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RESEARCH ARTICLE

STUDY OF MICROSTRUCTURE AND PHASE OF PLASMA SPRAYED HIGH MOLYBDENUM COMPOSITE COATING

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ABSTRACT

Automotive sectors now days are focused on improving the engine efficiency. To enhance the tribological properties of the engine, the thermal spray technique was used for depositing the coating on the cast iron substrate. To provide a reliable basis for the practical application in automotive industry, composite powder containing 60% high carbon molybdenum, 20% pure molybdenum, 10% CrC and 10% NiCr deposited on the cast iron substrate by atmospheric plasma spray method. In this paper, to investigate the microstructure and phases analysis of the deposited coating, scanning electron microscope and X-ray diffraction was performed. The results show that the obtained composite coating has a uniform, laminar and dense microstructure with good coating adhesion with the substrate. The thickness of the composite coating on the substrate is about 371µm.

INTRODUCTION

Manufacturers of the internal combustion(IC) engine facing the challenge to reduce fuel consumption and emissions by improving engine efficiency. Only 21.5% of the total fuel energy is available for car's motion (Holmberg *et al*., 2012; Tung and McMillan, 2004; Jost, 1990; Priest and Taylor, 2000; Andersson, 1991 and K and Jadhav, 2013). The major part of the fuel energy is consumed to overcome friction, only 28% (excluding rolling resistance) of the total fuel energy is used to overcome engine friction losses including piston skirt friction, piston rings, and bearings. Because of these losses, there is a requirement of considerable reduction in engine frictional losses. The components of the piston and cylinder system and crankshaft drive are at the centre of these development efforts. The piston ring pack has a significant potential for bringing down friction losses due to its impartially high (24%) share of mechanical frictional losses (Johansson *et al*., 2011; Quillen *et al*., 2006; He *et al*., 2016). When considering measures to optimise, the tribological system of the piston ring and cylinder surface, piston ring coatings play a progressively important role as they can directly influence the wear and friction behaviour and the resulting scuff resistance. Automotive manufacturers have specified hard chromium coating for piston rings for decades because of its wear and corrosion resistance, however hard chromium plating cause effects on human health because of the use Cr6+ ions in the galvanic process (Janka *et al*., 2016; Zhang *et al*., 2015; Ang *et al*., 2015 and Rastegar and Richardson, 1997). The improvements of the thermal spray process allow the chromium coating replacement with a comparable or superior surface and more environment-friendly coatings (Erdemir, 2005 and Hogmark *et al*., 2000). At present plasma, the sprayed coating has shown significant improvements in reducing wear and CoF in piston rings. Plasma spray processes utilise the energy contained in a thermally ionised gas to melt partially and propel fine powder particles on to a surface such that they adhered and agglomerated to produce coatings. Plasma itself consists of gaseous ions, free electrons, and neutral atoms. The functions of a plasma spray torch are to generate and sustain a captive high-temperature region so that powder particles introduced into that region can be heated and accelerated on to a workpiece (Ahn *et al*., 1997 and Gangopadhyay, 2017). In this study, a coating of 60% high iron carbon molybdenum powder blended with 20% pure molybdenum, 10% chromium carbide and 10% Nickel Chromium was sprayed using atmospheric plasma spraying technique on a cast iron substrate. The microstructure and phase of high iron carbon molybdenum based coating were studied in this paper, to provide a reliable basis for the practical application in automotive industry.

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EXPERIMENTAL PROCEDURE

Substrate Preparation: The substrate material is heated until it is in the liquid state. This melted material then poured into a mould containing the cavity having a shape similar to that of required for the experiment. Cope and drag used for the casting respectively. The after being poured, substrate melt is allowed to solidify. This solidified part is known as casting. This casting is then ejected from the mould and sent for further processing, i.e. grinding operation to obtain a clean and smooth surface with minimum possible irregularities. The experience of the designer and material's strength determines the soundness of casting (Blair *et al*., 2005). This entire substrate is prepared using stag casting all in one go to achieve maximum uniform composition. After solidification, the casting was ejected from the mould, by breaking the sand mould, and sand blasting operation performed. The powder of Carbon (C), Silicon (Si), Magnesium (Mg), Phosphorous (P), Sulphur (S), Chromium (Cr) and Copper (Cu) was used for the preparation of charge. The above charge used for Induction Arc Furnace at temperature 1540°C. The composition of the substrate material is shown in Table 1.

