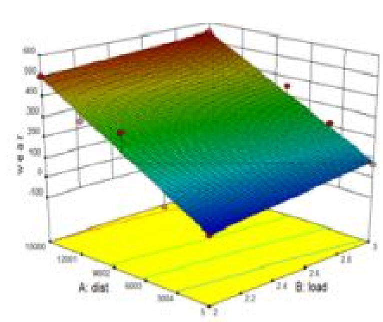



**Fig. 5 (a) Graphs showing the variation of wear with respect to single parameter for WC-Co Coating**



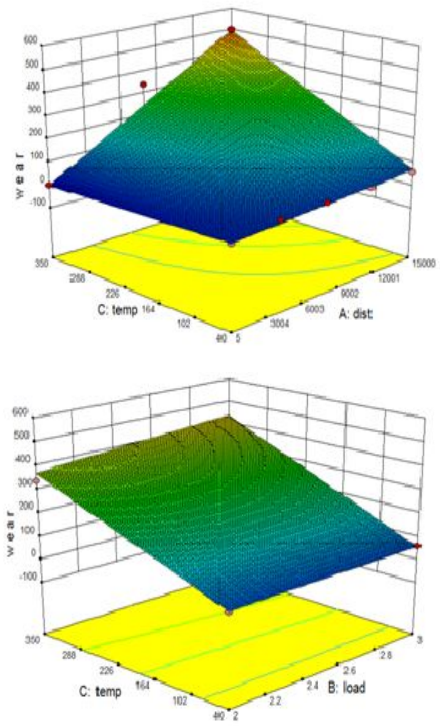
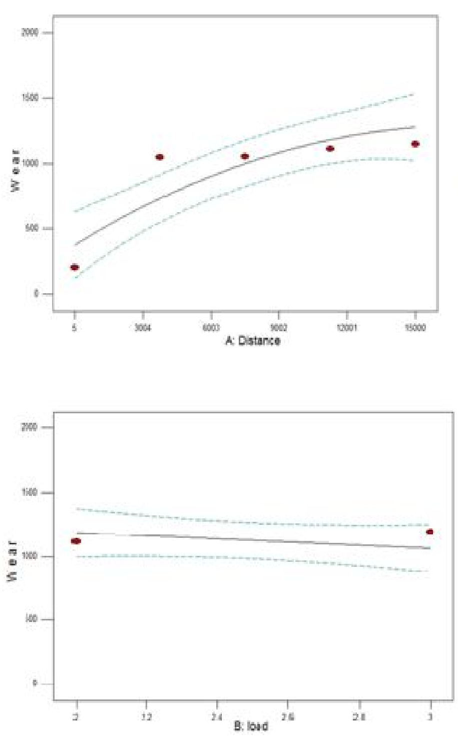



