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WEAR BEHAVIOR OF THERMAL SPRAY COATING DEPOSITED ON DIE STEEL

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Abstract-In the present investigation, 65% (NiCrSiFeBC)+35% (WC-12Co) coatings were deposited on H13 die steel with High-Velocity Oxy-fuel (HVOF) thermal spray technique. H13 die steel is mainly used as a tool material in hot forming of high strength steels. The aim of this research work is to study the performance of the selected coating composition. The as-sprayed coatings were characterized using scanning electron microscopy (SEM), Energy Dispersive Spectroscopy (EDS), optical microscopy and X-Ray diffractometry (XRD). The adhesion strength of the developed coatings was also determined. The physical properties like surface roughness, porosity, and microhardness of as-sprayed coatings have also been evaluated. Pin on disc tests was carried out to analyze wear behavior of coatings in comparison with uncoated H13steels. The results have been discussed with respect to the existing literature.
Keywords: hot forming steels; coatings; HVOF spray; characterization; wear

1. INTRODUCTION

Hot forming processes are being used since old times for metal forming and still have a big share in the fabrication of metal products. The forming dies are the most crucial components in every hot forming operation because it usually gives the object its final shape. As the die usually is costly to fabricate it has a key effect on the production costs. A high quality dies with a long lifespan is vital for a successful and cost-effective production [1]. Failure of hot forming dies can be a result of various mechanisms such as wear, plastic deformation, gross cracking, thermal fatigue and mechanical fatigue [2-4]. In hot forming processes such as hot rolling and stamping, medium alloy hot work dies steels such as H11 and H13 are used because of their ability to resist thermal softening along with sufficient strength and toughness to withstand the stresses encountered during hot forming. This steel has chromium as the main alloying element. Tungsten, vanadium, and molybdenum are also added. These elements provide deep hardening characteristics and resistance to abrasion and thermal softening at high temperatures. Molybdenum increases resistance to thermal softening, vanadium improves wear and thermal fatigue characteristics [5].

NiCrBSi coatings have high corrosion resistance, adhesive and abrasive wear resistance at elevated temperatures [6, 14-19]. Chromium and boron additions improve the wear and corrosion resistance and also enhance the mechanical properties such as the hardness of the coatings by supporting the formation of hard carbides, thus increasing the wear resistance of the coatings. Silicon promotes the self-fluxing properties [7, 20, 21]. It is found out from the literature that the wear resistance of NiCrBSi coatings can be significantly increased by adding carbides such as WC, VC, WC-Co, TiC to the metallic matrix. Cermet coatings, mainly carbide-type coatings, for example, WC-Co have given great results in different industrial applications. These coatings consist of carbide particles reinforced in the metallic matrix, combining the properties of ceramic-carbide-type materials, high hardness and toughness and ductility of metals. Addition of WC-Co thus increases hardness and wear resistance of coatings [8, 23, 24].

Thermally sprayed coatings have been extensively used for modifying the component surface properties in a variety of industrial applications, largely for wear resistance, thermal barrier and corrosive environment [9-11]. In thermal spray techniques, droplets of molten or semi-molten material are generated and projected onto the surface of the workpiece. The particles quickly solidify and stick both to one another and to the substrate to form dense protective coatings. Materials ranging from polymers through to metals, cermets, and ceramics are routinely sprayed using this technique [12].

High-velocity oxy-fuel (HVOF) thermal spraying has proved to be one of the better methods for deposition of conventional feedstock powders because of higher velocities and lower temperatures experienced by powder particles. Result in less decomposition of carbides during spraying resulting in more wear-resistant coatings, with higher levels of hard carbide particles and less porosity [13].

From the literature, it has been concluded that application and characterization of thermal spray coatings on hot forming die steels had not yet been studied in sufficient detail. The main objective of the current investigation is to analyze experimentally the high-temperature wear behavior of HVOF spray coatings deposited on the die steel.

