



# Influence of the HVOF Gas Composition on the Thermal Spraying of WC-Co Submicron Powders ( $-8 + 1 \mu\text{m}$ ) to Produce Superfine Structured Cermet Coatings

W. Tillmann, E. Vogli, I. Baumann, G. Matthaues, and T. Ostrowski

(Submitted May 29, 2008; in revised form July 25, 2008)

Thermal spraying technology represents a novel and promising approach to protect forming tools with complex surfaces and highest shape accuracy against abrasive wear and galling. However, due to high or nonuniform layer thicknesses or inappropriate surface roughness conventional coarse-structured coatings are not suitable to achieve this aim. The application of novel submicron or nanoscaled feedstock materials in the thermal spray process can provide the deposition of cermet coatings with significantly improved characteristics and is recently of great interest in science and industry. In this collaborative study, the feeding and HVOF spraying of WC-Co submicron powders ( $-8 + 1 \mu\text{m}$ ) have been investigated to manufacture superfine structured, wear resistant, near-net-shape coatings with improved macroscopic properties and smooth surfaces. The influences of varying HVOF gas compositions on the spray process and the coating properties have been analyzed.

**Keywords** deep-drawing tools, hard material coatings, HVOF, near-net-shape coatings, submicron powders, superfine structured and nanostructured coatings, surface engineering, thermal spraying, fine powder feeding

## 1. Introduction

Deep-drawing is a widely used sheet metal forming process in the aircraft and automotive industries. The manufacturing of modern parts with complicated shapes and curvatures requires forming tools with highest shape accuracy even at complex surface geometries. However, the application of novel, high-strength sheet metals combined with a continuous increase in productivity impose high tribological demands on forming tools and finally lead

This article is an invited paper selected from presentations at the 2008 International Thermal Spray Conference and has been expanded from the original presentation. It is simultaneously published in *Thermal Spray Crossing Borders, Proceedings of the 2008 International Thermal Spray Conference*, Maastricht, The Netherlands, June 2-4, 2008, Basil R. Marple, Margaret M. Hyland, Yuk-Chiu Lau, Chang-Jiu Li, Rogerio S. Lima, and Ghislain Montavon, Ed., ASM International, Materials Park, OH, 2008.

W. Tillmann, E. Vogli and I. Baumann, Institute of Materials Engineering, Technische Universität Dortmund, Leonhard-Euler-Str. 2, 44227 Dortmund, Germany; and G. Matthaues and T. Ostrowski, Thermico GmbH and Co. KG, Franz-Schlueter-Str. 10-16, 44147 Dortmund, Germany. Contact e-mail: wolfgang.tillmann@udo.edu.

to increasing wear. To reduce the high costs for the repair and the maintenance of such tools, the surface has to be modified by an appropriate modification process, which is able to preserve the high dimensional accuracy. Thermal spraying has been regarded as a promising and cost-efficient surface engineering technology, which has emerged as a viable solution for a wide range of wear and corrosion resistance applications to improve the service life of tools and other machine components (Ref 1-3). It is well known that HVOF spraying provides far superior results than any other thermal spraying technique for manufacturing cermet coatings, because of the much higher gas jet velocity and lower flame temperature. These result in coatings with extremely low porosity, low splat oxidization, and low carbide decomposition or dissolution (Ref 4). However, for the protection of deep-drawing tools with complex surface geometries and highest shape accuracy, conventional cermet coatings obtained by HVOF spraying of coarse-grained feedstock powders ( $>15 \mu\text{m}$ ) are not suitable, as they impose high or nonuniform layer thicknesses or inappropriate surface roughness. A subsequent cost-efficient surface finishing, for example, by grinding, is often necessary to obtain appropriate surface characteristics with high dimensional accuracy. Thermal spraying of novel submicron or nanoscaled feedstock materials provides significant improvements in macromaterial properties for many applications and is recently of great interest in science and industry (Ref 5). Fine powders not only show a distinct potential for the deposition of thin near-net-shape coatings with high surface quality, but are also very promising for the production of dense and homogeneous coatings with improved mechanical properties leading to an enhanced wear behavior (Ref 6, 7).

The mechanical and functional properties of engineering materials are to a large extent determined by their microstructure, particularly the distribution of phases and their crystallite sizes. Studies by Jia and Fisher (Ref 8, 9) on sintered nanostructured WC-Co materials have been reported to exhibit enhanced performance in both sliding and abrasive wear and substantially higher hardness than their conventional coarse structured counterpart. A similar approach of using sintered and agglomerated micron-sized powders with submicron or nanosized WC particles in a Co matrix to improve the performance of HVOF-sprayed cermet coatings has been pursued by other researchers (Ref 5). These studies concluded that in comparison to coatings with conventional coarse-grained microstructure, the deposition of superfine and nanostructured coatings leads to a reduction of the friction coefficient and the roughness by a factor of two and to a wear reduction by a factor of five (Ref 10). However, these advantages can only be achieved as long as appropriate process technologies are employed, which preserve the submicron or nanoscaled feedstock material structure in the final as-sprayed coating morphology. Due to the large specific surface of fine powders, they show high thermal susceptibility. A sensitive temperature control of the HVOF process is fundamentally required to avoid particle overheating. At the same time, the low specific weight of fine powder particles has to be compensated by higher process gas velocities to provide the particles with adequate kinetic energy. Conventional HVOF techniques cannot completely solve this problem. Therefore, the use of modern technologies to process fine particles from submicron to the nanometric scale for novel macro-applications is becoming more and more important in the spraying technology (Ref 11). In the present study, the feeding and HVOF spraying of WC-Co submicron powders have been investigated to manufacture superfine structured, wear resistant, near-net-shape coatings with improved macroscopic properties. During the spray experiments, the influence of different HVOF gas compositions with varying kerosene, oxygen, and hydrogen levels on the process dynamics and the corresponding coating characteristics have been analyzed. The focus was placed on achieving dense coatings with high hardness and a smooth surface. The velocity and the temperature of the thermally sprayed particles were measured in-flight. A special powder feeder system and a novel HVOF device, designed by Thermico and optimized for processing finest powder fractions, were used (Ref 12).

