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CHARACTERISTICS OF HVOF WC-Co COATING USED FOR ROCKET NOZZLE APPLICATION

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ABSTRACT

The high velocity oxy-fuel (HVOF) sprayed tungsten carbide – cobalt (WC-Co) coating have shown some attractive behaviors, which makes them applicable for high temperature application, such as for temperature insulator for rocket nozzle. This work studies the effect of surface preparation, in this case the grit blasting process, on the characteristics of the coating. The microstructure of the coating was examined by using optical microscope and scanning electron microscope (SEM) equipped with energy dispersive x-ray spectroscopy (EDXS). The results show that the higher the grit blasting pressure, the rougher the surface of the substrate. The surface roughness and topography affects the bonding strength of the coating, although quantitative results were not obtained in this study. The WC-Co coatings showed high hardness and low porosity. The composition of WC-Co coating varied in different region, but in average was close to the composition of the starting powder, meaning that there was no material loss during spraying. Microanalysis also indicates diffusion of tungsten to the interface between the coating and the substrate that may explain the high bonding strength possessed by the coating. The mentioned characteristics may partly suggest that the HVOF sprayed WC-Co is appropriate to be coated to rocket nozzle. More tests on erosion resistance are suggested.

1. INTRODUCTION

Technology for ballistic rockets has been being developed in Indonesia since early 1960's. The first rocket, named Kartika I, was launched in 1962 with a weight of 220 kg. Since then, various and numbers of ballistic rockets, all with solid propellant, have been launched. However, two main problems remain with the development of rockets, they are: control and weight optimization. For the latter, the ideal weight proportion for a rocket is: 99 % of fuel, 3 % of structure and 6 % of payload¹. In general, the weight proportion of rockets developed in Indonesia is far from the ideal proportion, with a relatively large proportion of structural weight². Nozzle contributes the most to the structural weight of the rockets, therefore, this research focuses on the weight reduction of a nozzle. Most rockets use massive solid carbon as temperature insulator for the nozzle. This research investigates the possibility to replace the massive solid carbon with a thin layer of thermal spray coating in order to reduce the weight of the nozzle. The material used for thermal spray coating is WC-Co powder, which has high hardness, high temperature resistance and high erosion resistance³⁻⁴. The WC-Co powder is applied by using HVOF (High Velocity Oxy-Fuel) process, which is readily available in Indonesia. It is common knowledge today that the HVOF process is capable of producing better carbide-metal cermet coatings with high density, high hardness, superior bond strength than many of the other thermal spray methods⁵⁻⁷. The quality of the HVOF thermal spray coating is dependent mainly on the surface preparation

of the substrate. This paper studies the effect of surface preparation on the characteristics of WC-Co HVOF coating to be applied on rocket nozzles.

2. EXPERIMENTAL PROCEDURES

The substrate for HVOF coating is S45C steel, which is used as the material for rocket nozzle. The composition of the substrate is detailed in Table 1. Cylindrical samples were made in accordance to ASTM C633 with the diameter of 25 mm and the height of 35 mm, with a threaded hole made on one sample surface (see Figure 1). The surfaces of samples were machined and grit blasted prior to HVOF thermal spray, by using 24 mesh - Al₂O₃ grit with air pressure of 1, 3, 4 and 5 bar. Roughness of the samples was then measured by using Surfcom 120A surface roughness tester. Thermal spray process utilized Sulzer Metco HVOF gun using 50 mesh WC-Co powder from Deloro Stellite GmbH, with the composition of 82.71 W, 5.49 C and 11.8 Co (wt. %). The HVOF process used a powder pressure of 45 psi and a temperature of 31.5 °C. Thickness of the coating was measured with a micrometer, which was confirmed by a measurement by using an optical microscope.

Adhesion strength of the coating was tested in accordance to ASTM C633 with Devco Epoxy adhesive in a Shimadzu servopulser machine with 2000 kg maximum load. The adhesive was manually applied and cured at room temperature. Fracture surfaces were visually observed.

Table 1: Composition of S45C steel substrate

Element	wt. %
C	0.423
Mn	0.639
P	0.0045
S	0.01
Cr	0.05
Cu	0.072
Si	0.191
Ni	0.045
Fe	Balance

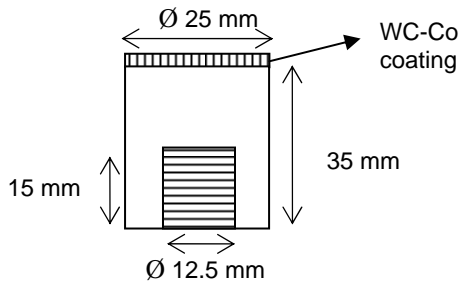


Figure 1: Dimension of samples in accordance to ASTM C633.