2 EXPERIMENTAL PROCEDURES

2.1 Preparation of Substrate Material and Development of Coating

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Selection of substrate material has been done after exhaustive literature survey. H13 cylindrical pins of 8 mm diameter and 50 mm length have been used as a substrate. Thereafter, NiCrBSi and (WC-12Co) powders with an average particle size of 45±15 µm were chosen. The two powders were mechanically blended in the ration 65:35. The powders are mixed by using ball milling apparatus for 6 hours. The chemical composition of the coating powder was [65%(Ni-71.7, Cr-15.7, Si-4.27, Fe-4.17, B-3.35, C-0.81) + 35% (88%WC-12%Co)]. The matrix phase is mixed with the dispersed phase by weight. The resulting coating composition 65% (NiCrSiFeBC) blended with 35% (WC-12Co) was deposited onto H13 steel substrates with the HVOF spray technique. The coatings were developed at Metallizing Equipment Company Pvt. Ltd., (MECPL) Jodhpur. The substrates were shot blasted prior to spraying to attain proper adhesion between coating and substrate. Coatings of thickness 180-220 µm were obtained. The coatings were deposited by commercial HVOF (HIPOJET-2700) apparatus operating with oxygen and liquid petroleum gas (LPG) as input gases. The substrate steels were cooled by compressed air jets during and after spraying. The spraying parameters adopted for the HVOF spray process are shown in Table 1. Powder feed rate for the NiCrBSi powder blended with WC-Co powder was 55 g/min.

2.2 Characterization of the As-Sprayed Coatings

The coated specimens were subjected to XRD analysis to identify various phases formed on their surfaces. The XRD analysis of the composite powder and the as-sprayed specimens was performed to identify the various phases present. It was carried out using an Expert Pro PAN analytical company (Netherlands) Model Expert Pro MPD with CuKα radiation and nickel filter at 20 mA under a voltage of 35 kV.The specimens were scanned with a scanning speed of 1 Kcps in 2θ range of 20º to 100º and the intensities recorded at a chart speed of 1 cm/min with 2º/min as goniometer speed. Assuming height of the most prominent peak as 100%, the relative intensities were calculated for all the peaks. The diffractometer

Table 1: Spray parameters as employed during HVOF spraying

Oxygen flow rate (SLPM)	300-350
Fuel (LPG) flow rate (SLPM)	60-80
N_2 flow rate (SLPM)	15-20
Oxygen pressure (kg/cm^2)	9
Fuel pressure (kg/cm^2)	5.6
Powder pressure (kg/cm^2)	3
Spray distance (mm)	250
Flame temperature (ºC)	5000
Particle temperature (ºC)	2200
Particle velocity (m/s)	450-500

interfaced with BrukerDIFFRAC^{plus} X-Ray diffraction software provides'd' values directly on the diffraction pattern. Expert Hi score Software was used to for identification of various phases and graph were plotted with defined 'd' values.

Further, the surface morphology of the coating powders and the surface, as well as, cross-sectional morphology along with elemental composition of the as-sprayed coatings was studied with the help of JEOL scanning electron microscope (SEM), JEOL JSM 6610LV; having resolution of 3 nm and magnification 300,000X equipped with energy dispersive spectroscopy (EDS) to understand the structure and composition of the coatings and identify oxide inclusions, unmelted particles, pores etc. The SEM micrographs along with EDS spectrum were taken with an electron beam energy of 20 keV. Although the compositions correspond to selected points on the as-sprayed surfaces, still the data could be useful to understand the formation of desired compositions in the coatings. To study the cross-sectional morphology, the coated specimens were sectioned with a diamond cutter (BAINCUT-LSS, Metallography Low Speed Saw, Chennai Metco Pvt. Ltd., Chennai, India). Thereafter, the cut sections were hot mounted in BAINMOUNT-H (Hydraulic Mounting Press, Chennai Metco Pvt. Ltd., Chennai, India) with transoptic powder so as to show their cross-sectional details. This was followed by polishing of the mounted specimens by a belt sanding machine having emery belt (180 grit). The specimens were then polished manually down to 1000 grit using SiC emery papers. Final polishing was carried out using cloth wheel polishing machine with 1 µm alumina powder suspension. Specimens were then washed and dried before being examined under scanning electron microscope. This SEM/EDS and XRD analysis was performed at Indian Institute of Technology, Roorkee (India).

The coating bond strength for the compositions of coatings was measured according to the ASTM Standard C-633 on Tensile Bond Strength Testing Machine. The values were compared with the other studies. Porosity was measured with Image Analyser software Envision 3.0 and the surface roughness was measured by Mitutoyo Surface Roughness Meter at MECPL, Jodhpur. Microhardness of the coatings was measured by microhardness tester (model-WILSON) with digital display. An indenting load was 2.45 N i.e. 300 GF was applied on the square pyramidal diamond indenter for penetration and the hardness values were based on the relation $Hv = 0.1891 \times \frac{F}{d^2}$ (Where F is load in N and d is the mean of the indentation diagonal length in mm). Dwell time for indentation was taken as 10 seconds. Each reported value of the microhardness is a mean of three observations.