## 2. Experimental

### 2.1 Powder Feedstock

88WC-12Co irregular and broken submicron powder (Thermico SJA 610/48,  $-8+1\ \mu\text{m}$ ), separated from a standard powder distribution by cyclone sieving, was employed as feedstock material. A SEM picture of the powder morphology is illustrated in Fig. 1. To minimize agglomeration effects, the fine powder was heated up to

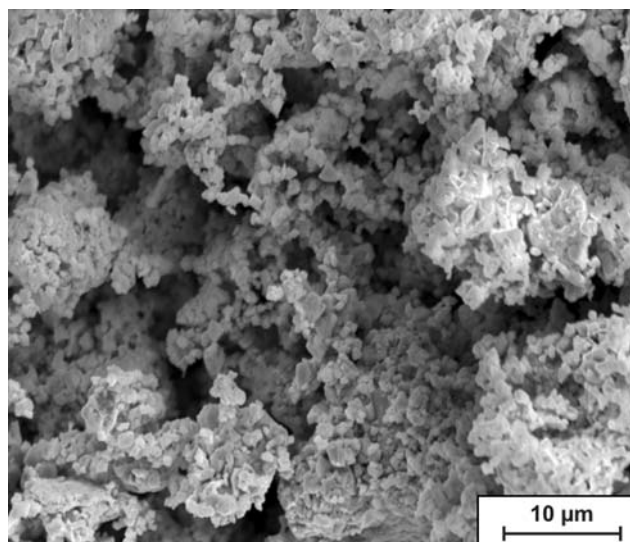


Fig. 1 88WC-12Co submicron powder used in this study

120 °C in a furnace for 1 h before using it in the feeder system.

### 2.2 Powder Feeding and HVOF Equipment

Thermico CPF2-Twin powder feeder system equipped with special features and optimized for the reliable processing of fine powder fractions was used to feed the fine particles into the HVOF gun. A further description of this powder feeder system is given by Mathaeus et al. (Ref 12-14). To minimize the risk of particle agglomerations, both powder containers were heated up to 60 °C with heating sleeves and were agitated with a vibrator pressure of 6.2 bar. In addition, a cyclical evacuation of the powder containers and a subsequent hot powder gas flushing at 80 °C were performed prior to the feeding operation. Thermico CJS-HVOF gun with novel K5.2 combustion chamber design and dual-radial powder injection was used to conduct the coating experiments. This HVOF system is optimized for the fusibility and the acceleration of fine-scaled powder fractions  $<15\ \mu\text{m}$ . It works with a two-stage hydrogen-oxygen and a subsequent liquid fuel (kerosene) oxygen combustion as described by Mathaeus et al. (Ref 12), which allows to control the velocity and the temperature of the process gas flow almost independently (Ref 6). The parameters for the powder feeding and the HVOF gun velocity as well as the standoff distance between the gun and substrate are summarized in Table 1.

### 2.3 Substrate Material and Preparation

Rectangular C45 steel specimens (1.0503) were employed as substrate material for the coating experiments. In preparation for thermal spray experiments, the surfaces were grit-blasted with fine-grained F100 alumina (106-150  $\mu\text{m}$ ) using a blasting air pressure of 3 bar. Afterward, the samples were cleaned for 15 min in an ethanol ultrasonic bath and heated to 110 °C in a furnace for about 30 min.

## 2.4 Coating Process

During the spraying experiments, the kerosene, oxygen, and hydrogen levels were varied in accordance with Fig. 2.

Whereas one gas/fuel level was varied in a set of experiments, the other ones were kept constant to analyze the influence on the process dynamics and the corresponding coating properties. The temperature and the velocity of the particles were measured in-flight at a standoff distance of 150 mm with Accuraspray-g3 device (Tecnar, Canada). Simultaneously, the chamber pressure and the combustion energy, transferred to the coolant, were detected by means of sensors, which are integrated in the HVOF system. During the coating experiments, the backsides of the specimens were constantly cooled with compressed air (2.7 bar). Characterization of the coatings in terms of hardness, porosity, and roughness were performed. In addition, the deposition efficiency was determined. The surface roughness was measured according to the DIN 4760 standard with a T-1000 tracing stylus instrument (Hommel, Germany). An M-400 microhardness tester (LECO, Germany) was employed to ascertain the coating hardness. Cross-section images of the coating

and the interface between coating and substrate material were taken by AXIOPHOT light microscope (Zeiss, Germany). In addition, the layer thickness was determined. The porosity was analyzed with integrated image analysis software Axiovision 4.63 Outmess at a magnification level of 200, degressive contrast amplification (using gamma function), and medium illumination level. Cross-section and fracture section images of the coating with higher magnification were made with JXA 840 scanning electron microscope with secondary-electron and backscattered electron detectors (Jeol, Germany). The sliding wear behavior and the friction coefficient were evaluated using a pin-on-disc tribometer (CSEM, Switzerland) with alumina ball ( $\varnothing$  6 mm,  $1985 \pm 193$  HV 0.1), a load of 10 N, and a distance of 10 km at ambient temperature.