Microstructure of the coating was observed under optical microscope and SEM (scanning electron microscope) equipped with EDXS (energy dispersive x-ray spectroscopy). The samples were ground, polished and then etched by an etchant composed of $K_3Fe(CN)_6 : NaOH = 1 : 1$.

3. RESULTS AND DISCUSSION

3.1 Surface Roughness

The effect of grit blasting pressure on the surface roughness of the surface is presented in Figure 2, in which the pressure of the grit blasting process is proportionally related to the roughness of the surface. It can be clearly seen that the surface roughness due to machining process is $4.25 \mu\text{m}$. After grit blasting with the pressure of 1 bar, the surface seems to be smoother, this is due to the fine size of the grit. However, with the increase in the pressure of the grit blasting, the impact energy of the grit blasting media is higher, so that the deformation of the sample surface is more severe.

This means more asperities on the sample surface which directly increase the roughness.

3.2 Coating Thickness

The target thickness of the WC-Co coating was $\sim 400 \mu\text{m}$. However, since the size of the samples is much smaller than the size of the HVOF torch, it was difficult to control the thickness of the coating. As shown in Figure 3, the thickness of the coating varies from $346.6 \mu\text{m}$ to $492.9 \mu\text{m}$, that is with a deviation of

$\sim 18\%$ of the target thickness. Previous results also showed that the deviation in the target thickness of HVOF coating is $15 - 40\%$. In application, the HVOF coating is usually machined to level the surface. However, this was not done since the focus of the research is on the surface preparation of the samples.

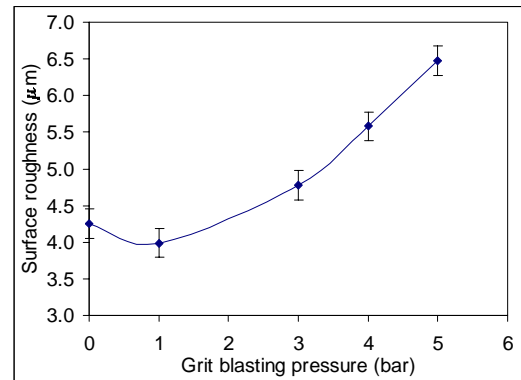


Figure 2: Effects of grit blasting pressure on the surface roughness.

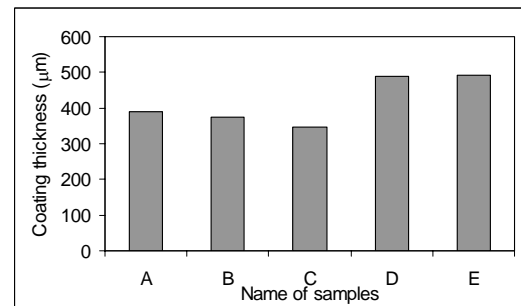


Figure 3: Variety of WC-Co coating thickness on samples with different surface roughness. Sample A: without grit blasting, and other samples with grit blasting with the pressure of: B: 1 bar, C: 3 bar, D: 4 bar and E: 5 bar.

3.3 Adhesion Strength

The coating adherence of WC-Co layer on unprepared surfaces is very low, so that it peeled off easily on adhesion strength testing, even though the surface has a certain level of roughness. This is shown in Figure 4. The peeling of the WC-Co coating is mainly due to the presence of dirt on the surface, such as oil and humidity, which prevents the bonding between the coating and the substrate⁴. This is confirmed by the micrograph in Figure 6 (a), where void between the coating and the substrate is clearly revealed.

On the other hand, samples which were grit blasted showed exceptional high WC-Co bonding strength. The surface roughness produced by grit blasting process assists in improving the coating adherence. The bonding strength of the coating is much above the strength of the epoxy adhesive ($\sim 40 \text{ MPa}$). So, when tested, the test piece failed on the epoxy, not on the coating. Figure 5 presents the fracture surfaces of the grit blasted samples. The dark surface is the fracture

that occurs within the epoxy layer, while the light surface is the fracture that occurs between the epoxy and the WC-Co coating. Because the fracture occurs outside the WC-Co coating, no quantitative results on the adhesion strength were obtained.



Figure 4: Fracture surfaces of unprepared samples after adhesion test. The shiny surfaces of the substrate show complete WC-Co coating peel off.

