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Surface Characterization of Fe Based Alloy Coating on Medium Carbon Steel

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ABSTRACT: This paper discusses about a new amorphous coating of FeCrB (composition of 59%Fe-26%B-15%Cr in wt %) that was done on medium carbon steel substrate (AISI 4340), by means of High Velocity Oxy-Fuel (HVOF) thermal spray. A variety of tests were performed to assess the quality of the coating obtained: The microstructure characteristics of powder and coating were investigated using Scanning Electron microscopy (SEM) coupled with Energy Dispersive Spectroscopy (EDS) and optical microscopy. The initial phase contents of powder and coatings were studied by X- ray diffraction (XRD). The microscopy studies showed that the coatings contained unmelted particles; moreover, oxides and micro-cracks were observed at the surface. The mechanical property of the coatings was characterized using micro hardness test. The test results showed that the coating has increased the hardness three times greater than the substrate. The coated surface showed lower level of porosity. All the above results reveal that the coated region shows better surface properties than the substrate.

KEYWORDS: Fe based amorphous coating, HVOF, Microstructure

I. INTRODUCTION

Iron and steel are the most versatile, least expensive, and most widely used materials for construction of many engineering systems. They are unequaled in the range of mechanical and physical properties with which they can be endowed by alloying and heat treatment. Due to safety, economic, and environmental reasons, significant amounts of effort have been spent to extend the service life of machine components and structures. To meet these demands, various surface modification technologies have become major interest because they can provide superior surface properties. Surface engineering involves the enhancement of certain properties of the surface of a component which are distinct from those of its bulk material. Surface engineering is an economic method for the production of materials, tools and machine parts with required surface properties, such as wear and corrosion resistance [6].

Since many types of attack such as corrosion, friction, wear, heat, radiation and the like occur on the surface of a component, or transferred via the surface into the component, surface protection is of a considerable significance as regards modern materials technology. The purpose of surface technology including thermal spraying is to produce functionally effective surfaces [10]. The wear resistance of materials can be improved by a wide range of coatings [5]. Fe-based amorphous metallic glasses are considered to be extremely viable candidates as surface protective coatings owning to their high crystallization temperature. Fe–Cr–B metamorphic alloys were first invented and commercialized by Scruggs [15]. Using FeCrB alloys in coatings provides two advantages: First, chromium has a very low coefficient of friction, making it an excellent metal for industrial use (requiring materials that are resistant to wear), and second, it has a great ability to protect against corrosion. Other researches concerned thermal spray coatings based on more complex Fe-based alloys [1,17,18], where the addition of elements such as B produces significant hardening on account of various phenomena (formation of interstitial solid solutions, development of small amounts of amorphous phase and/or of hard, fine-grained precipitates, etc.). The coatings of Fe–Cr–B metamorphic alloys have been fabricated by detonation gun thermal spray [7,8,9], high velocity oxygen fuel (HVOF) thermal spray [3,11,13], plasma transferred arc (PTA) weld-surfacing [11,12] and high-energy electron beam irradiation (HEEBI) [13] processes.

For all of these fabrication processes, microstructure analysis showed that the coating consisted of boride particles such as Cr1.65Fe0.35B0.96 and/or Cr2B dispersed in Fe–Cr solid solution matrix [8-13]. These specific alloys sometimes



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exhibit what is called a metamorphic transformation. When subjected to an external deformation such as wear, the solid solution matrix transforms to an amorphous phase, whereas the boride particles remain crystalline [7,8,9]. A number of Fe-based amorphous coatings have been successfully prepared by various methods [8-13], of which the high velocity oxygen fuel (HVOF) is very suitable for preparing Fe-based amorphous coating owing to its relative low processing temperature and high velocity, leading to a good quality of the resultant coatings.

Recently, interest has grown in the use of high-velocity oxygen fuel spraying (HVOF) for the application of surface protective coatings [12, 6]. A key feature of HVOF is that a high-density coating can be obtained since the starting powder collides with the substrate at a supersonic velocity and is severely deformed. Another key feature is that the process temperature is close to the range of the glass transition temperatures of metallic glasses. Around the glass transition temperature, metallic glass transforms into a super cooled state and shows viscous fluidity in spite of temperatures far below its liquids temperature. Therefore, high-quality metallic glass coatings can be achieved using HVOF spraying.