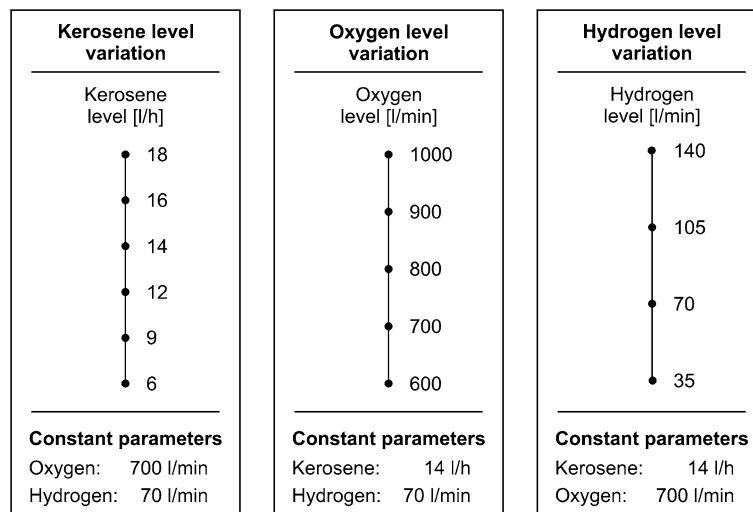
## 3. Results and Discussion

### 3.1 In-flight Particle Diagnostics

The in-flight particle measurements in this study showed that the chamber pressure and the thermal energy provided by the HVOF gas combustion allowed control of the temperature and velocity of the thermally sprayed submicron powders. However, the kerosene and oxygen levels showed a primary influence on the process dynamics. With rising kerosene level an almost linear increase in the thermal energy in the combustion chamber (32 kW at 6 L/h and 75 kW at 18 L/h), transferred to the coolant, is obtained (Fig. 3). However, kerosene flows between 6 and 14 L/h exhibited no significant change in the particle temperature around 1600 °C. It seems that the whole thermal energy in the HVOF flame is used for the particle melting. Higher kerosene levels (16-18 L/h) add more thermal energy to the process. A larger portion of the available fuel is burned within the combustion chamber

**Table 1** Parameters

Parameter	Values	Comment
Number of powder containers	2	
Feeder disc velocity, rpm	3	Per powder container
Carrier gas flow, L/min	13	Per powder container
Vibrator pressure, bar	6.2	Per powder container
Heating sleeve temp., °C	60	Per powder container
Powders gas heating temp., °C	80	Per powder container
Stand-off distance, mm	150	
Gun velocity, mm/min	30,000	In all directions
Step in vertical (Y-)direction, mm	4	
Number of passes	25	



**Fig. 2** Varied HVOF gas compositions

and the expansion nozzle. At the same time, a stronger postcombustion in the free jet is obtained, leading to higher particle jet temperatures at a maximum of 1900 °C. As shown in Fig. 3, the intermediate kerosene levels (12-14 L/h) showed highest values (747 ± 6 m/s) in terms of the particle velocity and should be favored to achieve a coating structure with high density. Whereas the oxygen level showed a significant influence on the particle velocity in Fig. 4, only a minor correlation to the particle temperature could be found. Rising oxygen levels from 600 L/min to 1000 L/min correlate with higher combustion chamber pressures from 8.8 bar (600 L/min oxygen) to 13.9 bar (1000 L/min oxygen). Subsequently, higher process gas velocities are achieved, leading to an increase in the particle velocity from 639 ± 3 m/s (600 L/min) to 931 ± 5 m/s (1000 L/min). This tendency has already been observed by Li et al. (Ref 15, 16). Due to higher process gas velocity the particles are supplied with more kinetic energy, which is transformed into higher impact forces on the substrate material. The high combustion chamber pressure also enables a particle adhesion of partially solidified particles, which impinge on the substrate material, as described by Kreye et al. (Ref 17). At the same time, a slight but continuous increase in temperature from 1580 ± 6 °C (at 600 L/min oxygen) to 1665 ± 2 °C (at 800 L/min oxygen), followed by a lower temperature increase to 1675 ± 3 °C (at 1000 L/min oxygen) have been observed with rising oxygen levels, as shown in Fig. 4. Due to corresponding higher chamber pressure and higher process gas velocities, the combustion is further transferred from the combustion chamber into the acceleration nozzle and the free jet.

Particles are exposed to a higher energy generated by the oxygen-kerosene combustion resulting in an increase

in the particle temperatures. However, at highest oxygen levels (from 900 to 1000 L/min) the combustion reaction is supersaturated. Hence, the excess of oxygen leads to a slight cooling of the HVOF jet. In contrast to these results, the variation of the hydrogen level showed no measurable influence on the thermokinetic behavior of the particles but an observable effect on the HVOF flame stability. Within the two-stage hydrogen-oxygen and kerosene-oxygen combustion, low kerosene levels (<9 L/h) lead to an unstable and strongly pulsating flame. However, in some cases, a low kerosene level is required to supply the HVOF flame with lower thermal energy in order not to overheat sensitive feedstock powder materials. Consequently, the use of higher hydrogen levels is indispensable to stabilize the kerosene-oxygen combustion. In this case, the hydrogen level has a strong influence on the thermokinetic flame behavior to achieve a homogenous particle melting and a uniform coating deposition by avoiding flame pulsations. However, at higher kerosene levels the two-stage combustion is dominated by kerosene and oxygen. A nonpulsating flame is obtained, which does not have to be stabilized by the addition of higher hydrogen levels. From a scientific point of view, the particle velocity and particle temperature are the main parameters affecting the thermal spraying process. They determine the deposition efficiency of the HVOF process and strongly influence the coating microstructure development (Ref 18). Studies by Li et al. on in-flight phenomena in HVOF spraying of WC-Co particles to deposit cermet coatings (Ref 15, 16) have shown that the particle-melting degree and particle velocity also depend on the morphology and the grain size of the feedstock powder. Furthermore, it has been reported by Bobzin et al. that in