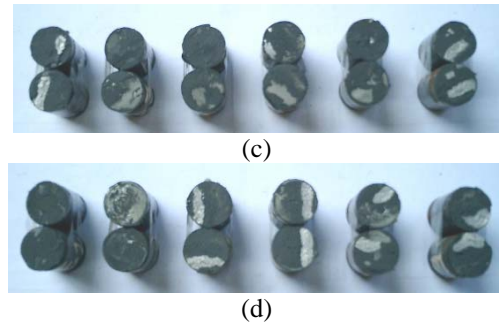
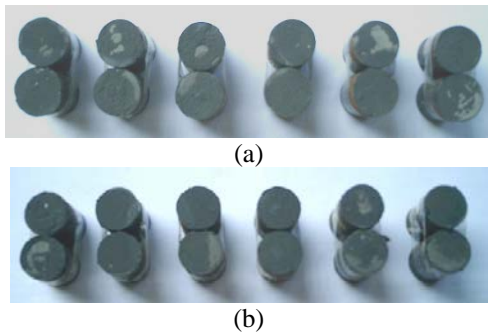


Figure 5: Fracture surfaces of adhesion test samples with grit blasting pressure of (a) 1 bar, (b) 3 bar, (c) 4 bar and (d) 5 bar. The dark surface is the fracture occurs within the epoxy layer, while the light surface is the fracture occurs between the epoxy and the WC-Co coating.

3.4 Microstructure of WC-Co Coating

Microstructures of WC-Co coating applied on different surface roughness are presented in Figure 6.

They show a high density structure of WC-Co coating with minimum porosity. This is one of the advantages of HVOF thermal spray method³⁻⁷.

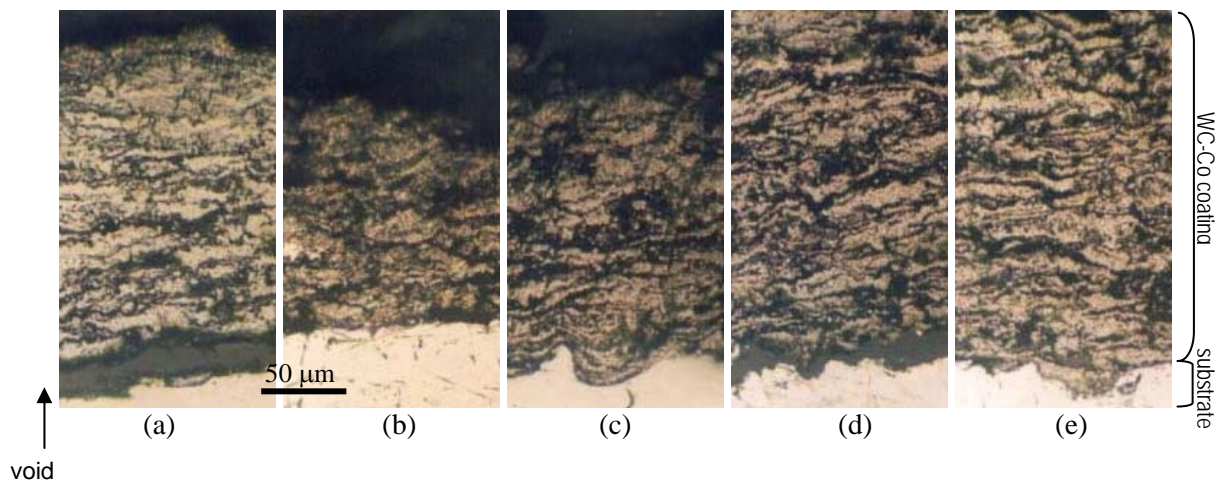


Figure 6: Micrographs of WC-Co coating on samples with grit blasting pressure of (a) 0 (without grit blasting), (b) 1 bar, (c) 3 bar, (d) 4 bar and (e) 5 bar.

Table 2: EDXS analysis results on positions 1 – 6, as shown in Figure 7.

Potition	C (wt. %)	O (wt. %)	Fe (wt. %)	Co (wt. %)	W (wt. %)
1	1.4	8.3	0	16.4	73.3
2	2.7	0.3	0	5.0	92.0
3	1.3	6.9	0	18.5	72.6
4	2.9	0	94.3	0	2.8
5	2.0	0	98.0	0	0
6	1.9	0	98.1	0	0

