# **A Study of High-Velocity Combustion Wire Molybdenum Coatings**

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**In this paper, coatings manufactured using the high-velocity combustion wire (HVCW) spray process have been studied. Molybdenum coatings were prepared in this study, and wavelength dispersive x-ray analysis (WDX) investigations were carried out to ascertain the oxygen content of the coating and its distribution. The x-ray diffraction (XRD) analysis of the coating was also carried out to determine the phases present in the coating. Based on the above data, the authors explain the HVCW-sprayed molybdenum coating microstructure properties. These coatings were also sprayed using a modified aircap design. The parameters varied for the molybdenum coatings by HVCW and were (1) the distance of the substrate from the spray gun and (2) the wire feed rate of the gun. The wear test and coefficient of friction measurements were also carried out for the coatings. Air plasma spraying of Mo-25% NiCrBSi coatings was carried out, and these coatings were further checked for wear friction properties.**

**Keywords** HVCW, HVOF, molybdenum, oxidation, SEM, WDX

### **1. Introduction**

High-velocity oxy-fuel (HVOF) coatings are being increasingly used in the industrial environment. The HVOF coatings offer a qualitative advantage over air plasma sprayed (APS) coatings. However, most of the HVOF coatings are applied using powders as the feed stock. The high-velocity combustion wire (HVCW) spray system uses wire as the feed stock, which benefits the process economics, because wires are cheaper than powders. As well, the operating cost of HVCW is lower than most other thermal spray systems.

Molybdenum coatings were prepared with an HVCW system under a different set of parameters; the coatings thus prepared were characterized using a scanning electron microscope (SEM), wavelength dispersive x-ray analysis (WDX), and x-ray diffraction (XRD) phase analysis. A wear test of the coatings was also carried out.

Oxidation in the coating alters the microhardness and the wear performance of the coating. The mechanism of oxide formation in the coatings is discussed; it is co-related to the observed oxygen content of the coatings. The mechanical properties such as wear friction were also studied by the pin-ondisk wear test. A Mo-NiCrBSi coating by air plasma spraying was prepared and characterized for its wear friction properties. This enabled a comparison of the HVCW APS coatings.

## **2. The Mechanism of In-Flight Reactions in the HVCW Coating**

There are differences in the mechanisms of in-flight reactions between the powder HVOF the HVCW spray systems.[2] The

heat imparted to the powder particles in the HVOF powder system is expected to be less than the heat transfer to the wire droplets in HVCW. This arises because, in most modern HVOF powder guns, the powder is injected in the barrel of the gun where the gas jet is in the expanded condition.<sup>[6,11]</sup> The conditions prevailing here are of low temperature but high velocity. The powder injected in the low-temperature low-pressure region of the HVOF torch attains very high velocities, but it is at relatively low temperatures.[6] The HVOF powder coating is formed due to the high impact velocity of the powder particles, which leads to impact fusion of the hot/molten powder particles.<sup>[1,6]</sup>

In the HVCW spray process, the wire droplet must be completely molten when it is being atomized.[2] Moreover, the wire droplet is being melted and atomized in the nozzle aircap region, which is a high-pressure high-temperature region.<sup>[1]</sup> This leads to a higher temperature of the atomized droplet. The liquid wire droplet does not attain the velocity from the gases until it is atomized and entrained in the high-velocity gases.[7] It is also possible that the metal is melted and atomized directly from the wire; in this case also, the atomized droplets will attain the velocity after they are entrained in the high-velocity gases.<sup>[2]</sup> This causes the liquid wire droplet to remain entrained in the hightemperature oxidizing jet of the gun flame for substantially longer duration as compared to the powder HVOF systems.<sup>[1–3]</sup>

As a result of the above discussion, it is apparent that

- powder particles in the powder HVOF systems have lower dwell time in the flame due to higher particle velocity, and
- temperatures at the point of wire melting and atomization are high in the HVCW spraying system.

