

Enhancement of wear and mechanical properties of thermally sprayed WC-Co coatings by HIPing post-treatment

S TOBE and Y ANDO

Ashikaga Institute of Technology, Tochigiken, Japan

R AHMED and V STOICA

School of Engineering and Physical Sciences, Heriot-Watt University, Edinburgh, UK

ABSTRACT

The aim of this investigation was to ascertain the beneficial improvements in tribo-mechanical properties of post-treated thermal spray WC-Co coatings. Thermally sprayed WC-Co coatings which are deposited by the high velocity oxy-fuel (HVOF) process are well known for their excellent wear resistance, and are widely used as machine parts. However, there is an ever-increasing demand to produce coatings which have even higher wear resistance and mechanical strength. One possible method to satisfy these requirements is the hot isostatic pressing (HIPing) post-treatment of thermally sprayed WC-Co coating substrate system. However, the application of HIPing process to WC-Co coating on steel substrates is not easy because of big differences in thermal expansion coefficient between the two materials. In this study, rather low HIPing temperatures (850 ~ 1000 °C) and high pressure (150 MPa) were employed. As a result, it was possible to get the hardness of the coating equal to hot pressed WC-Co material. Consequently, due to the improved hardness and other microstructural improvements after the HIPing post-treatment, the wear loss of HIPed coatings reduced to almost half of the as-sprayed WC-Co coatings.

1 INTRODUCTION

Thermally sprayed WC-Co coatings have high hardness and excellent wear resistance, especially when deposited by the High Velocity Oxy-Fuel (HVOF) process. For these reasons they are widely used to modify the surfaces of engineering components for improved wear resistance e.g. typical applications include steel and paper manufacturing, automotive, aerospace and marine industries (1), (2), (3).

However, thermally sprayed coatings suffer from low cohesive and adhesive bond strength.

Although the HVOF process has very high flame velocity, and it is possible to get dense coatings, the bonding strength of thermal spray coatings is still lower than that of hot pressed material. One possible method to overcome these coating defects is to apply post-treatments such as the Hot Isostatic Pressing (HIPing) process. However, the thermal expansion coefficient of WC-Co is about half of steel. Due to this reason, lower HIPing temperature and higher HIPing pressure were employed in this study. The aims of this investigation were thus to improve the hardness of post-treated (HIPed) coatings equal to that of hot pressed material, and also to increase its wear resistance. Measurements of through thickness residual stress profile, which is a key factor to comprehend coating's delamination resistance, also formed a part of this investigation. Furthermore, diffusion between the coating and substrate, which was inevitable during HIPing, was also investigated.

2 EXPERIMENTAL PROCEDURES

2.1 Thermal Spraying

The substrate material used in this study was high hardness bearing steel SUJ-2 in JIS (Japanese Industrial Standard). Substrate steel was 10mm square and 5mm in thickness. Blasting was carried out prior to thermal spraying using $\square 36$ alumina shot. An HVOF spraying process was used to deposit WC-12%Co coatings using a JP5000 spraying system. The coating thickness was about 300 μm .

2.2 HIPing Post-Treatment

The HIPing conditions were carefully chosen because of big difference between the thermal expansion coefficient of the coating and substrate materials. Difference of this coefficient can sometimes also result in crack formation and delamination, especially at high temperatures. Hence in this study HIPing temperature was varied from 850 $^{\circ}\text{C}$ to 1000 $^{\circ}\text{C}$ and the pressure was set at 150 MPa. HIPing was mainly performed using capsules. But in one case, no capsulation was employed. The HIPing conditions and the nomenclature used to discuss them are shown in Table 1. These HIPing conditions were selected to provide a broad range of temperature variations, whereas the heating and cooling rates were kept at minimum to avoid microcracking and excessively high residual stresses within the coating material.

Table 1, HIPing conditions.