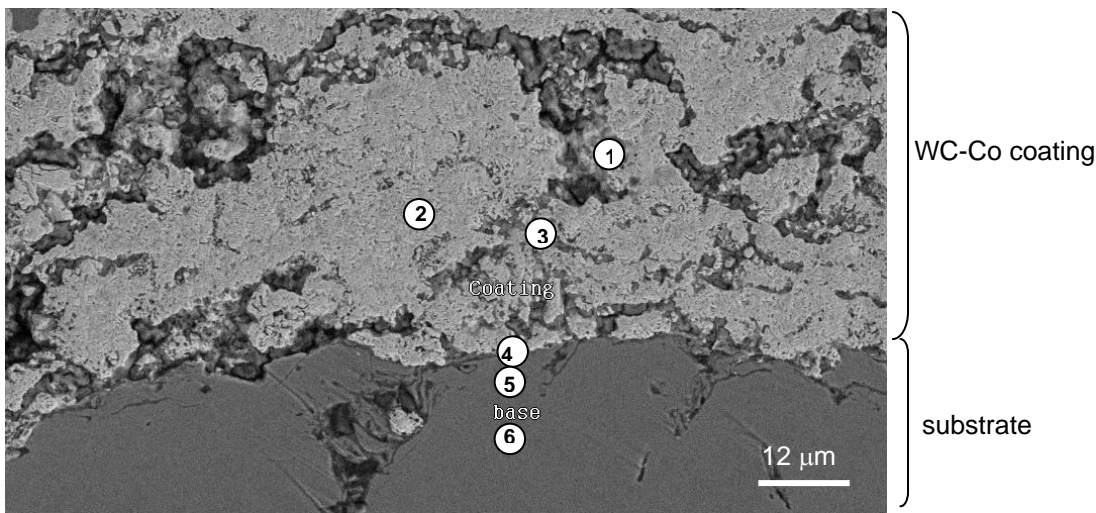


Figure 7: SEM micrograph of the coating - substrate interface region.

The unique wavy lamellae of the thermal spray coating are consistent with the nature of WC-Co droplets deposited on the surface at high pressure. The interlamellae spacing is around $0.01 - 0.1 \mu\text{m}$ with different morphology and this is consistent with previous results⁶⁻⁷. Difference in surface roughness is not directly detected in these micrographs, due to the small size of area which is covered by optical microscope. However, it is clearly seen that WC-Co coating completely cover the surface of grit blasted samples (Figure 6 (b) – (e)), while there is void between the coating and the surface that was not grit blasted (Figure 6 (a)). The mechanical interlocking between the WC-Co coating and the substrate contributes to the high coating adherence.

Detailed observation on the microstructure of the coating was conducted by using SEM and shown in Figure 7. Microanalysis was conducted on positions 1 – 6 as shown by Figure 7 and the results are tabulated in Table 2. The data in Table 2 is not the exact wt. % composition, because EDXS is not accurate in identifying light elements such as carbon and oxygen. However, the data still can be used for comparison.

The SEM micrograph verifies the presence of lamellae structure, although the boundaries between lamellae are also clearly revealed. No porosities were detected, confirming the high density of the WC-Co coating produced by HVOF process.

The composition of the coating varies from area to area (Table 2), but in average is close to the composition of the starting powder, meaning that there was no material loss during spraying. Areas with light colour (position 2) are rich in W and C, while gray areas (positions 1 and 3) tend to be rich in Co. This seems to be due to the melting and atomization of the powder before bombarding the surface. The melting points of WC and Co are significantly different (T_m Cobalt = $1493 \text{ }^\circ\text{C}$, T_m WC = $2780 \text{ }^\circ\text{C}$)⁴, so segregation of elements during the melting and transport of the powder is highly unlikely.

Oxygen was detected within the coating (positions 1 and 3), which may be interpreted to be due to oxidation of the starting particles during their transport from the HVOF gun to the surface of the substrate. The microstructures on the coating – substrate interface indicate superior bonding, in which no porosity or void between the two. It is noteworthy that a small amount of W ($\sim 2.8 \text{ wt. } \%$) was detected in the interface (point 4). This strongly indicates that diffusion of W from the coating into the substrate may occur. The diffusion of W seems to contribute to the extremely high bonding strength of the WC-Co coating. These all may partly suggest that the HVOF WC-Co is appropriate to be applied as rocket nozzle coating. Erosion tests need to be conducted to verify further whether or not the coating can be used as a thermal barrier on rocket nozzles.

4. CONCLUSIONS

1. Without grit blasting process, the coating adherence of WC-Co layer is very low.
2. The higher the grit blasting pressure, the rougher the surface of the substrate. The increase in grit blasting pressure from 1 bar to 5 bar, increases the the surface roughness from $3.99 \mu\text{m}$ to $6.48 \mu\text{m}$.
3. The adhesion strength of the HVOF WC-Co coating is high, that is more than 40 MPa. The high strength seems to be due to the mechanical interlocking between the coating and substrate. Diffusion of W on the coating – substrate interface may also occur.
4. The microstructure of WC-Co HVOF coating is dense with minimum porosity and layered lamellae, confirming the advantage of HVOF thermal spray process.

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