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Coating of High-Alloyed, Ledeburitic Cold Work Tool Steel Applied by HVOF Spraying

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This study demonstrates the processing of a cold work tool steel (X220CrVMo13-4) coating using HVOF spraying. The coating formation was analyzed based on microstructure, phase, hardness, porosity, oxidation, and adhesion characteristics. An online diagnostic tool was utilized to find out the in-flight characteristics of powder such as temperature and velocity during the coating process to identify the influencing parameters to achieve dense cold work tool steel coatings with low oxidation. The influence of powder size, process parameters, and in-flight characteristics on the formation of cold work tool steel coatings was demonstrated. The results indicated that thick and dense cold work tool steel coatings with low oxidation can be obtained by the selection of appropriate powder size and process parameters.

Keywords bond coat, cold work tool steel, HVOF, online diagnostic, thermal spraying

1. Introduction

Producing tool steels as coatings on commercially available low-cost materials could import cost efficiency in production technology. In such cases, tool steels are usually prepared by employing HIP cladding and laser cladding involving solid-state diffusion bonding and normally referred as powder metallurgy (PM) coating methods (Ref 1-3). However difficulties involved in the production of PM coatings are inevitable when it comes to coat larger-volume and complicated space contours.

Thermal spray technology represents a flexible and cost-effective route for coatings on large components with complex space contours and could be the best choice for the production of such tool steels (Ref 4). Among all thermal spray coating methods, HVOF proved its versatility for the preparation of wear resistance coatings because of its relatively low-temperature and high-velocity characteristics of spraying (Ref 5). Thus, HVOF coatings display a higher density as well as fine-grained microstructure, which lead to high hardness and excellent wear resistance. Additionally, HVOF process is economically viable with large productivity. Moreover, residual stresses associated with the HVOF spraying are relatively low compared to conventional plasma spraying. This provides the platform to work on crack free thick coating of several millimeters range. However, no reports have been seen from the open literature employing cold work tool steel coating using HVOF spraying.

This study evaluates the potential of HVOF spraying to produce cold work tool steel coating of ledeburitic type. It demonstrates the influence of various factors, such as spray parameters, powder particle size, and use of suitable substrate materials on the buildup of thick cold work tool steel coatings.

2. Experimental Details

A gas atomized powder of a hypoeutectic ledeburitic cold work tool steel (X220CrVMo13-4) was used as spray material. The chemical composition of the powder is presented in Table 1. The powder was categorized into three different size fractions as follows: 25-45 μm (small size), 45-63 μm (medium size), and 63-80 μm (large size). Three different substrate materials were used: low-carbon steel, low-carbon steel coated with a bond coat of MCrAlY, and austenitic steel. The substrates were degreased before sand blasting and ultrasonically cleaned prior to coating deposition. HVOF coating was performed using a DJ2600 (Sulzer Metco) gun with a six-axis robot manipulator. The HVOF spray parameters are given in Table 2. Coating temperature was measured using optical pyrometer during spraying. To keep the substrate temperature low, compressed air with a pressure of about 4 bar was applied from the back side of the substrate. Online particle diagnostic equipment (DPV2000, Tecnar, Canada) was employed to investigate in-flight characteristics such as temperature and velocity of the cold work tool steel powder during HVOF spraying. Microstructure examinations were carried out on sectioned samples using optical and scanning electron microscope. Microhardness and macrohardness on the cross section of the coatings were measured using Vickers hardness testers with

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Table 1 Chemical composition of cold work tool steel powder used for HVOF spraying

Alloying element:	C	Mn	Si	Cr	Mo	V	O	Fe
Wt.%	2.25	0.3	0.6	13.5	1.0	4.0	0.0137	Bal

Table 2 HVOF spraying parameters used for cold work tool steel coating

Oxygen flow, SLPM	145-230
Hydrogen flow, SLPM	630-753
Oxy-fuel ratio (and equivalence ratio) used for different powder size	
Small (25-45 μm)	0.23-0.33 (2.17-1.50)
Medium (45-63 μm)	0.26-0.33 (3.71-1.50)
Large (63-80 μm)	0.31-0.35 (3.19-2.79)
Mass flow rate of different powder (g/min)	
Small (25-45 μm)	17.90
Medium (45-63 μm)	19.41
Large (63-80 μm)	19.76
Spray distance, mm	200-300
Gun traverse speed, mm/s	500

different loads. An oxygen/nitrogen analyzer (Leco TCH600) was used to determine the oxygen content of the free standing coating. Phase analysis of the powder and the coating were carried out by x-ray diffraction using CuK_α radiation.

3. Results and Discussion

3.1 Powder Characterization

The morphology of the gas atomized cold work tool steel powder is shown in Fig. 1. Powders are highly spherical and the morphology of powder is composed of a radial network dendritic structure which is an inherent characteristic of rapidly solidified ledeburitic tool steel powders (Ref 6). Figure 2 shows the XRD pattern of the cold work tool steel powder. Diffraction confirmed the presence of austenite and M_7C_3 carbides along with a small amount of martensite. Behulova et al. also reported that the main microstructural constituent of cold work tool steel (X220CrMoV13-4) powder was retained austenite (Ref 6).