Sample	HC-1	HC-2	HC-3	HC-4	HC-5	AS
Capsulation	yes	no	yes	yes	yes	As-sprayed (not HIPed)
HIPing temperature ($^{\circ}\text{C}$)	850	850	900	900	1000	
Pressure (MPa)	150	150	150	150	150	
Holding time (min)	60	60	60	120	60	
Heating rate ($^{\circ}\text{C}/\text{h}$)	50	50	50	50	50	
Cooling rate ($^{\circ}\text{C}/\text{h}$)	30	30	30	30	30	

2.3 Microstructural Investigations

The microstructure of coating samples was observed using an optical microscope and Scanning Electron Microscope (SEM). Diffusion at the coating-substrate interface was examined using an Electron Probe Microscopy Analysis (EPMA) and x-ray diffraction processes. Pores in the coating were also carefully examined using microscopy.

2.4 Mechanical Properties

Residual stress measurements were made using x-ray diffraction and modified layer removal method. The coating was electro-polished every 20 μm to depth profile the residual stress

field. The x-ray stress measurement conditions are listed in Table 2. Coating hardness was measured using a Vickers micro-hardness tester.

2.5 Wear Tests

The wear tests were carried out using a block on disc type wear testing machine as shown in Figure 1. The counter material was an electroplated hard chromium coated disc. The contact load applied was 20N under the conditions summarized in Table 3. Total sliding distance for each test was set at 40,000m and intermediate values of coatings mass loss were recorded at distances of 10,000, 20,000 and 30,000 meters.

Table 2, X-ray stress measurement conditions.

Characteristic x-ray	Cr-Kα
Diffraction plane	(1 1 1)
Diffraction angle (degree)	124.4
Filter	V
Counter	PSPC
Tube voltage (kV)	40
Tube current (mA)	200
Collimator diameter (mm)	2

Table 3, Wear test conditions.

Sample size (mm)	10×10×5
Counter material and form	electroplated hard Cr disc
Disc diameter (mm)	200
Rotating speed (rpm)	625
Applied load (N)	20
Wear distance (m)	40000
Dry or wet	dry

3 EXPERIMENTAL TEST RESULTS and DISCUSSION

3.1 Coating Microstructure

Two factors were focused during the microstructural investigations. One was uniformity of coating structure including porosity. The second was diffusion at the coating-substrate interface including Kirkendall void formation. Microstructures of coatings cross-section are shown in Figure 2. Figure 2(a) shows the as-sprayed coating, marked as “AS”, whereas 2(b) to 2(f) show HIPed samples of HC-1 to HC-5, respectively.

It is widely recognised that the coatings deposited by the HVOF process has less than 2% porosity. This is consistent with the findings of few pores in the current investigation as shown in Figure 2(a) and 2(c), i.e. as-sprayed and uncapsulated HIPed samples, respectively. On the other hand, for the samples which were HIPed in capsulated condition, no pores were observed. Figure 2 (f) shows the cross-section of HC-5 sample which was HIPed at the highest temperature of 1000 °C. It can be seen that the coating is completely defect free and of uniform structure. These results thus indicate that whilst it is possible to eliminate pores using capsulated HIPing, it may not be possible to extinguish pores by the uncapsulated HIPing at the lower temperature of 850 °C.

The HIPing post-treatment also changed the configuration of interface between the coating and substrate. In the as-sprayed samples, the boundary is in a zigzag. This was due to the blasting carried out prior to spraying. After HIPing, the boundary is almost like a straight line as shown in Figure 2. The isostatic pressure during HIPing caused the substrate surface to minimize the surface area, thus the surface roughness disappeared at the coating substrate interface. Also from Figure 2 it can be appreciated that in HIPed coatings there is a zone affected by the diffusion near the coating substrate interface. It was also observed that the thickness of this zone increases with the increase in HIPing temperature. In order to understand the process of diffusion EPMA was performed. EPMA results of HC-5 sample that was HIPed at highest temperature of 1000 °C, are shown in Figure 3. It can be seen from Figure 3 that iron (Fe) diffuses in to the coating and Fe content of the surface of substrate decreases. Chromium (Cr) content in the substrate also decreases near the interface, however apparent diffusion of Cr into the coating was not detected. No change was seen on tungsten (W).