Tribological studies were done on a pin-on-disc tribometer under unidirectional dry sliding conditions. Before carrying out wear and friction experimentation, specimens were polished using sand papers of different grit sizes and R_a values for each specimen were reduced to less than 1 µm. The wear and friction tests were carried out according to ASTM wear testing standard G99-04. The pin test specimen was loaded against a rotating 20MnCr5 (hardened up to 60-80 HRC) disc test specimen. The wear and friction test parameters on tribometer were decided based on the typical values encountered in the hot metal forming processes. The tests were conducted under the constant load of 25 N sliding speed 0.5 m s^{-1}, sliding distance of 1500 m at room temperature. The total test duration was 50 min. The friction is measured by using a strain gauge force transducer. This tribometer is equipped with a computerized data acquisition and control system for controlling and monitoring of various parameters. Before and after the wear tests, the pin specimens were weighted to a precision of 0.1 mg and the weight change was considered as the wear weight loss. The wear volume loss (mm^3) was calculated for each test. All tests where repeated and good reproducibility was observed. The morphology and surface topography of test specimens were analyzed by using SEM/EDS technique.

3. RESULTS AND DISCUSSION

3.1 Visual Examination of the Coatings
Macrographs of the uncoated pins (Fig. 1) and as-sprayed samples (Fig. 2) are shown. Coatings are grey in color. The coatings have the smooth surfaces. Further these coatings are found to be free from any visible surface cracks.

3.2 *SEM Analysis of Coating Powders*
The SEM morphology of the powders is shown in figure 3. It is found from that the particles of NiCrBSi and WC+Co powders have spherical morphology. Particles of WC-Co have a porous appearance. WC-Co particles were uniformly dispersed in NiCrBSi phase.

Fig. 1 Camera macrographs of steel pins


Fig. 2 The camera macrographs of as-sprayed coatings on H13 substrate (a) top surface (b) full sample

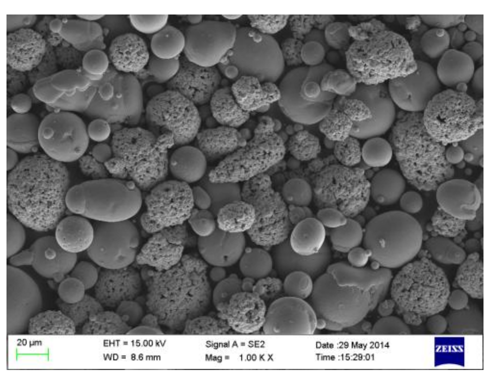


Fig. 3 SEM morphology of [65%NiCrBSi+35 %(WC-Co)] powder

3.3. XRD Analysis of Coating Powders
XRD diffractogram for the [65%NiCrBSi+35%(WC+Co)] powder is shown in Fig. 4 on reduced scales. Very Strong phase indications of Ni, CrB and WC phase was observed, whereas NiB and W_2C were revealed as medium intensity phases and W as a weak intensity phase.

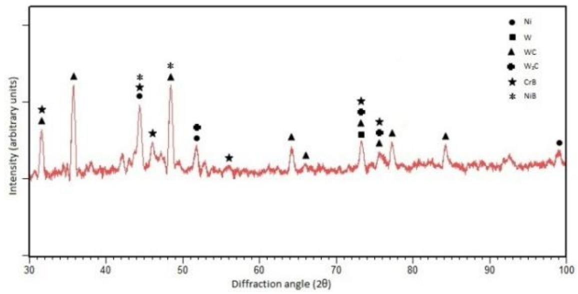


Fig.4 X-ray diffraction pattern for 65 (NiCrBSi)+35(88WC-12Co) powder

3.4 Evaluation of Coating Bond Strength, Porosity, Surface Roughness and Microhardness

The coating bond strength measurements were done at MECPL, Jodhpur on Tensile Bond Strength Testing Machine with ASTM Standard C-633. The coating bond strength values for the as sprayed specimens were 68.99 MPa. Porosity was found to be 1.71%. The measured values of surface roughness were 6.45 µm. Average micro hardness values for the coating were found out to be 719 Hv.

3.5 Scanning Electron Microscopy /Electron Dispersive Spectroscopy (SEM/EDS) Analysis of As-sprayed Coatings

FE-SEM/EDS morphologies for the HVOF sprayed coating on the tool steels are shown in Fig. 5. The SEM image of 65%(NiCrSiFeBC)+35%(WC-12Co)] coatings are found to have uniformly distributed splats along with the flattened portions depicting the fully molten areas. The EDS analysis showed the presence of all the elements present in the coating composition. The coatings are found to be free from any cracks. Some pores are visible on the surface.