3.2 Online Diagnostic

In-flight particle temperature and velocity are important characteristics during HVOF spraying that determine the microstructure and properties of the coatings. Therefore, investigations on in-flight characteristics of the cold work tool steel powders have been carried out during HVOF spraying in this study. Moreover, measurements of in-flight particle characteristics would give an idea of lower and upper limits of principal spraying variables such as oxy-fuel ratio and spray distance during optimization of the HVOF process parameters. In this study, in-flight particle temperature and velocity were measured by

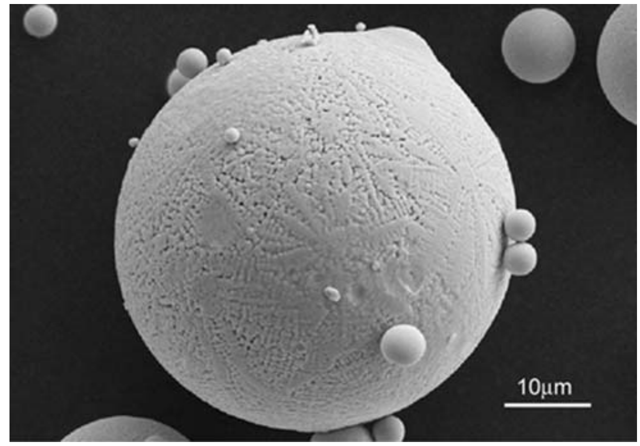

Fig. 1 Morphology of gas atomized cold work tool steel powder

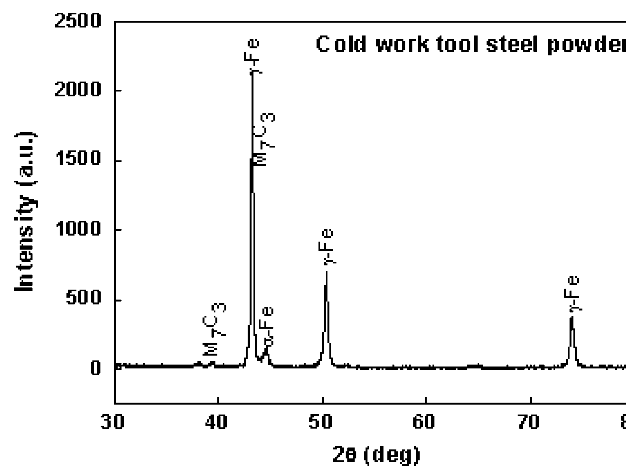

Fig. 2 XRD pattern of cold work tool steel powder

considering different values of oxy-fuel ratio, spray distance, and for different size fraction of the powder. Figure 3 shows the variation of particle temperature and velocity as a function of oxy-fuel ratio. This observation was made by varying the flows of oxygen while hydrogen flow and spray distance were kept constant. A significant increase in temperature and velocity of the particles was observed when the flow of oxygen was increased. The flame temperature is dependent on the equivalence ratio, and the maximum temperature occurs at roughly stoichiometric conditions. Hence increasing the oxygen will only increase the temperature up until an equivalence ratio of roughly one. Therefore, it implies that an increase in oxygen flow increases the flame temperature. However, changes in temperature above a certain oxy-fuel ratio were small. This can be attributed to significant oxidation of the cold work tool steel powder above a certain oxy-fuel ratio. Therefore, it might be concluded when the metallic powder is oxidized during HVOF spraying, changes in temperature during online measurements become less significant. Though low level of oxy-fuel ratio can help to

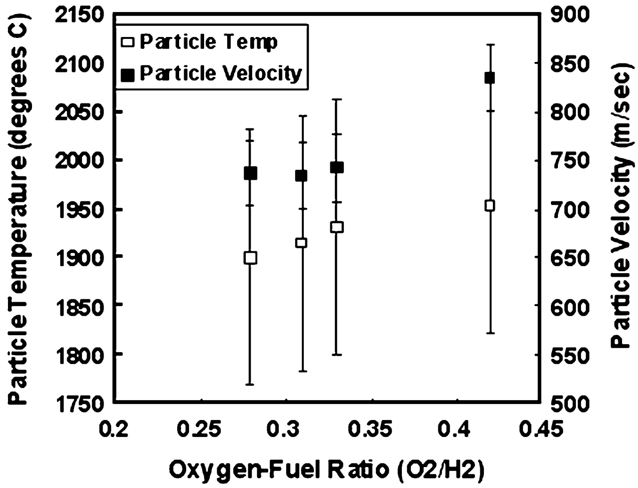

Fig. 3 The variation of particle temperature and velocity as function of oxygen-fuel ratio at a constant spray distance of 250 mm (small size powder: 25-45 μm)