This may be due to the fact that tungsten has large and heavy atomic size hindering its diffusion. In the case of carbon (C) in the coating, decreasing of content near the substrate was observed. Therefore, decomposition of WC to W_2C and metallic W can be expected. An x-ray study was carried out to identify the existence of W_2C and W near the interface. But W_2C and W were not detected by the study. There was also significant diffusion of cobalt (Co). A thin Co rich layer was formed during HIPing along the coating substrate interface and Co content of the coating near interface decreased. Diffusion of Co to steel substrate was also observed. Diffusion of these elements also influenced coating's hardness as will be discussed in the next section.

As described above, Fe considerably diffuses into the coating, whereas Co diffuses into the substrate. In this case, generation of Kirkendall pores was expected. A detailed inspection using an SEM was carried out to detect the Kirkendall pores. These pores can be observed in almost all samples except HC-5 which was HIPed at the highest temperature. SEM images of the pores are shown in Figure 4. It is believed that in the case of HC-5 sample, Kirkendall pores were eliminated by the plastic flow of substrate during HIPing at high temperature.

3.2 Microhardness

Hardness values were measured at coatings cross-section at three points, namely near surface, middle of the coating and near the (coating substrate) interface. These hardness values are shown in Figure 5. It can be seen from Figure 5 that the hardness of HIPed coatings was much higher than the as-sprayed coatings. This is thought to be due to the disappearance of pores and strengthening of intersplat interfaces. The maximum hardness of the HIPed coating was over HV 1800 which is approximately the same as hot pressed WC-Co material. The hardness of HIPed samples also showed some dependency upon the HIPing conditions. Although the hardness of samples marked HC-3, 4 and 5 have similar values, HC1 sample, which was HIPed at the lowest temperature had the lowest hardness for encapsulated HIPing conditions considered in this investigation. HC-2 sample, which was HIPed in uncapsulated condition, shows the lowest hardness among HIPed samples, but higher than the as-sprayed sample. Recalling the results of the diffusion zone described in Figure 4, and also considering the hardness values near the interface shown in Figure 5, it can be appreciated that the hardness of the diffusion zone is lower than the hardness at near surface and middle of coating. But these values are still higher than the as-sprayed samples.

3.3 Residual Stress

Residual stress is one of the key factors dictating coatings performance in tribo-mechanical applications. Control of residual stress magnitude and direction is thus critical during thermal spraying. However, in some cases a very high residual stress is generated in the coating and it causes coating delamination and fracture. Hence in this study of HIPed thermal spray coatings the residual stress of surface material was measured. The residual stress distribution through coating thickness of all samples was measured by an x-ray system. Previous studies have shown that the residual stresses within WC-Co coatings deposited on steel substrates are generally compressive (4). Main factors contributing to this compressive residual stress are the differences in the coefficient of thermal expansion of the coating and substrate materials, and also the supersonic velocity of jet stream carrying WC-Co particles during HVOF spraying resulting in shot peening effect.

The residual stress distribution of the as-sprayed and HIPed coatings is shown in Figure 6. In this figure, the vertical axis is the residual stress and horizontal axis is depth from the surface. The residual stress within the samples, which were HIPed at lower temperature of 850 °C, varies randomly with the depth of measurement. On the other hand, in the samples HIPed at higher temperatures, the residual stresses are lower and more uniform. One possible explanation of this phenomenon is that in an x-ray stress measurement process, stress which exists in a particular material is measured. In this study, the stresses which existed in WC particles were measured, and the stress in Co was not measured. The HIPing conditions employed in this study also indicate that

the HIPing temperature is low and the pressure rather high. In these conditions, it is thought that the stresses applied to WC particles during HIPing differed significantly relative to its position with depth. In HC-2 sample that was HIPed in uncapsulation, the scatter of the residual stress is large. Furthermore, Kirkendall pores were formed during low temperature HIPing, this implies that the plastic flow of even the substrate did not occur. Judging from these facts, stresses applied to individual WC particles differed significantly and thus there was a scatter in the residual stress results.