Recently, studies of Fe-based coatings that were applied by the HVOF process have appeared in the literature [7-12]. These reports demonstrated that the Fe-based coating remains amorphous after spraying and shows good adhesion to the substrate. They have examined two commercially available gas atomized Fc-Cr- B based alloy powders with different binder (e.g. Cr, Ni) and B contents namely Armacor M (compositions in wt% Fe-balance, 50%Cr-8.0%B-0.17%C-3.0%Si) and Armacor C (Fe-balance, 32.0%Cr-19.0%Ni-9.0%Co-4.0%Mo-2.4%Cu-4.0%B-0.12%C-1.4%Si in wt.%). It was reported that the Armacor M powder contained boride (Cr1.65Fe0.35B0.96) and ferrite matrix phases whilst the Armacor C powder contained boride (Cr1.65Fe0.35B0.96) and austenite matrix phase. This indicated that powders with different binder contents contain different matrix phase. Alloy powders of Fe63Cr8Mo2B17C5Si1Al4 (in at %,) were sprayed by the high velocity oxy-fuel (HVOF) and plasma spray process [2]. The XRD results of both assprayed HVOF and plasma-sprayed coatings showed significant broad diffusion peaks of primarily amorphous structures. These paper focuses on the micro structural characteristics of the amorphous coatings obtained under different spraying parameters were investigated.

In this paper we have analysed microstructural and mechanical characterization of the coated and uncoated surface. The microstructures of the coatings produced were studied using X-ray diffraction (XRD), optical microscopy and scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS).

II. EXPERIMENTAL PROCEDURE

Materials Selection and Preparation

AISI 4340 steel were used as a substrate material in the present study. The chemical composition of the substrate material is shown in Table 1. Suitable specimens (length 12 mm and diameter 24mm), were fabricated from the substrate bar by EDM wire cutting to a close proximity to the required size, followed by the grinding of both the faces, to remove surface irregularities as well as to deal with the consequences of any induced micro-level changes that might have occurred in the heat affected zone as a result of the EDM cutting.

Powders with nominal composition of Fe59B26Cr15 (in wt. %) were commercially purchased which was prepared by high pressure argon gas atomization method. The as-atomized powders with particle size in the range of $10 - 80\mu$ m were employed for spraying onto AISI 4340 steel substrates by HVOF spraying process. They were sand blasted and cleaned with acetone prior to spraying in order to obtain a roughness favourable for the mechanical bonding of the propelled particles. The coatings with 400 μ m thickness were prepared by JP5000 HVOF spraying system with kerosene as the fuel. The detailed spraying parameters are presented in Table 2. The spray distance between the gun and the substrate was approximately 25 cm because excessive standoff distances produce a more porous and oxidized coating with reduced cohesion and adhesion.



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Table1. Chemical composition of the substrate

S.No	Composition (%)	Gray Cast Iron
1	Fe	bal
2	С	0.38
3	Si	0.28
4	Мо	0.32
5	Cr	1.42
6	Mn	0.47
7	Ni	1.62
8	Р	0.014
9	S	0.008

Table2.	Spraying	parameters	employed	in the	HVOF	process

SI.No	Parameters	Qty
1	Gun type (Super jet Gun)	1 No
2	Pressure of Oxygen	2.5 Kg/cm2
3	Pressure of acetylene	0.6 Kg/cm2
4	Torch angle with respect to substrate	60°
5	Torch Speed	12cm/min
6	Distance of torch tip from the substrate	2.5cm
7	Preheat temperature	200°C

Microstructure Characterization

The microstructure of feedstock powders and as-deposited coatings was characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS). Samples from the surfaces of the coatings and from cross sections of the coatings were polished using a series of coarser to finer grades of silicon carbide emery papers and then finally polished with a diamond paste, applied on a velvet cloth that was attached to a rotating disc. In this way, micropolished surfaces were obtained for further analysis and evaluation. The sample was etched with Nital to reveal microstructure features of interest. X-ray diffraction (XRD) analysis of the specimens was performed on an X-ray diffractometer (Cu Ka radiation) with a range of 2θ diffraction angles from 40 to 50° .

Porosity Analysis

A percentage of the porosity in the coatings was evaluated using Stereographic image analysis system by analysing and averaging various optical micrographs taken at different areas of the coating, as per standard test method ASTM B276. The micrographs of stochastic cross sections of the coatings were taken to calculate the planar porosity by differentiating the contrast between lamellae and pores, and subsequently the average porosity of the whole coatings was calculated as an approximately statistic result.