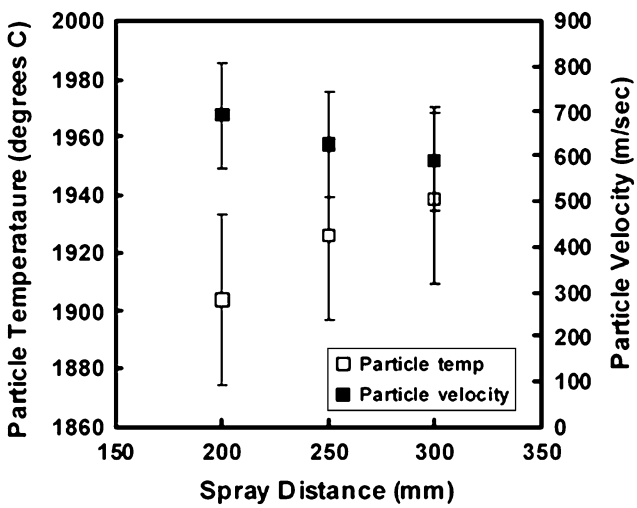

Fig. 4 The influence of spray distance on in-flight particle temperature and velocity (large size powder: 63-80 μm)

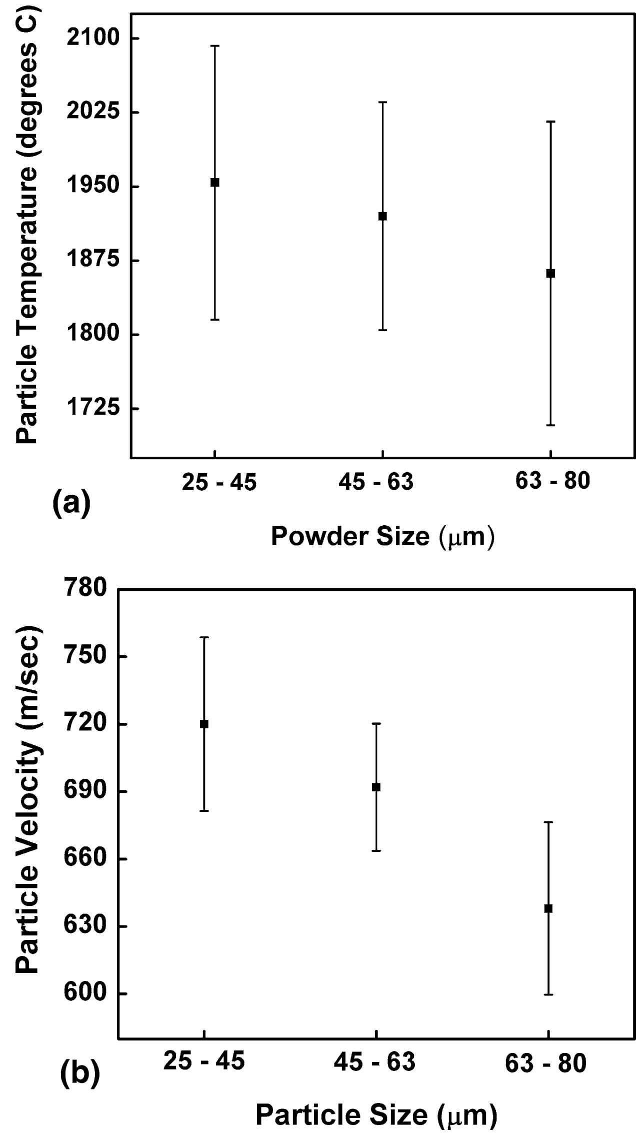

Fig. 5 The influence of particle size on in-flight particle temperature and velocity sprayed at constant oxy-fuel ratio (0.33) and spray distance (200 mm)

minimize the oxidation, this could not be employed by reducing the oxygen flow below certain level. While doing so, diamond shock waves started to disappear which leads to decrease in gas velocity by means of decreasing flame enthalpy. Therefore, a compromise has to be made on oxy-fuel ratio level for the residual oxide content in the coating (Ref 7).

Figure 4 shows the influence of spray distance on in-flight characteristics of tool steel powder at a constant fuel ratio. The results showed that the velocity of particles decreased with spray distance while the temperature was found to increase. A decrease in velocity of particle with spray distance is a quite common behavior of HVOF, and an increase in temperature with spray distance can be explained by exothermal heating of the particle surfaces due to in-flight oxidation (Ref 8). The oxy-fuel ratio employed during this online diagnostic measurement is believed to be high enough to melt the particle completely to enhance the in-flight oxidation.

Figure 5 shows the effect of particle size on the in-flight characteristics of the tool steel powder. This was carried out at a constant oxy-fuel ratio and spray distance. An apparent trend is exhibited between powder size and in-flight particle characteristics. The results show that particle temperature and velocity have been decreased with increasing particle size. These findings indicated that the size of the feedstock has to be considered as one of the important factor to refine the microstructure of coatings.