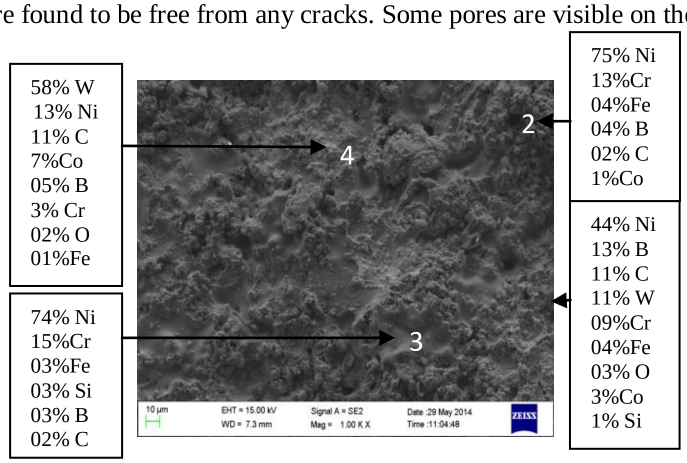


Fig.5 Surface scale morphology and EDS analysis of H13 tool steel with HVOF sprayed (NiCrBSi)+35%(WC-12Co) coatings showing elemental composition (%) at selected point.

3.6 XRD Analysis

XRD diffractograms for the surfaces of the HVOF sprayed [65% (NiCrSiFeBC) + 35% (WC-12Co)] coatings are shown in Fig. 6. Ni, WC and W were identified as the strong intensity phases and CrB_2 and W_2C as the medium intensity phases. similar phases are reported in literature. Ramesh at el [25] reported presence of γ-Ni solid solution, WC and Co as a principal phase. During the HVOF spraying, the flame temperature is usually lower than other thermal spray process, and the particle speed is very high resulting in shorter exposure time of powder with air so the decomposition and decarburization of WC are not serious. The HVOF spraying parameter adopted resulted in high retention of WC in the matrix [25].

3.7 Cross-sectional Morphology of As-sprayed Coatings

Cross-sectional morphologies of the HVOF sprayed coated die steel is shown in Fig 7. The cross-sectional images of the showed the building of coating thickness due to deposition and resolidification of molten or semi-molten droplets. The required thickness of the coatings was achieved. The coating

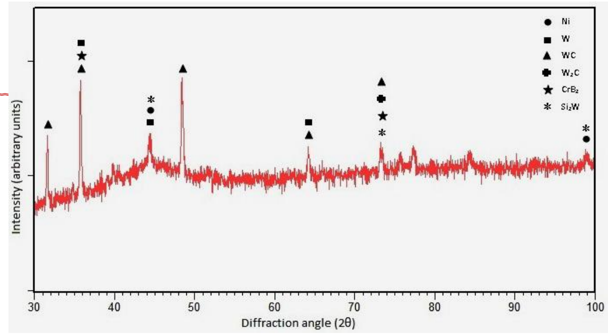


Fig.6 X-ray diffraction pattern for 65 (NiCrBSi) +35(88WC-12Co) coating on H13 steel by HVOF spray process

substrate interface is free from defects and coatings seem to have good adherence with the substrate**.**

3.8 Visual examination of worn surfaces

The worn surfaces of the uncoated and coated AISI H13 specimens subjected to wear and friction experimentation at room temperature (RT) observed by naked eye are shown in Figure 8. The pictures clearly indicate the presence of shallow sliding marks on the surface on the surafce of coated specimen and Deep plowing grooves are visible on uncoated H13 pin showing that the substrate suffers severe wear.

3.9 Wear and frictional behavior

Figure 9 (a) shows that SEM morphology of the uncoated worn specimen subjected to wear and friction experimentation on pin-on-disc tribometer. Severe wear was observed on the uncoated specimen thus contributing to more weight loss. The morphology indicates the presence of many regions of material pull out, delamination and scratch marks. Abrasive wear tracks can also be seen. The higher weight loss of the uncoated specimen was observed as shown in Fig. 10. On the other hand, the coated specimen showed the presence of small debris on the worn surface. Light abrasive wear tracks are visible. Formation of abrasive wear mechanism may be attributed to the disc counter body (two-body abrasive wear) or to the debris (three-body abrasive wear) [22].

