Characterization of WC-10Ni HVOF Coating for Carbon Steel Blade

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Abstract. High Velocity Oxy-Fuel, HVOF is a depositing methods of a material layer over a base metal or substrate with characteristics of high flame velocity and moderate temperature. Where, tungsten carbide, WC cermet HVOF coatings is widely used to protect machine components from wear and corrosion. The main purpose of this present paper is to characterize the WC-10Ni coating deposited by HVOF thermal spray onto a carbon steel blade. The morphology and chemical composition of the coating were characterized by Scanning Electron Microstructure (SEM), electron dispersive spectrometer (EDS), and X-ray diffraction (XRD). The hardness test was carried out by using Vickers micro-hardness tester with loads of 490.3 mN (HV0.05). From XRD results, no sharp nickel peak was identified and has been replaced by a hump which indicate the amorphous Ni. The major crystalline phases were compounds WC, W2C and metallic phase of W. The WC-10Ni coating shows high hardness with low porosity distribution.

1. Introduction

HVOF is one of the thermal spraying technique for WC feedstock powder in order to obtain good bond strength and higher density. It is widely used to deposit carbide-based cements, metals and alloys coatings such as WC-Co, WC-Ni and stainless steel material [1].

HVOF has special features of higher particle velocity and lower particle temperature compared to other spray processes. According to Oksa et al. [2], this combination leads to adherent coatings with lower porosity, less extent decarburization and higher wear resistance. A large number of researchers have reported that decarburization of WC has a detrimental effect on the abrasive wear resistance of coating. This is due to the increasing brittleness and decrease in hard particle content. Thus, the drawback of decarburization can be avoided by the cold spray technique, a recently emerging technique operating at temperatures much lower than the melting point of spray materials [3].

WC has been proven their superiority in great number of other tooling and engineering applications due to their unique properties. Over the years, many studies showed the fabricated WC-based coatings exhibited higher hardness, fracture toughness and correspondingly improve wear resistance compared to the other carbide [4]. According to Van Acker [5], WC were reinforced by the binders such as Cobalt (Co) and Nickel (Ni) in order to increase wear and corrosion resistance. For cemented WC, the properties of the components are superimposed where the carbide phase WC provides hardness and wear resistance. While, the ductile binder contributes to toughness and strength [6]. However, the microstructure of WC cracks, large pores and poor bonding occurred as the percentage of binder’s content increase [5-7]. From the previous researches, due to the large pores, the coating facing the abrasion and erosion behaviour, where it leads to reduction of wear resistance.
Co has been the dominating in metal-ceramic composites known as hard metals since the emergence of the first WC-Co cemented carbide in 1923 [8]. This is due to the chemical bonding between WC and Co resulting in a very low interfacial energy, nearly perfect wetting and a very good adhesion in solid state [9]. However, the high price Co metal together with the need for improving the performance of cemented carbides under severe working condition such as corrosion, alternative binder’s Ni have been promoted [10]. Ni is a metal that has similarities as Co in structure and properties, besides it is a good corrosion resistance.

In this study, the Blade was used to stir the mixture of sulphuric acid (H$_2$SO$_4$) and ilmenite in order to maximizing Titanium Dioxide (TiO$_2$) pigments. This blade exposed to deterioration, wear and corrosion due to the corrosive (acid) and erosive (ilminite) properties of the mixture. Thus, as modern method is quite effective in improving the wear and corrosion resistance, one of the option is thermal spraying process (HVOF) with WC-based materials. As many coating enhancements have been reported by researchers, more understanding on the characteristics of the HVOF thermal spraying is needed, so that this technique could be commercialize for various industrial applications in the next future. In this paper, the characterization of WC-10Ni HVOF coated on the carbon steel blade was analysed.

2. Methodology

2.1. Coating Deposition

The powder employed in the present work are known as WOKA 3302. This powder is mainly composed of 90% WC and 10% Ni, where it prepared by agglomeration and sintering. Table 1 summarizes the deposition condition, gun characteristic and particle size for the powder, which correspond to those typically applied industrially. The substrate (blade) that used in this study was a mild carbon steel (Cast steel BS 3100 GR A3) supplied by vendor (Huntsman Tioxide). The chemical composition of both substrate and powder materials shown in Table 2.