3.3 Influence of Powder Size on Coating Microstructure

HVOF coating was performed using three different feedstock sizes on three different substrate materials:

Table 3 Properties of cold work tool steel coating

Powder size, μm	Porosity, %	Hardness, $HV_{0.5}$	Oxygen content, wt.%	
			Low	High
Small	>1.3	789.66 ± 69.0	0.670 ± 0.003	1.240 ± 0.005
Medium	>0.5	730.17 ± 30.5	0.485 ± 0.001	0.829 ± 0.006
Large	>2.6	667.83 ± 53.4	0.396 ± 0.002	0.443 ± 0.001

Note: Carried out hardness and porosity measurements correspond to low-oxygen content

low-carbon steel substrate, low-carbon steel substrate coated with bond coat MCrAlY and commercially available austenitic steel. Microhardness, porosity, and oxygen content of optimized coatings are listed in Table 3. Figure 6 shows the influence of the oxy-fuel ratio on the formation of microstructures using the particle fraction 25-45 μm at a constant spray distance of 200 mm. As seen from the micrographs, the degree of oxidation was found to be decreased when reducing the oxygen flow. Figures 6(a) and (c) represents the microstructures of coatings sprayed at high and low level of oxygen flow, respectively, and their corresponding value of oxygen contents are given in Table 3. It is of significance to investigate the oxygen content of the cold work tool steel coating in this study because oxides in the coating will strongly impair mechanical behavior and corrosion resistance of the coating (Ref 5). At a high oxy-fuel ratio, the oxygen content of free standing coatings produced with small size powder was measured as 11.45 wt.%, implying that the coating was oxidized heavily due to higher degree of melting of small powder particles present in the feedstock. The heavy oxidation of the particles at high oxy-fuel ratios may also be attributed to unreacted oxygen within the mixture. Zhao and Lugscheider reported on HVOF-sprayed duplex stainless steel that convection dominated oxidation on the melted particles, inducing heavy oxidation of more than 8 wt.% when the powder particles are totally melted (Ref 9). The high oxygen content is attributed to the presence of a high amount of oxide forming elements such as Cr, V, and Mo. EDS investigations and elemental mappings confirmed that oxide zones were composed of iron, chromium, and vanadium. Zhao et al. also showed that HVOF coatings with low-oxygen content can be obtained using low level of oxygen to fuel ratio when employing stainless powder (Ref 10). This study showed that the oxygen content of coating deposited with small size powder could be reduced to 0.44 wt.% when depositing at low oxy-fuel ratio. It is also seen from the results that residual oxygen content in the coating could not be completely avoided (see Table 1). Dobler et al. studies on oxidation of HVOF stainless steel coating pointed out that oxidation can be controlled by oxy-fuel ratio, and they also pointed out that particle oxidation could not be completely avoided even at under stoichiometric conditions (Ref 11). Though coatings produced using small size powder with low oxidation, the porosity level in the coating was about 1.6%. This can be attributed to in-flight particles about 1542 ± 135 °C measured during spraying by online diagnostic at 0.23 oxy-fuel ratio. As this temperature is close to the melting point of the powder that leads to a large volume fraction of unmelted particles in the coating microstructure. Results showed that cold work tool steel coatings can be deposited using small size powder with a low degree of oxidation by controlling the oxy-fuel ratio upon compromising some porosity level in the microstructure.

Figure 7 shows the microstructures of coating sprayed with medium size powder 45-63 μm. The idea of using this narrow size distribution (45-63 μm) was to avoid the small particles that induce oxidation and large particles that remain largely unmelted. Sobolev and Guilemany studies on stainless steel coating emphasized that a narrow size distribution helps to decrease the level of the in-flight oxidation and the thickness of oxide layer (Ref 12). The influence of a narrow size distribution (medium size powder) in this study was also realized when employing cold work tool steel powder. When spraying at an oxy-fuel ratio of 0.30, a dense coating with low porosity was achieved. Its corresponding oxygen content was 0.83 wt.%. The coating microstructure is composed of melted and partially melted powders where partially melted particles were evenly distributed in the pool of melted regions. This observation indicated that a considerable amount of melting has to be taken place during coating deposition to obtain dense coatings. This can be achieved when using a medium size powder. Studies on HVOF stainless coatings showed that dense coatings can be achieved when the microstructure of the coating is composed of a combination of melted and partially melted particles during HVOF spraying (Ref 13, 14).