Due to the higher temperature during melting and atomization, it is expected that, in the HVCW spray, reactions take place prior to impact upon the substrate and at the site of wire droplet melting.[3,5] The wire droplet melting environment is one of turbulent mixing of the liquid droplet, the combustion gases, and their reaction products.[3,5,7] The molten droplet, when it is formed, diffuses the gases of combustion.<sup>[3,5]</sup> The rate of diffusion at or near the melting temperature is quite high. The oxidizing conditions that prevail during wire melting and

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atomization oxidize the wire droplets.[3,5,7] Initially, the surface of the droplet is oxidized; thereafter, further oxidation of the molten droplet depends upon the diffusion of oxygen through the oxide layer to the metallic surface.<sup>[3,5]</sup>

The high-velocity gas flow is turbulent in nature, and it imparts turbulence to the liquid metal droplet. The oxides and metallic phases intermingle due to this turbulence.[3] The intermingling of the oxides and the metal in the liquid droplet creates regions of low surface energy within the droplet (where oxides are present).[5] These areas of low surface energy create further sites for atomization of the droplet.<sup>[3,5]</sup> The choice of fuel gas also affects the oxidation in the coating during HVOF spraying. Coatings sprayed using acetylene as fuel have lower oxygen content as compared to coatings sprayed using propane as fuel.[4]

Usually, the density of the oxide layer is less than that of the metal; hence, it tends to get blown away to that side of the droplet that is facing the substrate.[3] The relative movement of the oxide and metallic phase occurs because the oxide and metal phase have different densities.[5] Whether the droplet actually gets blown away will depend upon the surface tension between the metallic droplet and the oxide layer.[4,5] If the oxide layer can overcome the surface tension force of the metallic droplet, then the oxide layer forms a discrete splat in the microstructure; otherwise, it will be seen on the bottom of the splat. This phenomenon can occur in two ways:

- the velocity imparted by the gases is sufficiently high to overcome the surface tension forces between the metallic droplet and the oxide layer, and
- the oxide layer becomes progressively thicker and a stage reaches where the depleted metallic droplet does not have the surface energy in it to hold the enlarged oxide layer.

This process is repeated until the droplet is deposited on the substrate. Most metallic oxides have a higher melting point than their corresponding metals; hence, the oxide layer would have solidified immediately on formation. Therefore, further oxidation of metal due to diffusion from the oxide layer is minimum.[5] Molybdenum oxides melt at a lower temperature; hence, they are essentially molten when they strike the substrate.<sup>[14]</sup> The solidified oxide layer becomes part of a composite coating, wherein the oxide particles are evenly distributed in a metallic matrix.[3,5]

The presence of this oxide layer in the molten droplet or splat may also lead to porosity in the coating microstructure, as the oxide layer, which is already solid when it hits the substrate, may

#### **Table 3 Molybdenum spray parameters**







The formula used for calculating the coefficient of friction ( $\mu$ ) is  $\mu =$  fictional force/normal load

rebound from the substrate surface, thus leaving a gap between the already deposited splat and the incoming splat.[5] Moreover, the oxide layer thus deposited may come out during subsequent processing of the coating because it lacks sufficient cohesion with the matrix.

# **3. Experimental Procedure**

The wire HVOF gun used for the experiments in this case was developed by the R&D unit of the Metallizing Equipment Company (Jodhpur, India). It is commercially know as HIJET™- 9600. Molybdenum wire 3.17 mm in diameter and 99.9% pure was used for preparing the coating samples. The HVCW system works on oxygen-propane/liquefied petroleum gas (LPG) flame. The present experiments were carried out using an oxygen-LPG flame (propane is not available in India). The composition of the LPG gas is given in Table 1.

The parameters of spraying by HVCW are shown in Table 2.

The thickness of the coating for microstructure evaluation and wear testing was  $250 \mu m$ . The samples were prepared by abrasive grit blasting using the suction-type blasting machine; 20 mesh aluminum oxide was used as the blasting media. The samples were subsequently cleaned in the ultrasonic cleaner.

The samples were prepared for (1) metallographic evaluation of molybdenum coatings, (2) wear friction testing of the coatings, and (3) tensile bond strengthening of the coatings (per the ASTM C633-79 method). The parameters of spraying were varied, as shown in Table 3.

The wear testing measurement of the coefficient of friction for the molybdenum coatings was done by the pin-on-disc wear test apparatus. The test was carried out per ASTM G99. The test conditions were maintained as shown in Table 4.

Apart from the above, trials were also conducted for the molybdenum coating using a modified air cap for the HVCW torch. In this aircap, the distance between the nozzle and aircap in the torch was increased (*i.e.,* the length of the air cap was increased). Air plasma spraying of the following powder blend was also carried out:  $Mo 75% + 25% NiCrBSi$ ; the composition of NiCrBSi powder that was used is given in Table 5.