Microhardness

Vickers microhardness measurements were conducted on the cross section of the HVOF coating surface in Wolpert Wilson equipment, with an applied load of 0.1N (100 g) and indentation time of 12 s, and calculated as an average of 5 measurements.



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III. RESULTS AND DISCUSSION

Micro-Structural properties of the powder

Figure 1 presents the SEM image of the FeCrB feedstock powders. It can be seen that the majority of the particles prepared by gas atomization in the argon atmosphere are near-spherical although some have small satellites attached as seen in the secondary electron image. The satellite particles were typically $5-10 \mu m$ in size whereas spherical particles size ranging from 20-80 μm as show in figure 2.



Figure 1 A Typical SEM Image Of Fecrb Alloy Powders (500x)



Figure 2 A Typical SEM Image Of Fecrb Alloy Powders (2000x)

In figure 3(a), EDS pattern of powder shows that there is no contamination with impurities presents in the powders other than Iron, Chromium and Boron. In figure 3(b), EDS results shows that the weight percentage of various elements of powders of Iron, Chromium and Boron as 52.32%, 15.11% and 25.53% respectively.



Figure 3(a) Backscattered electron image of FeCrB powder

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Figure 3(b) Weight percentage of various elements in FeCrB powder

Micro-Structural properties of the coating

SEM micrographs of the coating surface showed open pores, unmelted particles and micro-cracks. The BSE image of coating surface (see figure 4(b)) shows that the coating thickness is 400μ m. The presence of unmelted particles in the spray will induce some porosity in the coating, and this porosity justifies the use of very fine powders, which increases the number of melted particles and decreases the porosity.

Figure 4 Cross-sectional SEM images of a FeCrB coated specimen.

The coatings appear to consist of multiple particle layers (see Figure 4(a)), which result from successive impacts of unmelted, semi-molten and molten particles (molten particles being required for the formation of homogeneous coatings).

Microhardness

Vickers microhardness values of FeCrB coating and substrate are shown in Figure 6. Microhardness values that were taken along the cross-section of the sample were decreased regularly. At the same time, the average value of the microhardness of FeCrB top coatings is significantly higher than that of the substrate. However, Cr played a major role in the difference in hardness of FeCrB top coating due to its moderate strengthening effect [16]. In addition to Cr content, some inhomogenieties such as porosity, oxides and unmelted particles cause a change in hardness values of the top coating. As indicated in a research [4], concerning flame sprayed coatings, local variation in hardness results from the variation in particle temperatures and velocity which are inherent in the spray process.

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Distance (µm)

Figure 6 Microhardness variation

In the figure 6, the Microhardness variation graph shows that average hardness at the surface of the coating reaches maximum as 960 HV0.1, about three times greater than the substrate hardness which is around 310-330 HV0.1 and the hardness value at the interface of coating and substrate shows 780 HV0.1.

X-ray diffraction (XRD) of the milled powder and the coating

Figure 8 shows the XRD pattern of the coating. Some broad diffraction peaks appearing at $2\theta = 45^{\circ}$ and 52.5° indicate the presence of an amorphous phase. However, the sharp peaks due to crystalline phases are also observed. The major crystalline phases are compounds CrB, Cr2B, Fe3B, FeB and solidification α -Fe (Cr). And the peaks of CrB and Cr2B are less sharp, which implies the increasing of the formation ability of the amorphous phase. These results are similar to those obtained by the rapid quenching of FeB mechanically produced alloys. The diffraction pattern confirms the BCC structure of the α -Fe matrix.

Figure 7 XRD pattern of the received FeCrB powder

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Figure 8 XRD pattern of the FeCrB powder coating

Estimation of the porosity level

Most of the metallographic coating showed a porosity level in the range of 3–4%, which was much lower than that of usual flame spray coatings (in which the typical porosity is between 10% and 20%).

Figure 9 (a) Optical microscopy view and (b) Image Analyser software view

IV. CONCLUSION

Gas atomized powders with nominal composition of Fe59B26Cr15 (in wt. %) were coated on medium carbon steel. The microscopic studies showed that the coatings contained unmelted particles; moreover, oxides and micro-cracks were also observed during testing time on the surface.

Optical microscopy with image analyser software results showed that the coating was achieved with low level of porosity as 3-4%. The Micro Vickers Hardness test result showed that coating increased the hardness three times greater the substrate hardness.

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