Figure 8 shows the microstructure of coatings sprayed with large size powder (63-80 μm) at different oxy-fuel ratio. The degree of melting was found to be improved with oxy-fuel ratio. As seen in the microstructure, porosity and volume fraction of unmelted powder particles were found to be decreased when the flow of oxygen increased. Therefore, it is clear that the porosity level has been influenced by the melting state of the tool steel powder. Oxygen content of coating produced with large size particle was measured as 0.3964 wt.%. This result implies that the feedstock with large particle size leads to the formation of a coating with low-oxygen content due to the presence of large volume fraction of unmelted particles. However, the porosity level of 3.5% could not be avoided in the coating produced with large size particles. Though operating in high-flame temperature by means of

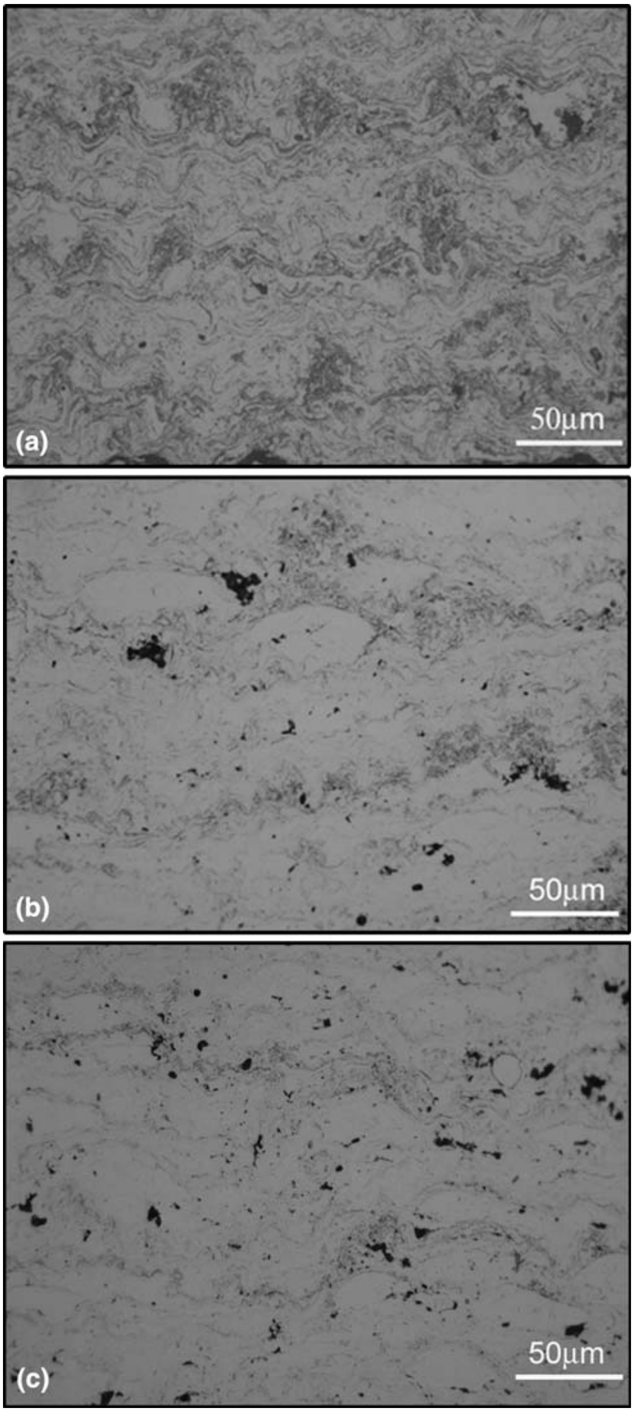

Fig. 6 Micrographs showing the effect of oxy-fuel ratio on the quality of microstructure using small powder 25-45 μm: (a) 0.29, (b) 0.25, and (c) 0.23