**Fig. 5(b) 3D graphs showing the interaction effect of different parameters for WC-Co Coating**



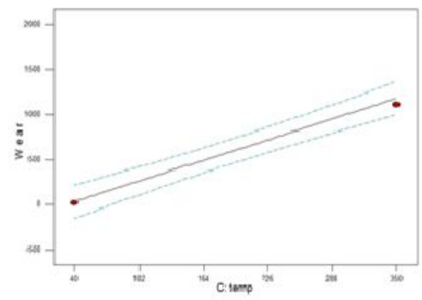




**Fig. 5(c) Graphs showing the variation of wear with respect to single parameter for NiCrBSi Coating**

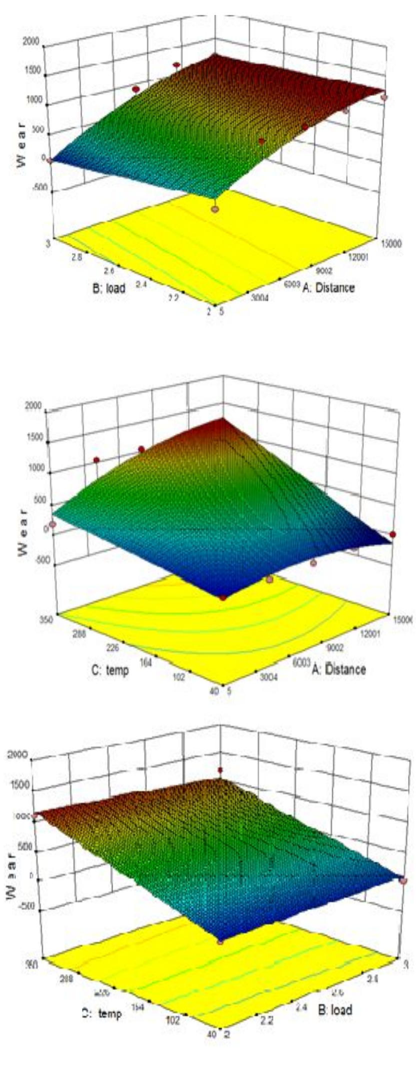



**Fig. 5(d) 3D graphs showing the interaction effect of different parameters for NiCrBsi Coating**

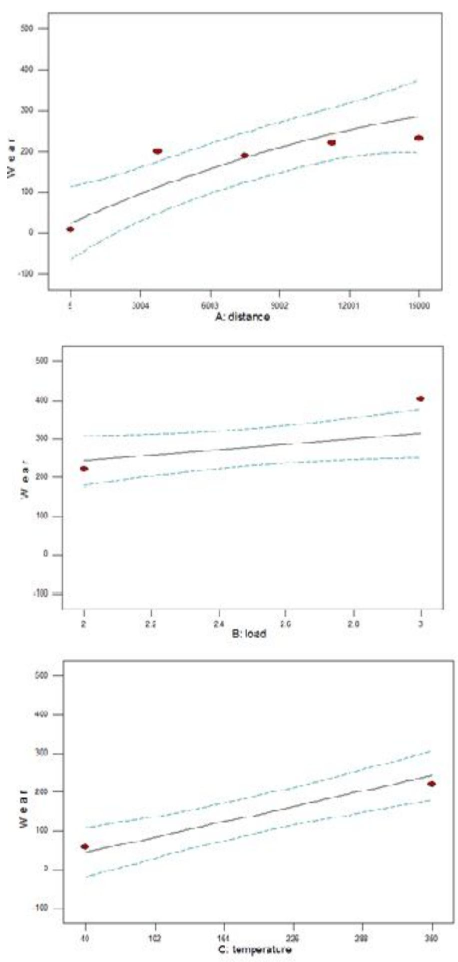
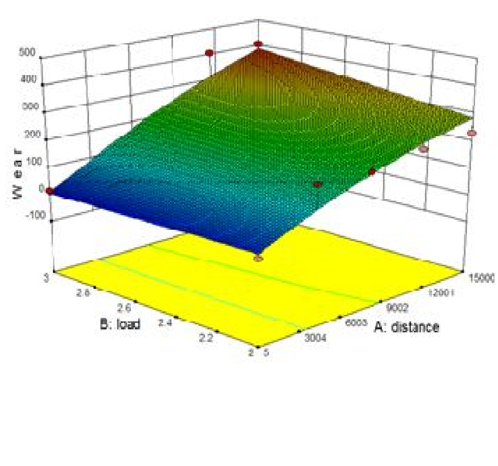



**Fig. 5(e) Graphs showing the variation of wear with respect to single parameter for WC-Co+NiCrBSi Coating**



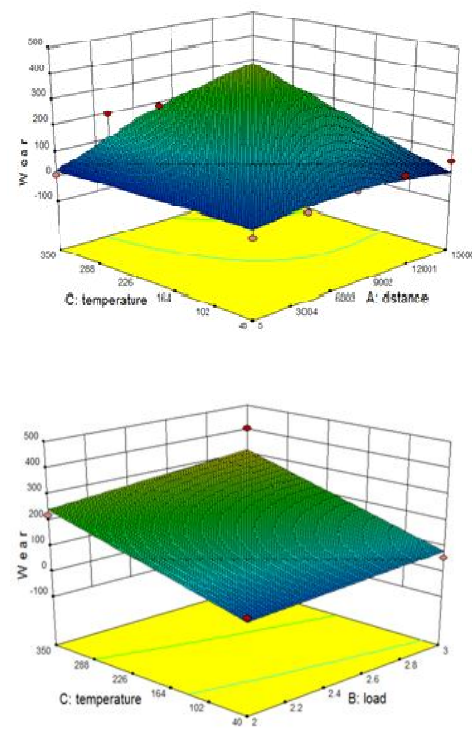




**Fig. 5(f) 3D graphs showing the interaction effect of different parameters for WC-Co +NiCrBSi Coating.**

The effect of Load, Distance, and Temperature on wear is shown in figure 5 (a-f).From graph 5a, 5b sliding wear for the coating WC-Co It is observed that the increase in the input factors load and temperature have the same effect of increasing the wear, but the increase in temperature has greater effect when compared to load at low sliding distances. From graphs 5c, 5d it is evident that the coating NiCrBSi has good wear resistant properties at room temperature, but as the temperature increases the resistance to wear decreases. At high temperature irrespective of loading conditions wear is high for NiCrBSi coating. As the distance increases irrespective of the loading condition the wear is found to be high in case of NiCrBsi .This may be due to fact that the carbide, borides and silicates may be ripped away during the test. With varying load at constant temperature wear is almost constant. The blend NiCrBSi +WC-Co shows good wear resistance at high temperatures also as evident from the graph. This can be explained by the fact the addition of Chromium to WC-Co, improves binding of the metallic matrix with WC, Ni grains and provides better wear properties. The figures 5b, 5d & 5f shows the interaction affect of load and distance on response (R) wear rate. Whereas in case of the WC-Co the wear rate is uniform at low load and as load increases the wear rate increased by 5 %. The blend demonstrates good wear properties at high load at relatively low distances which makes them a potential substitute for coatings in brake linings of cars. Though coating WC-Co exhibits low wear initially, but as load increases the wear is high when compared to blend.

## 7. Conclusions

The Sliding wear of WC-Co, NiCrBSi and the blend were successfully evaluated and modelled. The results showed that the blended coating exhibited good wear resistance at high temperatures and loads. Addition of Cr to WC-Co improves binding of the metallic matrix with WC, Ni grains and provides better wear resistance at higher temperatures. RSM can be used for developing a statistical model for predicting and determining the wear behaviour of the coatings in terms of the individual factors (sliding distance, load, temperature) and also the combined effects on different coatings.

## Acknowledgments

Authors sincerely thank DMRL for SEM and Micro hardness measurement; authors also thank VTC, Visakhapatnam for coating the samples.

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