Plasma spraying was carried using the SG-100 Gun of Miller



**(a)**



100 µm 15kV X200 00136 **(c)**

**Fig. 1** (**a**) Photomicrograph of HVCW-sprayed molybdenum coating—round 1. (**b**) Photomicrograph of HVCW-sprayed molybdenum coating—round 2. (**c**) Photomicrograph of HVCW-sprayed molybdenum coating—round 3



**Fig. 2** XRD of HVCW wire-sprayed molybdenum

**Table 5 Composition of NiCrBSi powder (all compositions are in weight percent)**

Ni	Сr		Si	
83.4	9.0	30	3.0	

**Table 6 Physical properties of the molybdenum coating by HVCW gun**



The values in parentheses are the number of tests done

Thermal (now Praxair); the air plasma spraying system was operated at a 18 kW power level.

## **4. Results and Discussion**

The coatings were evaluated for microhardness, tensile bond strength, and surface roughness. The results are given in Table 6. The bond failure for all the coatings was by cohesive failure. The values reported are the average of the readings taken.

The SEM photomicrographs of the molybdenum coating for all three rounds are shown in Fig. 1(a) to (c), and the XRD curve for round 1 is shown in Fig. 2. The photomicrographs show that oxides are present at the splat boundaries as well as in the form of small spheres. The round 2 coating shows higher oxidation as compared to round 1, while the oxidation in round 3 coatings is intermediate between rounds 1 and 2. The round 3 coating shows some segregation of the oxide metal phases; this may be due to less torch and to substrate distance, which restricts oxidation of some of the droplets. Some small size porosity is also seen in the coating. The larger size pores, particularly in the round 1 coating, are pullouts during polishing of the coated samples. It seems that some particles are solidified before they strike the substrate





**Fig. 3** HVCW molybdenum coating wear trend for rounds 1, 2, and 3

**Fig. 4** Frictional force trend of HVCW coatings over a period of time **Table 7 HVCW molybdenum coating wear data (weight loss in grams)**

<b>Sample Round</b>	1	2	3	<b>Mild steel</b>
1	$3.00e-4$	0.01	$1.20e-3$	0.04
	$(m1-i)$	$(m2-i)$	$(m3-i)$	
2	$3.50e-3$	$2.00e-4$	$3.00e-4$	.
	$(m1-ii)$	$(m2-ii)$	$(m3-i)$	
3	$1.00e-4$	$9.00e-4$	$5.00e-4$	.
	$(m1-iii)$	$(m2-iii)$	$(m3-iii)$	
4	0.01	$4.00e-4$	$6.00e-4$	.
	$(m1-iv)$	$(m2-iv)$	$(m3-iv)$	
The values in parentheses are the specimen numbers				

leading to poor cohesion of these particles in the coating. This indicates insufficient heating of the particles in round 1. Molybdenum metal oxidizes catastrophically above 400 °C. The temperature in the spray plume of the HVCW torch is much higher than 400°C; hence, it is quite possible that mechanisms such as the shear of the liquid droplet due to intermingling of oxide breakage of the oxide from the liquid metal droplet also occur, but, due to catastrophic oxidation of the liquid droplets, the metal surface reoxidizes, and when it strikes the substrate, an oxide layer is present on the droplet surface due to subsequent oxidation.

The lower values of surface roughness of the HVCW coatings indicate a more pronounced atomization in the droplets of HVCW spray as compared to the oxy-acetylene flame-sprayed molybdenum. This supports the hypothesis given previously, that atomization in HVCW spraying of molybdenum is more pronouncedly affected by the oxidation of the droplet as compared to other factors. Further studies are required to study the atomization behavior of different materials essentially due to differences in densities of metal oxides, oxidation behavior, and surface tension.

The wear test of the coatings was carried out and the coefficient of friction of the coatings was determined (Fig. 3 and Table 7, and Fig. 4 and Table 8). Four samples for each round were prepared.

The lowest wear was observed for the round 3 coating. This corresponds well with the low values of the coefficient of friction observed for the round 3 coatings. In fact, for the round 3 coatings, the average value of the coefficient of friction is 0.35



and some values are as low as 0.25. If the initial values of friction load are discarded as running in period, then the average value of  $\mu$  will be much below the value of 0.35 and closer to the value of 0.3. These values of  $\mu$  are comparable to the values obtained by the powder HVOF system<sup>[12]</sup> and lower than the values of APS molybdenum cited in Ref 13. The reason for such a low value of  $\mu$  may be the higher cohesive strength of the coating, as seen in Table 6, coupled with the higher oxygen content of the coating.