Before the deposition of the coating, the substrate surface was thoroughly cleaned by acetone in order to remove mill and rust scale, dirt and foreign materials. Grit blasting was carried out by using an Empire 350 abrasive equipment at a pressure of 2 kg/cm$^2$ with alumina grit. The purpose of this process is to ensure the spray powder (coating) strongly bond to the substrate. The WC-10Ni coating subsequently deposited had average thicknesses in the range of 200 µm to 300 µm.

| Table 1. Deposition parameters supplied by the industrial coating facility |
|---------------------------------|-------------------------------|
| Thermal spray gun               | The Diamond Jet (SULZER METCO) |
| Powder feeding rate (SCEH)      | 28.5                          |
| Particles size                  | -45+15 µm                     |
| Oxygen Pressure (psig)          | 150                           |
| Fuel Pressure (psig)            | 110                           |
| Air Pressure (psig)             | 100                           |
| Air flow (SCEH)                 | 893                           |
| Fuel flux (SCEH)                | 432                           |
| Oxygen flux (SCEH)              | 635                           |
| Spraying distance (in.)         | 9                             |
| Spray Rate (lbs/h)              | 5                             |
Table 2. Chemical composition of powder (WC-10Ni) and substrate (medium carbon steel)

|         | Wt. [%] | W   | C   | Ni   | Fe  | Si  | Mn  | P   | S   |
|---------|---------|-----|-----|------|-----|-----|-----|-----|-----|
| Powder WC-10Ni | Bal     | 5.2-6.0 | 8.5-11.5 | <0.2 | -   | -   | -   | -   | -   |
| Carbon Steel (BS 3100) | -     | 0.45 | -   | -    | 0.06 | 1.0 | 0.05 | 0.06 | -   |

2.2. Microstructure Characterization
Samples were grind by using sand paper starting from grit 360, 400, 600, 800, 1000, 1200 and 1500. Metcon polisher was used to polish the samples with additional of alumina 0.05 µm. The morphological characterization of the coatings was carried out by means of optical microscope (OM) and scanning electron microscope (SEM, Jeol 2000) with energy dispersive spectroscopy (EDS). The coatings analysis was conducted both on the deposition surface (S) and cross sectional area (CS). The apparent porosity of the coating was determined by using SEM. The porosity values correspond to the analysis of a mean of 20 images taken at a magnification of 200x. X-Ray Diffraction (XRD) with Cu Ka (λ = 0.154 nm) radiation were used to identified the phase present in the coating. All the patterns were determined in the interval 2θ < 2θ < 80° at a scanning step of 2°/ min and 0.02° with time step of 0.3s.

2.3. Mechanical Characterization
The coating hardness was evaluated by means of the Vickers Micro-Hardness. The measurements were carried out under indentation loads of 490.3 mN (0.05HV) within 10 s with flat shape of test piece for 10 times and the average of the readings were calculated. From the other researches, at least 5 indentions were conducted at each applied load, both on the coating surface and cross section. The test was carried out randomly in zones apparently homogeneous, free of pores and other discontinuities, as far as the observation under the optical microscope is concerned. All the test was done using Micrometer (USA) micro-hardness measurement set up.

3. Result and Discussion
3.1. Microstructure Analysis of Powder and Coating.
The SEM micrographs (Fig. 1) shows the characteristic of an agglomerated and sintered powder. The particles size of the powder was from the range of 11 µm to 50 µm. The powder particles consist of W, C and Ni was proven by EDS in Fig 2. While, Fig.3 shows the existence of W, C, and Ni in the coating of WC-10Ni.

Fig.4 illustrate the Backscattered electron (BSE) microstructure with high magnification of the WC-10Ni coating layers. It can be seen that the coating consists of angular carbide which dissolve into the metallic binder of Ni. Furthermore, WC-10Ni shows a greater amount of rounded carbide particle which means the carbide grains of the WC is preferentially melted into the Ni phases in high temperature during thermal spray process [11].