Coating is by and large intact to the surface with presence of both coating powders. Lesser weight loss was observed indicating the better wear resistance of coated pin (Fig. 10). Figure11 represents the coefficients of friction obtained during the wear test at different conditions for the uncoated and HVOF coated AISI H13 specimens. It was observed that HVOF sprayed coatings exhibit lower value of COF which is due to the high value of surface hardness of coatings. WC particles can effectively decrease the penetration of abrasive particles because of their superior hardness and strength. The γ-Ni matrix having excellent ductility and toughness plays a dominant role in preventing the carbide particles from removing. They are main factors for improving the wear resistance of the coatings [26,27,28]. Dissolution of tungsten carbide in the Ni-rich matrix, also enhances the sliding wear resistance of the coating.

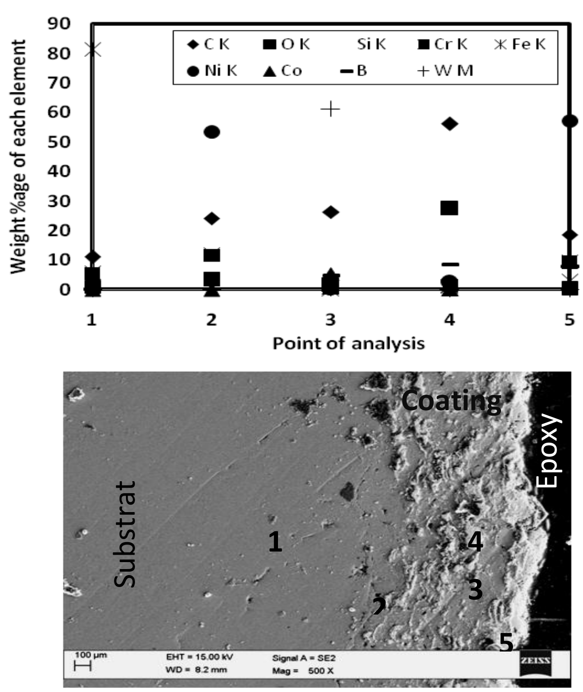


Fig.7 Cross-sectional SEM/EDS morphology of HVOF sprayed 65%NiCrBSi + - 35(WC-12Co) on H13 substrate.

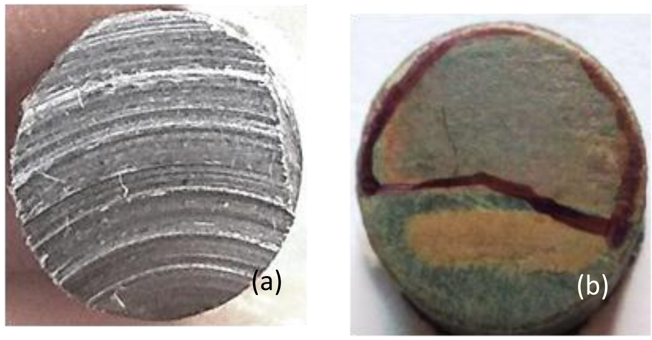
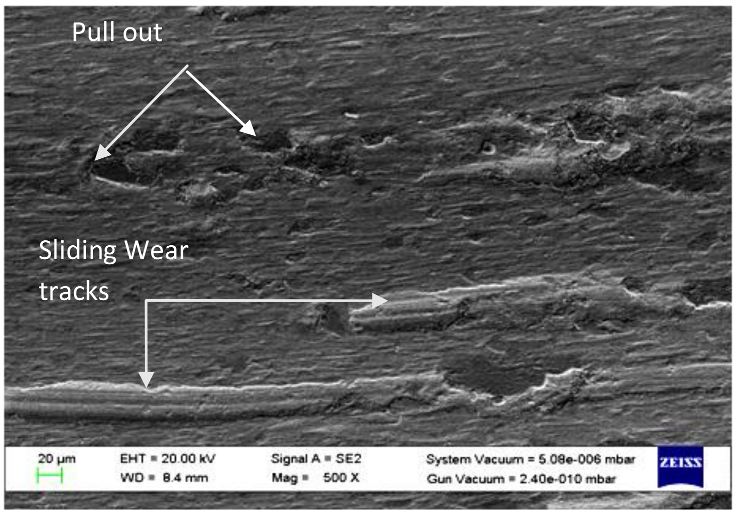
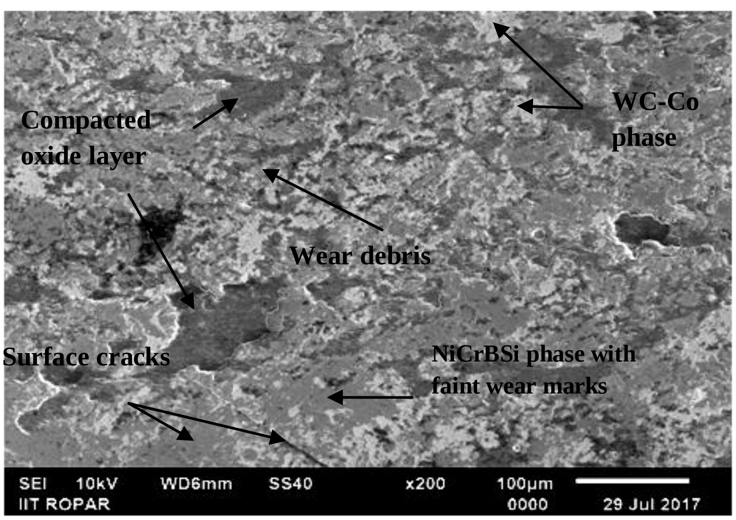