3.4 Wear Performance

Enhancement of wear performance of WC-Co coating was one of the main purposes of this study. The wear resistance of all coatings was tested using a block on disc type wear testing machine (Figure 1). The results of this investigation are summarized in Figure 7. The vertical axis is the mass loss in milligram, and the horizontal axis is the sliding wear distance in meters. These results indicate that WC-Co coatings deposited by the HVOF process have excellent wear performance. The mass loss of the coatings, even the as-sprayed coatings, is very low. It is clear from Figure 7 that the HIPed coatings have even superior wear performance than the as-sprayed ones. The mass loss of sample HC-3, 4 and 5 is about half of the as-sprayed samples. It can also be appreciated from Figure 7 that the mass loss decreases with increasing HIPing temperature. In terms of the influence of capsulation on wear performance, the mass loss of the uncapsulated HIPed sample was a little higher than capsulated samples, which is consistent with the trend seen during the hardness measurements (Figure 6). It however needs to be appreciated that apart from the increased hardness, the much more improved wear performance of HIPed samples was achieved by the densification and homogeneity of coating microstructure, and also higher bonding strength of lamella interfaces.

3.5 Future Projections

In this section, other expected properties of the HIPed WC-Co coatings are described. First one is the corrosion resistance. There is a strong demand to use WC-Co coatings under corrosive environments (5). In these cases, both sliding wear and corrosion resistant properties are required. Many papers have been published on this subject (6), (7). However, the most significant problem of thermal spray coatings is porosity, especially existence of through pores. Therefore, a sealing material must be painted on the surface of coatings. However, the results presented in this paper indicate that it is possible to get defect free coatings by HIPing post-treatment. Due to this reason, very high corrosion resistance is expected.

The next is the adhesive strength of HIPed coatings. As already described earlier, the interface of as-sprayed coatings appears “zigzag” when the cross-section is observed by microscopy. This zigzag line was formed by blasting. The bonding mechanism of the as-sprayed coatings is believed to be “anchor effect”. The bonding mechanism of HIPed sample is thought to be metallurgical, which is formed by diffusion. Undoubtedly the metallurgical bonding will result in much higher adhesive strength than the mechanical interlock caused by the anchor effect. The authors of this paper also experienced this fact through other experiments.

4 CONCLUSIONS

The conclusions obtained from this study can be summarized as follows:

1. HIPed coatings have much higher wear resistance than the as-sprayed coatings. The mass loss of the HIPed coatings was about half of the as-sprayed coatings.
2. HIPing post-treatment can obtain uniform and defect free WC-Co coatings. Moreover, diffusion

takes place near the coating substrate interface, indicating a metallurgical bonding after the HIPing post-treatment.

3. The hardness of HIPed coatings was much higher than the as-sprayed coatings. The hardness values measured during this study were approximately similar to the hot pressed WC-Co material. The hardness of the diffusion zone near the coating substrate interface is lower than the other areas of the coating.
4. Just like as-sprayed WC-Co coatings on steel substrates, HIPed coatings also have compressive residual stresses, but its values vary very much with depth.
5. It can be expected that the HIPed WC-Co coatings will have excellent corrosion resistance and high adhesive strength.

5 REFERENCES

- (1) M. Sawa and J. Oohori, Application of thermal spray technology at steelworks, *Proceedings of International Thermal Spray Conference*, 1995, pp.37-42.
- (2) J. Wigren, L. Pejryd, D. J. Greving, J. R. Shadley and E. F. Rybicki, Performance evaluation and selection of tungsten carbide thermal spray coatings for mid-span dampers, *Proceedings of International Thermal Spray Conference*, 1995, pp.113--118.
- (3) D. J. Varacalle, G. Irons, R. J. Lalumiere, W. D. Swank and J. Lagerquist, *Proceedings of International Thermal Spray Conference*, 1998, pp.347-353.
- (4) L. Pejyrd, J. Wigren, D. J. Greving, R. T. R. McGram, J. R. Shadley and E. F. Rybicki, *Proceedings of International Thermal Spray Conference*, 1996, pp.863-868.
- (5) C. Fiedrich, R. Gadow and D. Scherer, Functional ceramic and Metallurgical coatings on Magnesium components, *Proceedings of International Thermal Spray Conference*, 2001, pp41-47.
- (6) Y. Koga, H. Obata, M. Noguchi, K. Tarumi and K. Ogi, Effect of sealing on electrochemical characteristics of WC-system sprayed coatings, *Journal of Japan Thermal Spray Society*, Vol.39, No.4, pp.154-157, 2002.
- (7) M. Takeda, N. Morihiro, R. Ebara, Y. Harada and M. Kido, *Journal of Japan Thermal Spray Society*, Vol.39, No.1, pp.7-12, 2002.