The cross-sectional micrograph revealed a good adhesion between dense coating layer and substrate as shown in Fig 5. The high velocity impact of coating materials to the substrate accompanies some severe plastic deformation and rapid solidification. Thus, spray coating layers are likely to possess inevitable pores and cracks [12]. The thickness of the coating was within the range 130µm to 150µm.
**Figure 1a.** SEM micrographs of experimental WC-10Ni powder particle

**Figure 1b.** Particle size of WC-10Ni powder

**Figure 2.** EDS analysis of WC-10Ni powder

**Figure 3.** EDS analysis of coating

**Figure 4.** BSE micrograph of coating

**Figure 5.** Cross section micrograph of coating
3.2. XRD characterization of powders and coatings.

Fig. 6 illustrate the results of the X-ray analysis conducted on the WC-10%Ni powder and corresponding HVOF sprayed coatings. The peaks of HVOF coatings are broader than those of the corresponding spray powders. Broader peaks are the result of a smaller crystal size and/or microstrains inside crystal which is occurred due to the thermal and kinetic process encountered by powder particles during thermal spraying. Indeed, the particles are heated and accelerated with high velocity, and might impinge on the substrate in unmelted, partially molten or fully molten condition [13].

The structure of the powder showed that only WC and Ni phases existed in the powder. There was no evidence of intermetallic phases based on Ni and WC had been formed. While, the XRD patterns for coatings showed WC, W_2C, W and Ni phases. In coating, there is a larger hump in the intensity of the background of the XRD pattern at 2θ between 40 and 46°. It indicates that some development of amorphous phases has taken place during the thermal spray process. The amorphous region was formed as a consequence of the carbide or tungsten dissolution and/or diffusion into the matrix, which leads to a supersaturation of the matrix [14]. In addition, previous researches also stated that the formation of amorphous phases is due to the combination of extremely high cooling rate of impinging molten droplets and the slow crystallization kinetics of the sprayed coating process [15].

During HVOF thermal spraying, it is possible that the secondary phases of W_2C and W are formed due to the decarburization process at high temperature in the flame. The loss of carbon from WC phase occurred during the coating degradation which leads to formation of metallic W phase [9].

![Figure 6. XRD patterns and the powders and corresponding as-sprayed HVOF coatings.](image)

3.3. Mechanical Properties of Coatings.

The hardness distribution is illustrated in Fig 7. Mechanical analysis show that the hardness of the WC-10Ni coating ranges from 1480 Hv to 1544 Hv indented under load of 490.3 mN (0.05HV). It can be seen that the hardness of substrate is 80% lower than hardness of coating. Where, the hardness of the coating is a result of balance microstructure, phase contribution (amounts of W_2C, amorphous phase) and porosity [16-17]. It is reasonable that the hardness increases with existence of W_2C in the coating due to the phase is harder than WC phase. Additionally, the lower hardness of the coating can be explained by the high porosity content. The porosity in terms of area fraction of pores and hardness of the coating are compared in Table 3.
Table 3. The hardness and the porosity value of the coating

|            | Sample 1 | Sample 2 | Sample 3 |
|------------|----------|----------|----------|
| Hardness (Hv) | 1544     | 1540     | 1480     |
| Porosity (%)  | 1.22     | 1.28     | 1.34     |

4. Conclusion
The blade coating with WC-10Ni has been analyzed in terms of surface morphology, particles in the coating, thickness and hardness of the coating. The conclusion that can be drawn from this study are:

- It has been shown that the carbide grains of the WC are preferentially melted into the Ni phases, where the coating has a greater amount of rounded carbide particles.
- The secondary phases of W$_2$C and W are formed due to the decarburization process at high temperature in the flame during HVOF thermal spraying.
- The hardness of the coating depends on the pores distribution. The hardness increases with decreasing of porosity contents, where WC-10Ni coating has high hardness with low porosity distribution.
- Thus, HVOF WC-10Ni sprayed coating has strong ability to increase the performance of blade due to results obtained in this research.

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