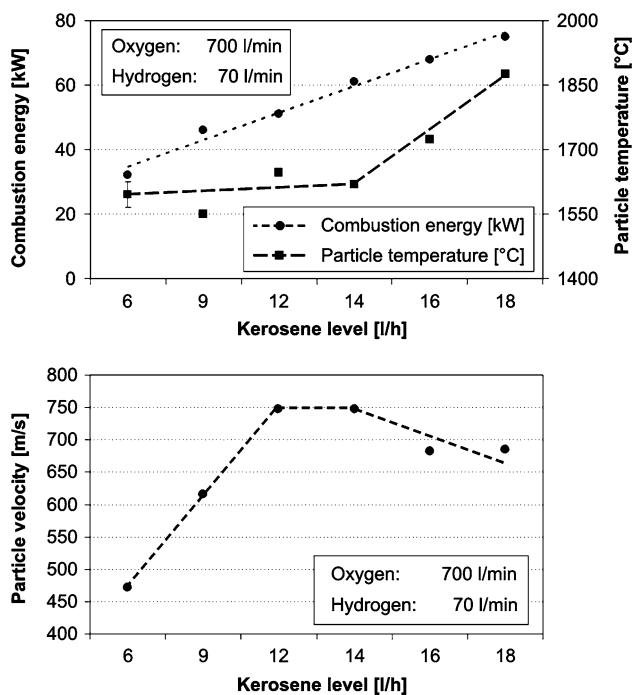


Fig. 3 Influence of the kerosene level on the HVOF process

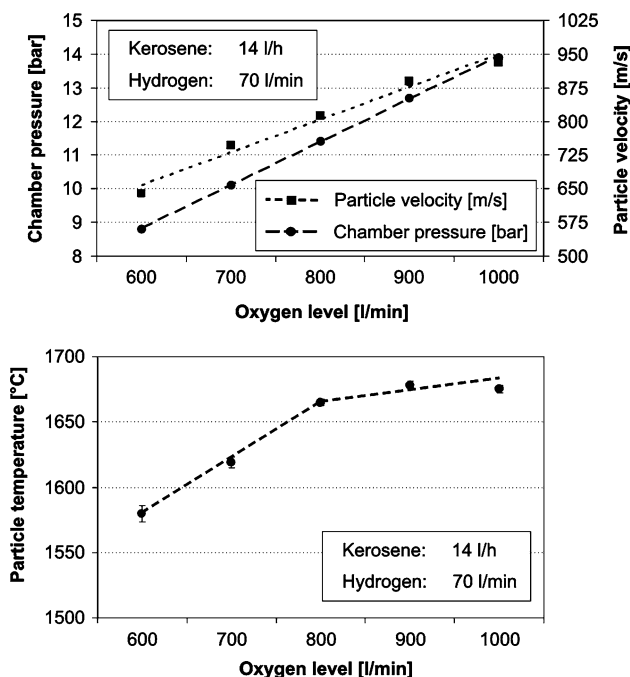


Fig. 4 Influence of the oxygen level on the HVOF process



contrast to conventional coarse-grained WC 10Co 4Cr powders ( $-30+16\ \mu\text{m}$ ) finer particles of the same composition ( $-15+1\ \mu\text{m}$ ) show continuously higher particle temperatures even at “colder” spray parameters in the HVOF process. They explained this effect by the higher surface energy of finer powders (Ref 6).

### 3.2 Deposition Efficiency

The deposition efficiency of WC-Co submicron powders sprayed at varying HVOF gas compositions has been determined. Figure 5 shows the results as a function of the kerosene and oxygen levels. The experiments indicate that both gas levels have a detectable influence on the deposition efficiency. Whereas highest values of deposition efficiency (68%) and a layer thickness of  $432 \pm 8.38\ \mu\text{m}$  could be achieved with 25 passes of the gun at lowest oxygen level (600 L/min), a continuous decrease in deposition efficiency has been observed with increasing oxygen level. At 1000 L/min, the lowest value of deposition efficiency (34%) and a layer thickness of  $244 \pm 14.66\ \mu\text{m}$  have been determined. Higher oxygen levels lead to faster particle acceleration and consequently to a shorter dwell time of the powder particles in the HVOF jet. This has an effect on the fusing of the particles. A lower number of particles are melted leading to a decrease in deposition efficiency. The kerosene level also showed an influence on the deposition efficiency. With the lowest kerosene level of 6 L/h, a

coating deposition could not be achieved using submicron WC-Co powders. The particles are not supplied with enough thermal energy in the HVOF process and cannot be sufficiently transferred in a molten state. Consequently, the predominant solid particles bounce off as they hit the substrates surface, as reported by Li et al. (Ref 15, 16). As demonstrated in Fig. 5, the values of deposition efficiency rise from 48% (at 9 L/h kerosene flow) and a layer thickness of  $308 \pm 6.03\ \mu\text{m}$  with increasing kerosene level to a maximum deposition efficiency of 57% (14 L/h) and a layer thickness of  $385 \pm 17.84\ \mu\text{m}$ . However, at higher kerosene levels (14–18 L/h), the particles are exposed to higher thermal energy, which leads to a slight decline in deposition efficiency (54%) and to a layer thickness of  $348 \pm 10.72\ \mu\text{m}$ . Investigations on varying hydrogen levels showed no significant influence on the deposition efficiency.