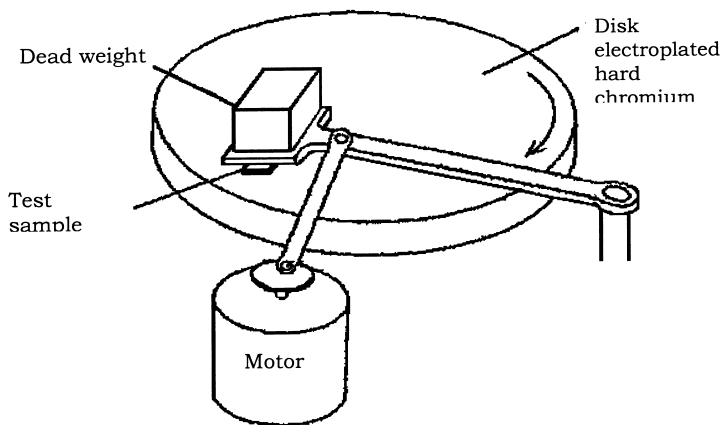


Figure 1, Schematic illustration of wear testing apparatus.

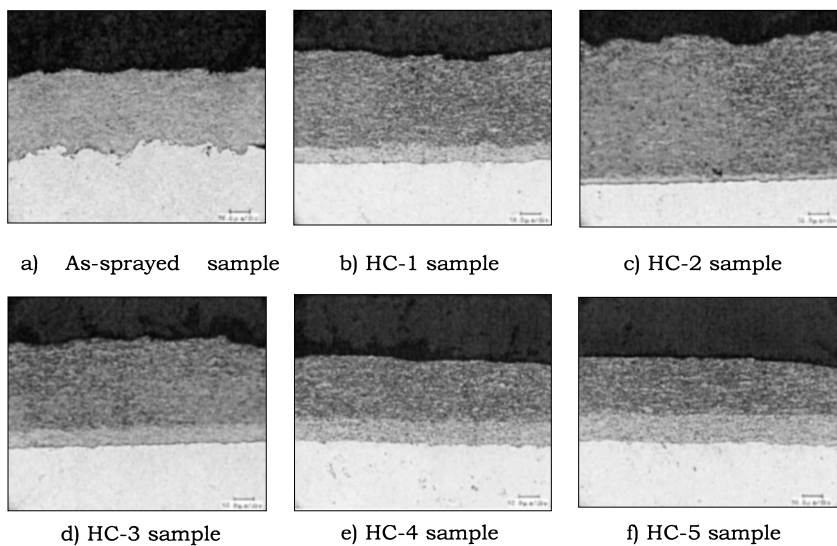


Figure 2, Micrographs of cross-section of as-sprayed and HIPed samples.

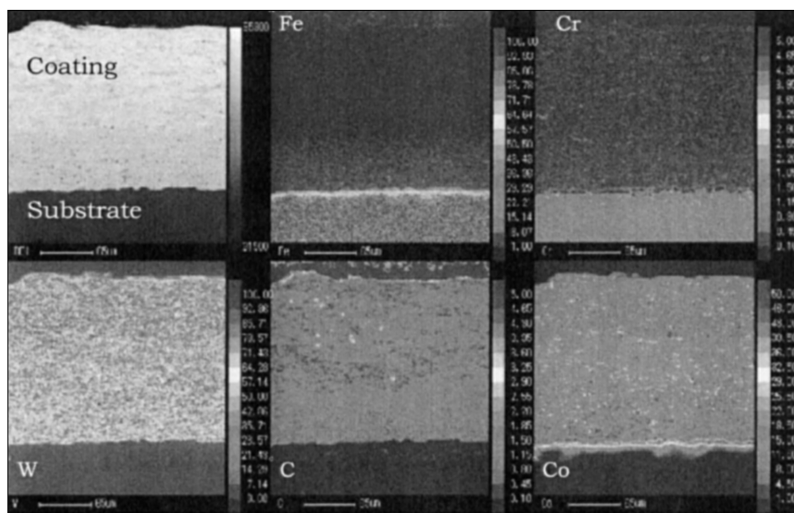


Figure 3, Analysis results of sample HC-5 by EPMA.