Table 6 shows that the highest adhesive bond strength of the coating is obtained for round 1; this is expected because the WDX shows the lowest oxygen content for these coatings. The lower oxide content in the coating improves the cohesion in the coating splats. The poor cohesion of the oxide particles with the surrounding metallic/oxide particles is the primary cause of bond failure of molybdenum coatings.<sup>[10]</sup>

The coatings for all three rounds show higher values of microhardness than the oxy-acetylene flame-sprayed molybdenum coating, the value of which is given for comparison. The higher hardness of the HVCW-sprayed molybdenum over the oxyacetylene wire flame-sprayed molybdenum can be explained by the higher oxide content and their fine atomization distribution in the coating.[8,15]

The low values of the as-sprayed surface roughness of the HVCW coatings are also due to high pressure in the nozzle, which results in a higher temperature in the nozzle and, consequently, a higher temperature of the wire and the wire droplet. The high temperature of the molten droplet causes a fall in the viscosity of the droplet, which results in greater atomization of the droplet. The high pressure and velocity of the gases is also responsible for the finer atomization of the droplet.

Apart from the greater atomization of the liquid droplet, another factor for the low values of roughness could be the greater amount of oxidation of the molybdenum particles, leading to more fragmentation of the liquid droplet, which causes very fine atomization of the liquid droplet, and thus resulting in lower values of roughness. The higher values of roughness observed for the coatings in round 3 may be due to the fact that sufficient time is not available for the fragmentation of the oxide particles at such a low gun-to-substrate distance.

#### **Table 8 Coefficient of friction**



#### **Table 9 WDX results**



The microhardness of the coating follows the trend expected, *i.e.,* at a lower wire feed rate, the hardness of coating is lower, except for round 3, where the microhardness value is less than that for round 1. This seems to indicate that there are factors other than fine atomization and oxidation of the coating that contribute to the microhardness of molybdenum coatings. This may be due to the stress relief in the coating due to the heat from the flame at such a small gun-to-substrate distance. Another reason may be the higher particle velocity prevalent at such a low distance, which leads to the formation of coatings with low tensile stress.<sup>[9]</sup>

The microstructures of the coating for all three rounds show splat structure and some porosity. Otherwise, the coating structure seems to be homogeneous. The porosity is observed between two splats and some spherical porosity is also seen. The presence of spherical porosity supports the hypothesis of the oxide particle solidifying during flight and then rebounding from

the surface or getting removed subsequently during polishing due to its poor cohesive bond with the surrounding particles. The coating microstructures for rounds 2 and 3 show less intersplat porosity than those for round 1. This shows better coating integrity for rounds 2 and 3 coatings. This may be due to the superheating of the atomized particle during spraying, which is greater for the higher feed rate in rounds 2 and 3. In fact, during spraying, the brightness of the flame for rounds 2 and 3 was more than for round 1, thus indicating an exothermic reaction and higher temperature of the particles.

As can be seen by the results of the WDX in Table 9, the oxygen content of the coating is highest for the coatings prepared in round 2. The round 2 coating parameters had a higher feed rate of 25 cm/min, as compared to round 1, where the feed rate was kept at 11 cm/min. This shows that the higher wire feed rate increases the amount of oxidation of the coating. As the wire feed rate is increased, the wire tip advances more into the flame and a longer length of the wire tip gets heated to red-hot condition before melting and atomizing. The diffusion of oxygen through the red-hot wire tip is quite rapid. The oxidizing condition of the flame causes rapid oxidation of the wire tip and the resulting molten droplet.

The round 3 coating was carried out at a high wire feed rate but less substrate-to-gun distance. The coating here shows an oxygen content much greater than the value of the oxygen content for round 1 but slightly less than the value for round 2. These results indicate that, for the HVCW spraying, the spray gun-tosubstrate distance parameter does not influence the oxidation of the coating species as much as the wire feed rate parameter, and increasing the wire feed rate increases the oxide in the coating. While spraying, it was observed that increasing the wire feed rate increases the red-hot length of wire. The oxidation of the coating species occurs mainly in the red-hot tip and the liquid droplet before it is pulled away from the tip and accelerated toward the substrate. Oxidation of the coating species during flight, *i.e.,* after leaving the wire tip and before deposition on the



**Fig. 5** Weight loss of plasma-sprayed samples in pin-on-disk wear test

substrate, is negligible. This is apparent when the oxygen contents of the coating for rounds 2 and 3 are compared, wherein decreasing the spray distance from 15 to 8 centimeters reduced the oxygen content of the coating only negligibly (wire feed rate was 25 cm/min for both rounds). Figure 2 shows the XRD curve taken for round 1 of molybdenum coating. This shows the various phases that can form in the coating. The oxygen content of the oxy-acetylene flame-sprayed molybdenum is 7 to 9 wt.%, as in Ref 4. This shows that, irrespective of the spraying conditions maintained, the oxygen content of the HVCW coating is much higher than the oxy-acetylene wire flame-sprayed coatings.