### 3.3 Hardness and Wear Resistance

It is generally known that hardness is an important parameter, which significantly affects the sliding and abrasive wear resistance of coatings. The deposited superfine structured coatings in this study showed Vickers hardnesses between  $1027 \pm 47\ \text{HV } 0.1$  and  $1312 \pm 45\ \text{HV } 0.1$ . It has been found that variations of the HVOF gas composition have an influence on the coating hardness. Whereas lower hardness values but no clear trend could be determined between a level of 9 to 12 L/h kerosene, highest hardness values of  $1244 \pm 89\ \text{HV } 0.1$  could be measured on coatings deposited with medium kerosene (14 L/h) and medium hydrogen (70 L/min) flows (Fig. 6a). This effect may be attributed to the larger proportion of available fuel, which is burned in the combustion chamber, the acceleration nozzle, and the free jet using increasing levels of kerosene from 9 to 14 L/h. Consequently, higher thermal energy is applied in the HVOF process leading to better and more homogenous particle fusing. As reported by Li et al., a higher melting degree is considered to be desirable to obtain dense coatings. When the particle-melting ratio is low, many large particles may bounce off as they hit the substrate and the deposited coatings tend to have a high porosity (Ref 15, 16). However, higher levels of kerosene ( $>14\ \text{L/h}$ ) resulted in a slight reduction in hardness again to a value of  $1163 \pm 32\ \text{HV } 0.1$ . This may be explained by the fact that further addition of thermal energy to the particles leads to decarburization and thermally activated carbide dissolution, as has been described in many studies. It has also been reported that fine powders show a higher thermal sensitivity in the HVOF process than their conventional coarse-grained counterparts. A reduction of the powder grain size corresponds with higher tendency toward decarburization effects. The higher surface area-to-volume ratio of fine particles leads to enhanced kinetics of the decarburization process (Ref 19).

As shown in Fig. 6(b), only a slight increase in the coating hardness could be observed with increasing oxygen level in the thermal spraying process. Whereas lowest hardness of  $1182 \pm 84\ \text{HV } 0.1$  has been found at 600 L/min oxygen flow, the highest value of  $1312 \pm 45\ \text{HV } 0.1$  has

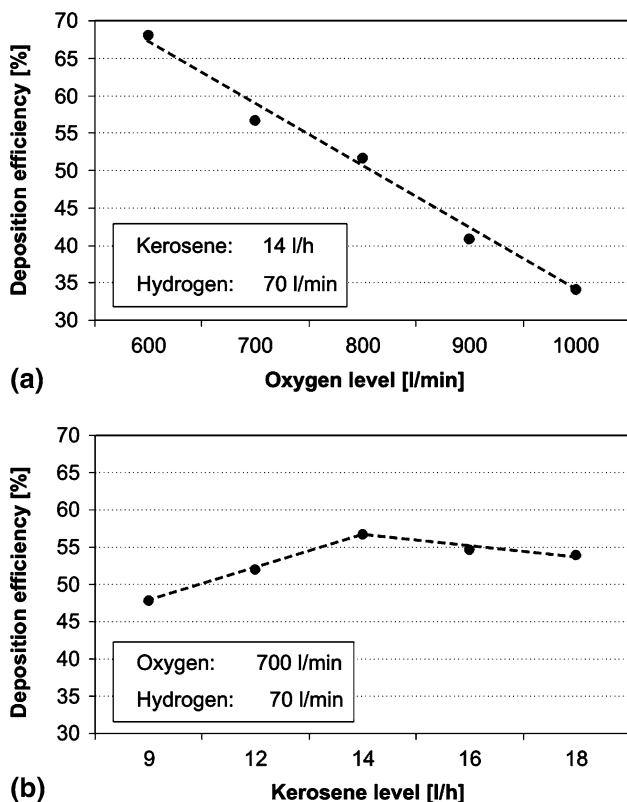
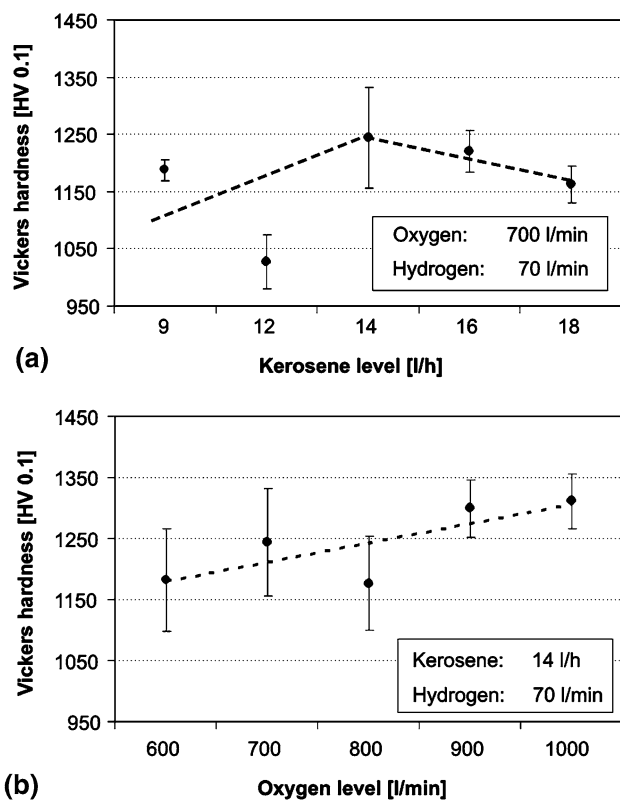