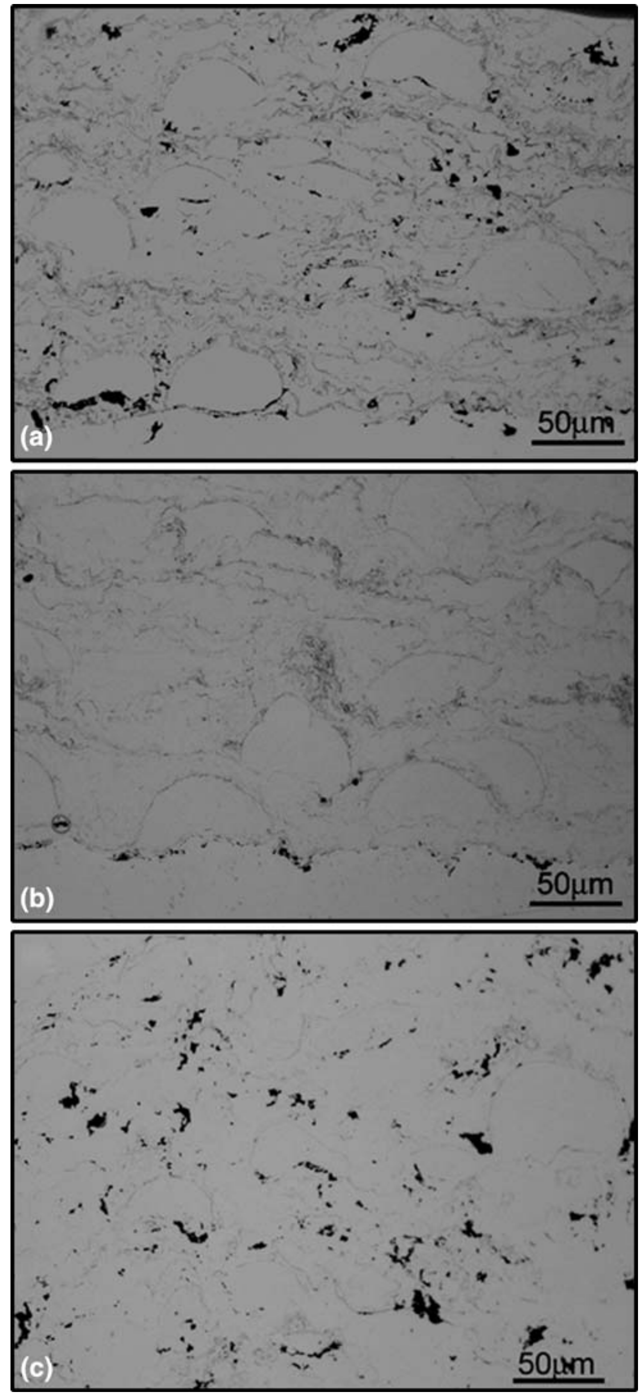

Fig. 7 Micrographs showing the effect of oxy-fuel ratio on the quality of microstructure using medium size powder 45-63 μm: (a) 0.29, (b) 0.30, and (c) 0.31

increasing oxy-fuel ratio, larger particle could not be melted completely to avoid porosity. This is attributed to the shorter dwell time (time of particle flight during spraying) associated with HVOF spraying. He et al. showed that large particles did not melt completely even at high-flame temperature due to a shorter dwell time (Ref 15).

In this study, inter-lamellar oxidation between each passes was also influenced the resultant oxide content of the coatings. This can be attributed to the high-substrate surface temperature during the spraying. Substrate surface temperature during spraying was about 520 °C and remained constant during spraying. However, this temperature was found to be high enough to induce the

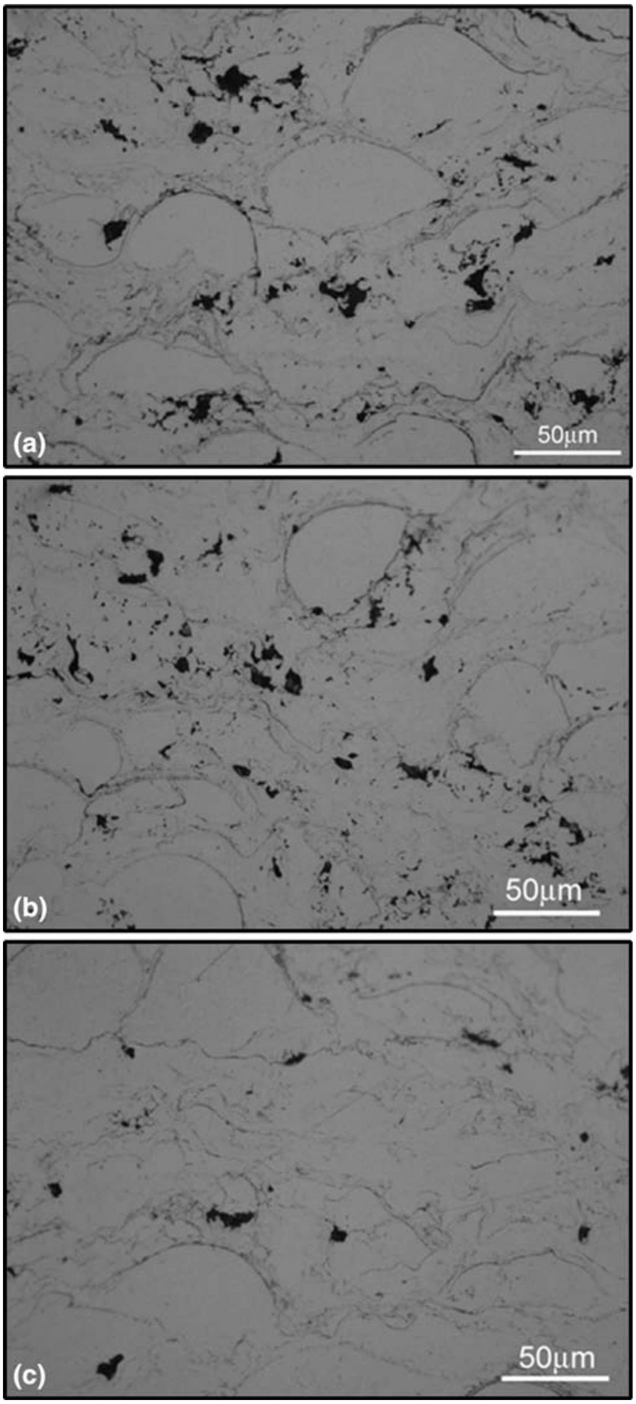

Fig. 8 Micrographs showing the effect of oxy-fuel ratio on the quality of microstructure using large size powder 63-80 µm: (a) 0.27, (b) 0.30, and (c) 0.33

inter-lamellar oxidation between each passes. This could be avoided by applying an air cooling from the back side of the substrate that showed the significant reduction of substrate temperature of about 100 °C during spraying. In addition, employing with relatively high gun traverse speed showed significant reduction coating temperature (500 mm/s).