Fig.8 Macrographs of worn surfaces of HVOF sprayed (a)AISI H13 uncoated pin (b) 65 NiCrBSi - 35(WC-12Co) coatings specimens tested at RT & 25N.



(a)



(b)

Fig.9 SEM micrographs of worn surfaces of specimens after 50 min run (a) uncoated and (b) HVOF sprayed 65 NiCrBSi+35%(WC-12Co) coated AISI H13 specimens tested at RT and 25N

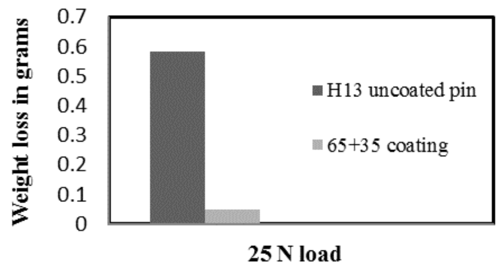


Fig.11 Weight loss for the uncoated and 65 NiCrBSi - 35(WC-12Co) HVOF spray coated H13 steel pin subjected to wear and friction testing at room temperature and 25N load.

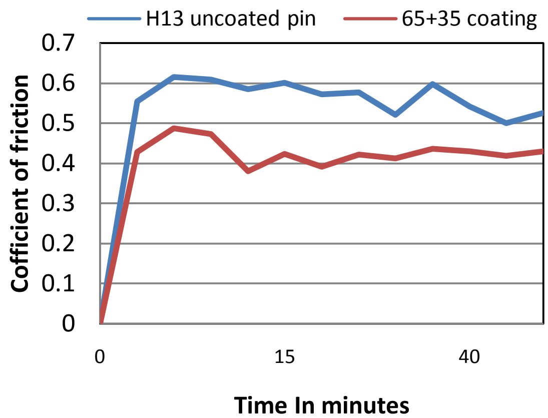


Fig.12 Coefficient of friction of uncoated and 65 NiCrBSi - 35 (WC-12Co) HVOF sprays coated H13 steel pin subjected to wear and friction testing at room temperature and 25N load.

4. CONCLUSIONS

1. Coatings of thickness ranging from 180-220 µm were obtained using HVOF process. Porosity of as-spray coatings was 1.79%. These values are lower than other processes due to high particle velocities resulting in high impact forces and compaction of splats leading to formation of dense coatings.
2. Hardness of coatings is a very important property and these coatings exhibit hardness values (719 Hv) which are quite higher than heat treated tool steels. Higher hardness of coatings can be attributed to hard phases present in coatings and high velocity and impact forces created in HVOF process.
3. Bond strength is another important parameter which affects life of thermal spray coating. In this study 65 NiCrBSi-35(WC-12Co) coating on H13 showed value of 68.99 MPa. This high value indicates a strong mechanical bond between coating and substrate.
4. These coatings exhibit good microstructure, physical and mechanical properties which make them suitable candidate for testing on hot forming applications.
5. Wear results clearly justify application of coating over H13 die steel. Weight loss of coated specimen was many times lower than uncoated pin and values of coefficient of friction was also lower.
6. Severe a wear occurred in uncoated H13 pin causing removal of layers of pin material (delamination) however in coated pin mild wear occurred as oxide layers are formed on the surface which aid in wear resistance. Very faint scratch marks are visible on coatings.

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