Fig. 5 Deposition efficiency depending on the oxygen (a) and the kerosene (b) levels

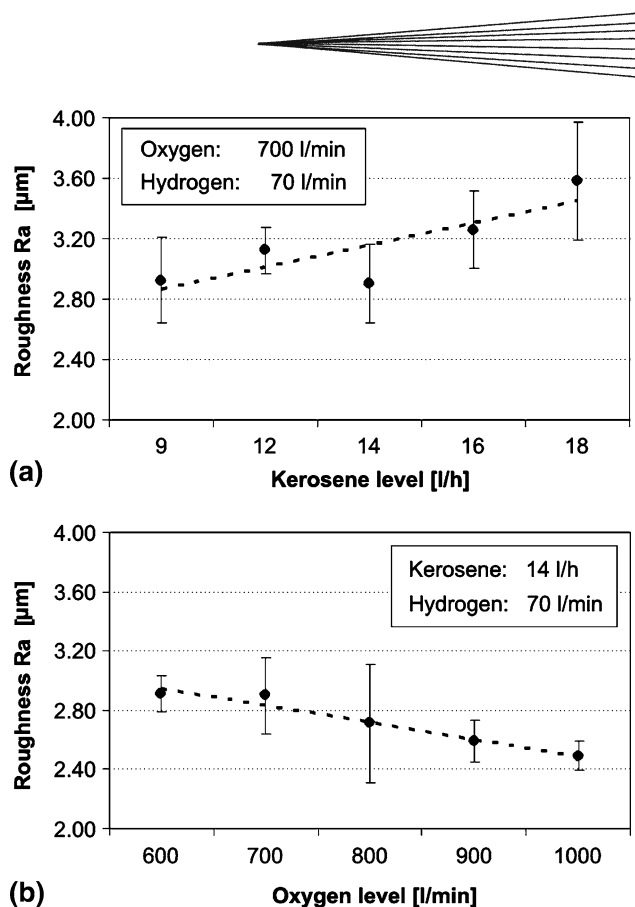


**Fig. 6** Coating hardness depending on the kerosene (a) and the oxygen (b) levels

been detected at the highest oxygen level of 1000 L/min. Higher oxygen levels provide the HVOF particle jet with more kinetic energy, which is transformed into higher impact force. This causes strong splat deformation and a subsequent densification. Therefore, a fine and dense coating structure could be achieved. Due to their outstanding combination of high resistance against abrasive, sliding, and erosive wear, high adhesive strength, and high impact stress, WC-Co cermet coatings are mainly used for industrial wear protective applications (Ref 20, 21). Studies on the wear behavior of conventional and nanostructured HVOF-sprayed coatings by Shipway et al. have reported achieving higher hardness with reduced powder grain size leading to enhanced sliding and abrasive wear characteristics of the coatings. They ascribed this effect to greater material strengths and reduced tendency for binder phase extrusions with decreasing grain size in the coatings (Ref 19). In this study, the superfine structured WC-Co coatings show comparable high resistance against sliding wear. The pin-on-disc test did not lead to a measurable weight or volume loss after a wear distance of 10 km and a load of 10 N. The friction coefficient was determined to be  $0.74 \pm 0.06$ .

### 3.4 Surface Roughness

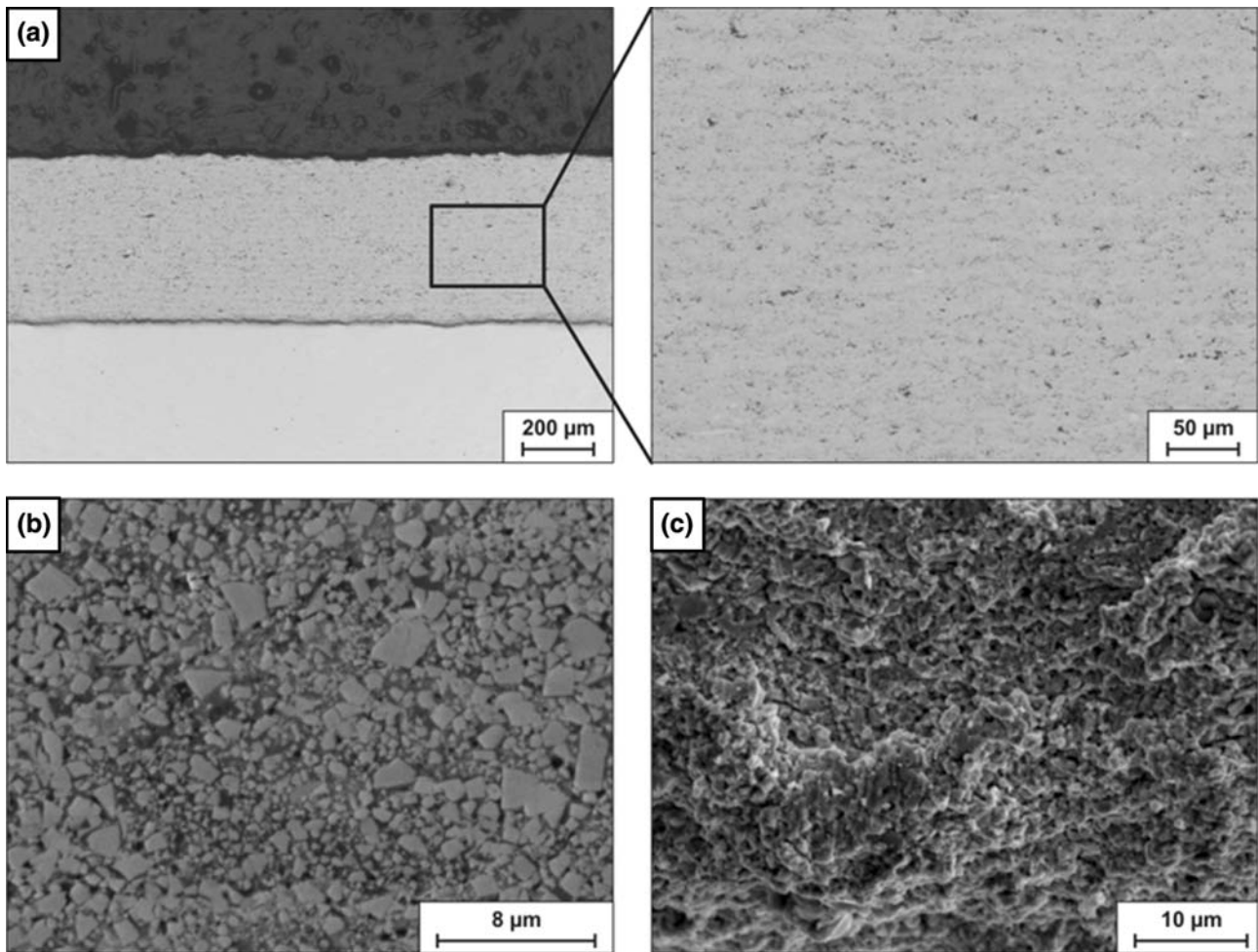
The deposited superfine structured WC-Co coatings show a smooth surface as-sprayed with low roughness Ra values ranging from  $2.49 \pm 0.10 \mu\text{m}$  to  $3.58 \pm 0.39 \mu\text{m}$ .