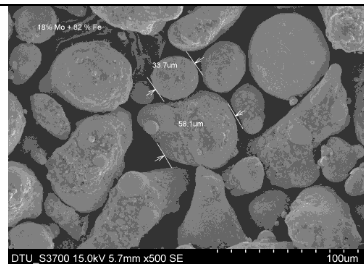
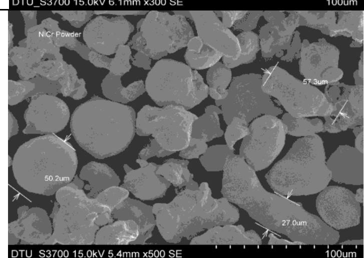
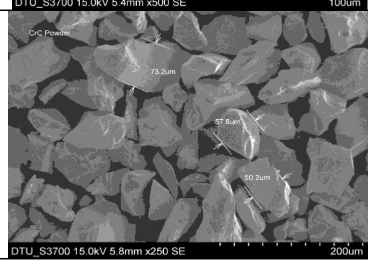
Coating preparation: Atmospheric Plasma Spray Coating

The substrate is made of cast iron similar to that piston ring material, with dimension 90x90x2 mm was plasma sprayed with a Sulzer-Metco PT-F4 torch in an isolated environment using a robot. The robot ensured controlled and reproducible trajectories and speed. Ar-H_2 were used as primary gas and secondary gas respectively. Before spraying, the surface of the substrate was sandblasted with 35-mesh al_2o_3 particles to a roughness value of 10μm to enhance the adherence while coating. During spraying, a cooling system applied that consisted of air jets and venturi nozzles. The dependency of microstructure and the power of the plasma jet (13-19.5 kW) were a primary concern during the process. The substrate prepared through casting, which was further cleaned by sand blasting, was used in plasma spray for coating purpose. The substrate fixed in the fixture of the Plasma Spray Machine. Table 3 shows the operating parameters of coating setup. The coating material used to spray was a composite powder of 60% wt. high iron carbon molybdenum blended with 20% wt. pure molybdenum, 10%wt chromium carbide, 10%wt nickle chromium.

Table 1. Substrate Composition

Element	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Cr (%)	Cu (%)
Target	3.75±0.05	2.70±0.05	0.53±0.01	0.36±0.02	.06±0.005	0.04±0.01	0.035±0.03

Table 2. Powder Morphology

Sr. No	Name	Composition	Density/Uses/ Melting Point	SEM Micrographs
1	Metco (350NS)/ Iron molybdenum composite powder	Fe16Mo2C0.25Mn	2.8 g/cm^3- 3.4 g/cm^3 Wear and scuff resistance 1400 °C	
2	Amdry(313)/ molybdenum powder	99.5% (by wt.) of Molybdenum	2.5 g/cm^3 - 5.9 g/cm^3 Wear and scuff resistance 2620^{0}C	
3	Metco(43) F-NS / nickel chromium alloy powder	Ni20Cr	3.1 g/cm^3 - 4.6 g/cm^3 Corrosion-resistant bond coat 1400 ^{0}C	
4	Metco(70) CNS / chromium carbide powder	Cr_3C_2	2.7 g/cm^3 - 3.0 g/cm^3 Wear resistance 1930 °C	

Whose chemical composition and SEM images are shown in Table 2. The powder particle size varied from 10 – 50 µm.

RESULT AND DISCUSSION

XRD Analysis

X-ray diffraction (XRD) is the most powerful and the least expensive technique for identifying coating crystallographic structures. In the as-deposited condition, the phase analysis of the coating is shown in Fig. 2, the main phase of the coating are Cr_2C_2, $Fe_{0.875}Mo_{0.125}$, $Cr_{0.5}Mo_{0.5}$. It can be inferred that Cr3C2 decomposed during plasma spraying, forming carbides of Cr element. These carbides are the hard phase, having a high hardness and a strong wear- resistance. These hard phases distribute uniformly, disperse in the coating and have a high strength with the matrix, which can heighten the coating's resistance to wear.

Table 3. Process Parameter of Plasma Spray Coating

Sr. No	Process Parameter	Specification
1	Powder Port Internal Diameter	2.2 (mm)
2	Water Flow Rate	4.0 (l/m)
3	Temperature Of Chiller	17 (ºC)
4	Distance Between Spray Gun & Mandrel (At gun angle 300º)	140 (mm)
5	Argon Flow Rate	1.87 (m^3/s)
6	Hydrogen Flow Rate	0.21 (m^3/s)
7	Argon Pressure	6.5 (bar)
8	Hydrogen Pressure	5.5 (bar)
9	Powder Flow Rate	50 (g/min)
10	Voltage	70 (V)
11	Current	460 (A)
12	Gun Feed	10 (mm/min)
13	Gun Angle During Spray	30 (º)
14	Cooling Air Pressure	46 (bar)
15	Powder Driving Temperature	120 (ºC)
16	Powder Mixing time in V-type mixer	5400 (s)