Wear testing results are shown in Fig. 3 and Table 7; this shows that coating in round 3 has the lowest wear rate of all three rounds. This indicates that higher particle velocities prevalent at the lower distances lead to a lower tensile stressed coating and, consequently, to higher cohesive adhesive bonding. The improved wear resistance of the coating sprayed at a lower spray distance is the result of the high cohesive bonding of the coating. Studies of particle velocity at different distances from the gun air cap will shed some light on this phenomenon. The results of tests for measuring the coefficient of friction of the coating show that the coating for round 3 has the lowest coefficient of friction.

A modified air cap was prepared for the HIJET Gun, wherein the length of the air cap was increased. The modified air cap was 38 mm long, while the conventional air cap for the HIJET is 33 mm long. The wire feed rate of the gun with modified air cap increased to 75 cm/min from the standard wire feed rate of 25 cm/min. This large increase in the wire consumption results from the increase in the length of the combustion chamber, where the heating and melting of the wire tip take place. This is consistent with the results cited in Ref 1. Although the increase in the size of the combustion chamber did cause some heating of the air cap, it was not high enough to warrant water cooling of the air cap.

The pin-on-disc wear test results obtained for the APS Mo-NiCrBSi coatings are shown in Fig. 5, and the results of the coefficient of friction measurement are shown in Fig. 6. The conditions maintained while conducting the test: are given in Table 10.

Four samples were prepared for the purpose of the wear friction test. These samples are numbered as 173(1), 173(2), 173(3),



Fig. 6 Variation of coefficient of friction with the linear distance traveled by the pin on disk

**Table 10 Pin-on-disk wear test conditions (plasma coatings)**

Test standard	ASTMG99		
Sliding velocity	$6.28 \text{ m/s}$		
Test time	$15 \text{ min}$		
Total linear distance traveled	5.65 K m		
Applied stress	$0.876$ N/mm <sup>2</sup>		
Disc material	EN31, in hardened condition		

and 173(4). The results of all four samples show that the coefficient of friction for the APS coatings is consistently higher than the coefficient of friction of the HVCW-sprayed molybdenum coatings, as seen in Table 8. The weight loss of the plasmacoated specimens in the wear test is higher than the HVCW molybdenum-coated specimens; this can be seen from the results shown in Table 7 and Fig. 5.

## **5. Conclusions**

This paper studies the influence of parameters on the molybdenum coating produced by the HVCW spray system. The HVCW molybdenum coatings were compared with the plasmasprayed molybdenum-NiCrBSi coatings for wear performance.

Conventionally, the wire flame-sprayed molybdenum coating is used for piston ring coating applications. The plasmasprayed Mo-NiCrBSi coatings are applied where higher wear resistance, a low coefficient of friction, and tougher coatings are required. The HVCW molybdenum coatings have a lower wear rate coefficient of friction than the plasma-sprayed Mo-NiCrBSi coatings. The HVCW system coatings give harder and more wear-resistant coatings than the conventional flame-sprayed coatings.

The oxygen content of the coating plays a very important role in determining the properties of the HVCW coatings; however, the test results also show that parameters such as gun-to-substrate distance also affect the coating properties despite having nearly the same oxygen content. This may be due to the stress relieving in the coating occurring at the small gun-to-substrate distance and also due to the higher particle velocities expected. Further studies of these parameters are required.

The wire HVOF spraying differs fundamentally from the powder HVOF spraying with respect to the melting atomization behavior of the sprayed particles. The HVCW spray system is capable of producing coating microstructures, which are nonconventional with properties that are different from the powder HVOF wire flame spray system.

Applications of the HVCW system will have to be developed, considering the coating microstructure and properties attained; *e.g.,* in the case of molybdenum coating, the low coefficient of friction and the high hardness make this coating a good candidate for antiscuff coating on piston rings.

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