**Fig. 7** Influence of the kerosene (a) and oxygen (b) levels on the surface roughness Ra of the as-sprayed coatings

These results clearly indicate an improvement in surface roughness, which can be provided by thermal spraying of submicron powders. Comparative experiments on HVOF spraying of conventional coarse-grained agglomerated and sintered 88WC-12Co powders (Sulzer Metco WOKA 3102,  $-45 + 15 \mu\text{m}$ ) with spherical form have been conducted. The cermet coatings, which have been deposited on C45 steel substrates, feature a higher surface roughness Ra of  $4.21 \pm 0.74 \mu\text{m}$  as-sprayed. In studies on HVOF spraying of conventional agglomerated and sintered 88WC-12Co powders with spherical morphology performed by Magnani et al. (Ref 22) and Fedrizzi et al. (Ref 23), similar results in terms of the surface roughness were achieved. Magnani et al. used Amperit 518.074 ( $-45 + 15 \mu\text{m}$ ) powders and Sulzer Metco DJH 2700 gun in their research work on the tribocorrosion behavior of HVOF cermet coatings. The deposited coatings showed an average roughness Ra of  $5.50 \mu\text{m}$  (Ref 22). Fedrizzi et al. investigated the influence of HVOF parameters on the corrosion and wear resistance of cermet coatings deposited onto C45 steel using a Tafa/Praxair JP-5000 HVOF gun and powder grain sizes in the range of 20 to  $40 \mu\text{m}$ . Thereby, an as-sprayed coating roughness Ra between 3.7 and 4.9 was determined (Ref 23).

During thermal spraying of WC-Co submicron powders, it has been found that the combustion and gas dynamics, mechanical and thermal in-flight behavior of the powder particles, and droplet flattening have



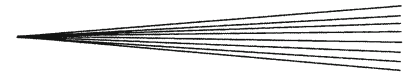
**Fig. 8** Polished cross-section images of the coating morphology taken by light microscope (a) and SEM (b) and SEM image of the fracture cross section (c)

considerable influence on the coating quality, as already reported by Sobolev et al. (Ref 24). Whereas rising kerosene levels lead to an increase in surface roughness  $R_a$  from  $2.93 \pm 0.28 \mu\text{m}$  (at 9 L/h kerosene) to  $3.58 \pm 0.39 \mu\text{m}$  (at 18 L/h kerosene), shown in Fig. 7(a), an improvement in roughness can be obtained with higher oxygen levels. The roughness  $R_a$  decreases from  $2.91 \pm 0.12 \mu\text{m}$  at 600 L/min oxygen to  $2.49 \pm 0.10 \mu\text{m}$  at 1000 L/min (Fig. 7b). Correlated to the in-flight particle measurements, the reduction of the surface roughness can be explained by increasing particle velocity at higher oxygen levels. Consequently, it can be concluded that higher oxygen but lower kerosene levels have to be favored for the HVOF spraying of submicron WC-Co powders to obtain coatings with smooth surfaces.