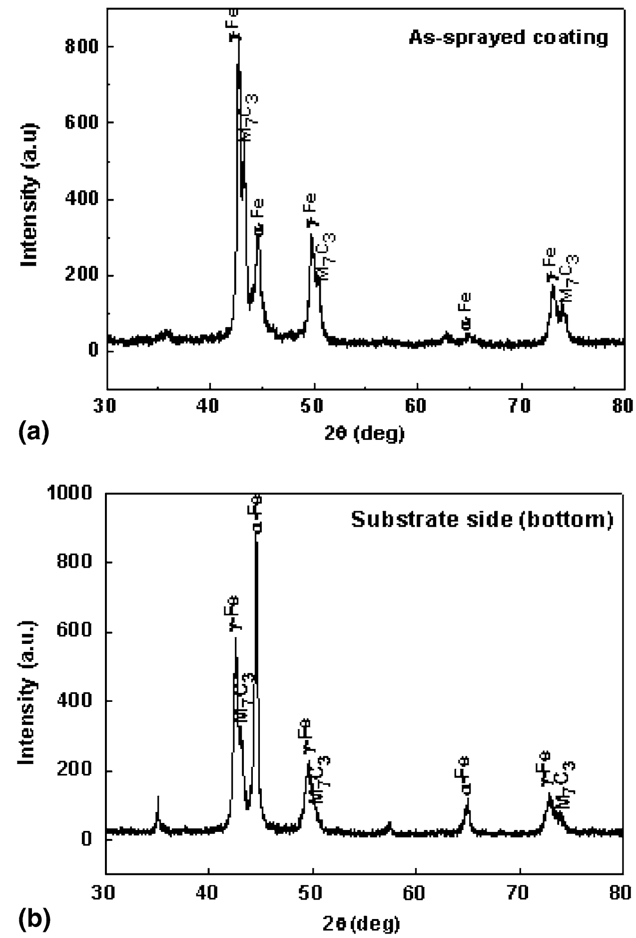

Fig. 9 XRD pattern of free standing (a) top surface and (b) bottom surface (substrate side)

Figure 9 shows the XRD pattern of cold work tool steel coatings. Investigations were carried out on as-sprayed free standing coating surfaces (top) as well as on the bottom sides (which was connected to substrate side). Phase constituents of the top surface were identified as mainly retained austenite, M_7C_3 carbide along with a small fraction of martensite. On the other hand, the phase composition of the bottom side was identified as mainly martensite, M_7C_3 carbide, and a small fraction of retained austenite. The results confirm the occurrence of martensitic phase transformation in the bottom side coating due to rapid cooling.

Because of their high hardness, cold work tool steels have been used in many wear-resistant applications (Ref 2, 16). Using the HVOF spraying process, a high hard cold work tool steel coating is realized in this study. The effect of powder size on microhardness of cold work tool steel coatings can be seen in Table 3. The results show a clear trend between feedstock size and microhardness of the coating. High microhardness of coatings produced with small size powder can be attributed to oxidation while the low microhardness of coatings produced with large powder could be due to the presence of porosity.