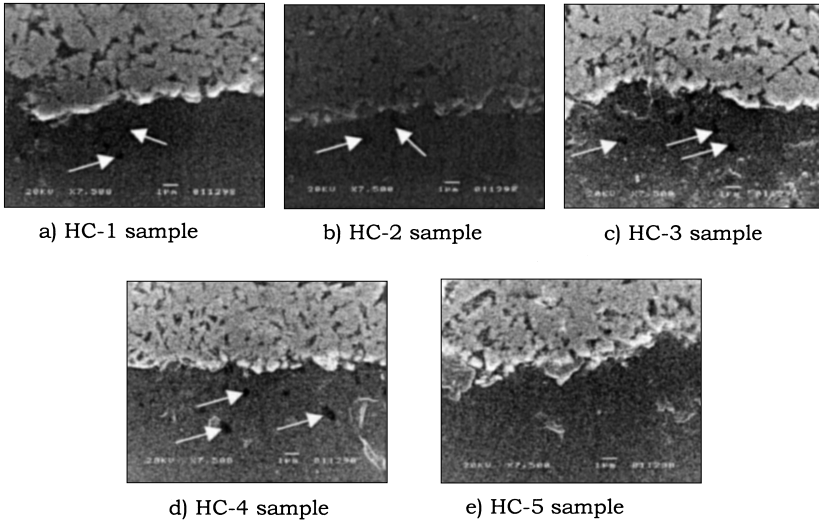


Figure 4, Cross-section at interface of each HIPed sample.
(Kirkendall pores marked by white arrows)

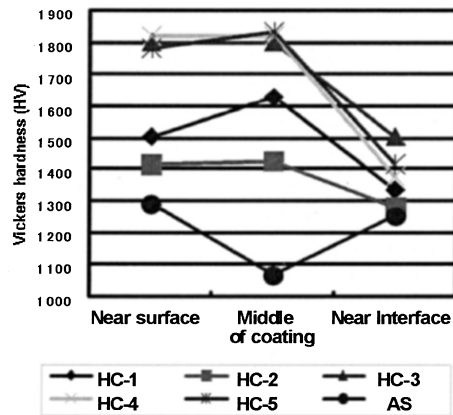


Figure 5, Hardness distribution of coatings.

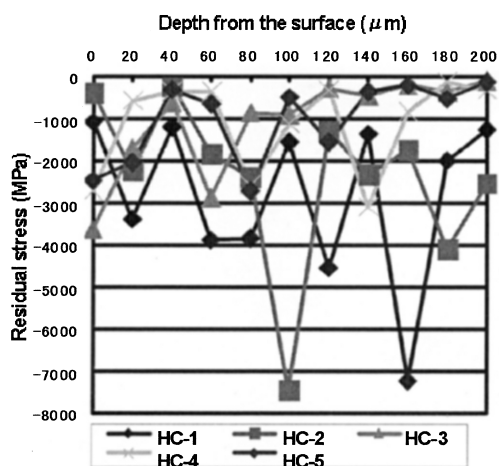


Figure 6, Residual stress distribution of HIPed coatings.

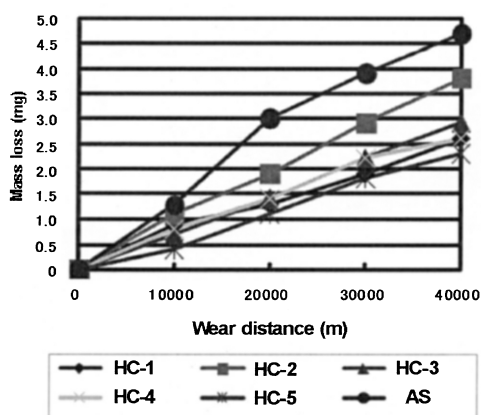


Figure 7, Wear loss of each coating.