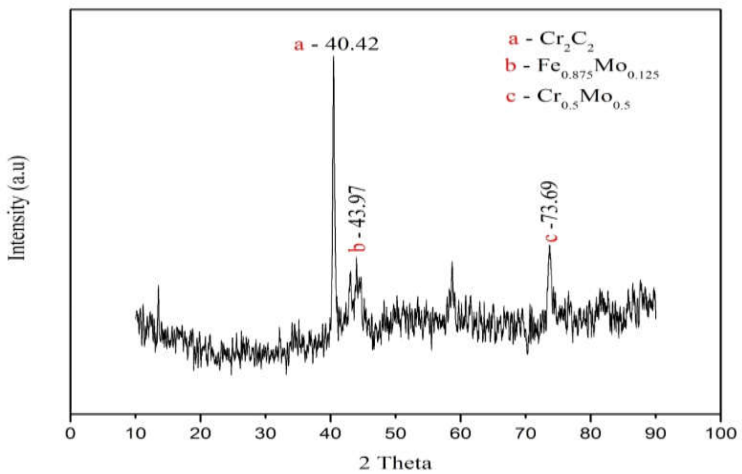


Figure 2. X-ray Diffraction Pattern of Coating

Coating Structure Analysis

Scanning electron micrographs of the thermally sprayed coating under investigation are shown in Fig 3. The sprayed coating progress as the spray gun repeatedly traverses over the surface and applies the coating in layers, with a typical layer thickness 371 µm as shown in Fig 4. Plasma spraying is a process where particles typically 10–100 mm in diameter are rapidly melted and accelerated to produce a stream of molten particles onto a substrate. On impact the liquid droplet flattens to form a disc, the detailed shape of which is determined by surface tension, density, viscosity and velocity of the liquid droplet. In most conditions solidification occurs as flattening of the liquid droplet proceeds, i.e. 'splat' type morphology. Therefore, within a limited size range, the particle morphology in the coating is dependent on the both the degree of superheat in the droplet and the velocity of the droplet as it impacts the substrate surface. The lower the degree of superheat and particle velocity, less flattening of the droplet will occur, i.e. the 'splat' is less elongated in the direction parallel to the surface. Oxides can also be formed during the time between the passes on the outer surface of the layer. This oxidation can be decreased by spraying in a vacuum or inert atmosphere. The microstructure of the coating, observed by the scanning electron microscope is shown in Figure 3. The micrograph of coating surface shows a typical lamellar structure. The interior coating body is composed of numerous flat particles, partially melted Mo, an irregular layer of coating, fully melted, disc splat, partially melted region and unmelted Mo particle. The micrographs of the coating suggest that the splat of the sprayed material does not seem to form a continuous layer in figure 3(e), but at the cross section, it was observed that the coating was more homogeneous and regular in Fig.4. The microstructure seen in the present study is analogous with the findings reported in the studies of various researchers (Vicenzi *et al*., 2008; Lin *et al*., 2015; Pandey *et al*., 2017). Figure 4 shows a photomicrograph of x-section of the sprayed coatings. The coating thickness can be up to several microns shown in

Figure 4(a). Here the typical coating thickness is from 350 to 400μm. Figure 4 (b) recognizes the laminated structure and existing porosity (small black regions). The round black spot (Figure 3b) in micrographs is those that did not melt completely before re-solidifying.

Conclusion

Plasma sprayed composite coating (60% high carbon molybdenum, 20% pure molybdenum, 10% CrC and 10% NiCr) has been deposited on the piston ring is investigated by scanning electron microscope and X-ray diffraction. The following points are concluded from the experimental results are as follows:

- The grain size of the different powder varies from 30 - 70 μm
- The X-ray diffraction results of the coated sample show the sharp peaks of Cr_2C_2, Fe $_{0.875}Mo_{0.125}$ and $Cr_{0.5}Mo_{0.5}$; it can be inferred that Cr_3C_2 decomposed during plasma spraying, forming carbides of Cr element. This is the clear evidence for the formation of the different structure in the coating.
- The microstructure of plasma sprayed coating shows a uniformly dense and laminar structure with an excellent coating adhesion to the substrate. It also shows unmelted Mo, partially melted Mo particle, disc splat, and an irregular layer of the coating investigated by SEM results.
- The observed coating thickness of the coating was found to be about 371 μm, as observed by SEM.

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