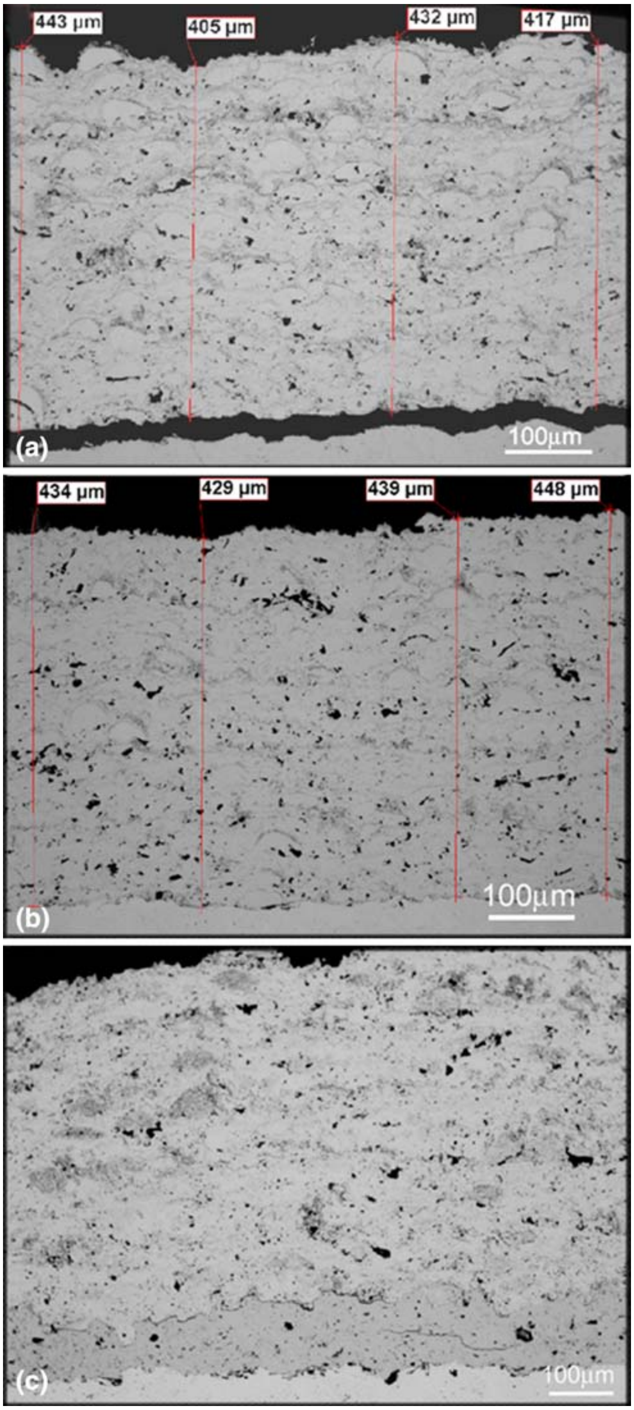

Fig. 10 Micrographs showing the influence of substrate material on interface adhesion: (a) low-carbon steel, (b) austenitic steel, and (c) low-carbon steel with bond coat (MCrAlY)

He et al. reported that a large volume fraction of unmelted particle leads to high porosity in the coating, significantly affecting microhardness (Ref 15). The macrohardness measured on the dense coating produced by medium size powder was 730 HV30 (HRC ~ 61). This showed the potential of HVOF-sprayed cold work tool steel coatings.

3.4 Adhesion of Tool Steel Coating

It is essential to discuss the adhesion characteristics of HVOF-sprayed cold work tool steel coatings on different commercially available steel substrates. Cross section micrographs are shown in Fig. 10, revealing adhesion characteristics of cold work tool steel coatings deposited on different substrate materials. Using a low-carbon steel substrate, the coating was found to be adhered when the coating thickness was lower than 100 μm. As the deposition thickness of the coating increased above a certain level, the coating was found to be delaminated from the substrate. Coating delamination was observed in the cross section microstructure. However, interfacial delamination was not observed while employing low-carbon steel with a bond coat and austenitic steel as substrates. This interfacial adhesion behavior is attributed to the residual stresses induced by martensitic phase transformation of the cold work tool steel occurring at 190 °C during cooling. As shown in the previous section, the bottom side of the coating (substrate side) is mainly composed of martensite with a small fraction of retained austenite while the feedstock powder was mainly composed of retained austenite that subsequently transformed to martensite upon cooling from high temperature. During the rapid cooling, face centered cubic (FCC) phase of retained austenite is transformed into metastable body centered tetragonal (BCT) martensite due to the lattice contraction accompanied by the lattice strain. Thus, this martensite transformation causes internal tensile residual stresses associated with volumetric expansion, leading to premature failure at the interface during HVOF spraying of a cold work tool steel coating. Using austenitic steel substrate or a bond coat, this volumetric expansion was compensated by the high-thermal expansion ability of the austenitic steel compared to low-carbon steel substrate. It is also believed that austenitic stainless steel has the ability to control the delamination cracking at the interface due to its good plasticity as long as the residual stresses generated at the coating do not exceed the tensile strength of stainless steel substrate (Ref 17). Importantly, coatings up to few millimeters in thickness can be deposited.

4. Conclusions

In this study, the formation of thick cold work tool steel (X220CrVMo13-4) coatings was realized through HVOF thermal spraying. Influence of various factors, such as spray parameters, powder particle size, and use of suitable substrate materials on build up of thick cold work tool steel coating was demonstrated. The following conclusions were drawn:

1. Oxy-fuel ratio (O_2/H_2) was identified as the most influencing parameter through online diagnostic and microstructural analysis to produce dense tool steel coatings with low oxidation.
2. Dense coatings with low-oxygen content can be achieved by means of narrow size distribution of

powder. In this study, medium powder size (45-63 μm) was identified as appropriate size to produce dense coating with low oxidation.

3. Importantly, HVOF-sprayed cold work tool coatings are prone to martensitic phase transformation upon subsequent cooling from high temperature, leading to interface delamination. This interface delamination as a consequence of martensitic phase transformation can be overcome by appropriate choice of substrate materials or using a bond coat of MCrAlY.

Acknowledgments

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