As reported in literature, the application of fine powders leads to an increase in layer homogeneity attributed to a better fusibility of finescaled materials and a finer dispersed phase distribution in the coating structure (Ref 25). This is typically correlated with a decrease in surface roughness (Ref 7, 8). The splat densification level and the corresponding roughness of the as-sprayed

coatings are mainly affected by the particle velocity and the molten state of the sprayed particles. The high velocity of the particles provided by the HVOF process, in combination with a better and more homogenous molten state when using finescaled WC-Co powders, helps to deform or flatten the particle upon impact. Consequently, a very fine-grained and dense coating structure composed of many densified splats is formed. In comparison to HVOF flame spraying of conventional coarse-grained WC-Co powders with a particle velocity of 450 m/s (Ref 17), the novel HVOF system, used in this study, provides fine particles with velocities to a maximum of  $931 \pm 5 \text{ m/s}$  at 1000 L/min oxygen by special K5.2 combustion chamber and nozzle design. The higher velocity may be converted into additional heat aiding the flattening of the sprayed particles (Ref 26, 27). The surface roughness can be regarded as an important indicator of the quality of thermally sprayed near-net-shape coatings. Zimmermann et al. have already reported on lower surface roughness and enhanced wear resistance of the as-sprayed coatings using agglomerated and sintered WC-Co 83/17 powders with finer WC hard materials particles ( $0.8\text{-}1 \mu\text{m}$ ) in the HVOF





spraying process (Ref 7). In addition, Landa et al. found that smooth coatings could provide a significant reduction in friction (Ref 10). This is very important for applications, where the surface quality of the coatings plays a significant role, such as calendar rolls and corrugator rolls for the paper industry (Ref 7). It can also be expected that smooth surfaces may reduce material adhesion and galling on tools in the deep-drawing process (Ref 10).

### 3.5 Coating Morphology

Polished cross-section images of the as-sprayed coatings taken by light microscope and a fracture cross-section image analyzed by SEM are shown in Fig. 8(a) and (c), respectively. The coatings feature an extremely dense, superfine structure and homogenous morphology with porosities <1%. Only very small-sized micropores on a submicron and nanosized level are detectable. The figures also show excellent interface bonding between the coating and the roughened C45 steel surface. It seems that substrate preparation using fine-grained alumina as blasting medium (106-150  $\mu\text{m}$ ) leads to an appropriate roughness profile for the subsequent coating operation in terms of the coating adhesion. Figure 8(b) shows a SEM image of the coating morphology with high magnification. It can be seen that the finescaled, spattered WC particle structure is preserved in the coating morphology, well embedded in a homogenous molten Co binder phase. The morphology consists in a primarily submicron structure with partially nanosized WC phases. It has also been found that medium kerosene levels (14 L/h), medium hydrogen levels (70-105 L/min), and oxygen flows  $\geq 700$  L/min have a positive influence on the coating morphology in terms of lower porosity and finer and more homogenous phase development. This is attributed to a more homogeneously molten state of the particles and an improved coating adhesion on the C45 steel substrate material. The results in these experiments are consistent with studies on HVOF spraying of nanocrystalline WC-18wt.%Co powders, which have been carried out by Ban and Shaw (Ref 28). They have also reported that low process temperatures and short dwell time of the powder particles passing the HVOF flame jet mainly lead to the preservation of the powder structure within the coating morphology.

## 4. Conclusion and Outlook

Feeding of submicron 88WC-12Co powders ( $-8 + 1 \mu\text{m}$ ) has been performed with Thermico CPF2 powder feeder system. A novel CJS-HVOF gun, designed by Thermico, has been used to deposit superfine structured cermet coatings. In-flight diagnostics on varying HVOF gas compositions showed a significant influence on the process dynamics. Different HVOF combustion gas compositions enable to adjust the particle velocity and temperature. In this way the deposition efficiency, the coating structure, and their macroscopic properties can be affected. Coatings with smooth surfaces ( $R_a = 2.49 \pm 0.10 \mu\text{m}$ ), extremely dense

structures (porosities <1%), and high hardness of  $1312 \pm 45$  HV 0.1 were achieved. A high process efficiency of 68% could also be realized. The deposited superfine structured coatings in this study show a high potential to be used as wear resistant, near-net-shape coatings for deep-drawing tools without any posttreatment or surface finish. It is expected that further improvements will be achieved by reducing the feedstock powder to nanoscale grain size.

## Acknowledgments

The authors gratefully acknowledge the financial support of the DFG (German Science Foundation) within the Collaborative Research Centre SFB 708.

## References

1. T.N. Rhys-Jones, Thermally Sprayed Coating Systems for Surface Protection, Clearance Control Applications in Aero Engines, *Surf. Coat. Technol.*, 1990, **43-44**, p 402-415
2. R.V. Hillery, Coatings for Performance Retention (in Military Aircraft Engines), *J. Vacuum Sci. Technol.*, 1986, **4A**(6), p 2624-2628
3. J.K.N. Murthy and B. Venkataraman, Abrasive Wear Behaviour of WC-CoCr, Cr(ind 3)C(ind 2)-20(NiCr) Deposited by HVOF and Detonation Spray Processes, *Surf. Coat. Technol.*, 2006, **8**(200), p 2642-2652
4. R. Nieminen, P. Vuoristo, K. Niemi, T. Mäntylä, and G. Barbezat, Rolling Contact Fatigue Characteristics of Thermally Sprayed WC + Co Coatings, *TS96 Thermal Spraying Conference*, Mar 6-8 1996 (Germany), DVS, 1996, p 354-359
5. P. Chivavibul, M. Watanabe, S. Kuroda, and K. Shinoda, Effects of Carbide Size, Co Content on the Microstructure and Mechanical Properties of HVOF-Sprayed WC-Co Coatings, *Surf. Coat. Technol.*, 2007, **3**(202), p 509-521
6. K. Bobzin, F. Ernst, J. Zwick, and G. Matthäus, Analysis of In-Flight Particle Properties of Thermal Sprayed Ultrafine Powders, *Mat. Sci. Eng. Technol.*, 2007, **2**(38), p 149-154 (in German)
7. S. Zimmermann, H. Keller, and G. Schwier, Improved Coating Properties by Optimized Carbide Powders for Modern HVOF Systems, *6th HVOF Spraying Colloquium*, Nov 27-28 2003 (Germany), 2003, p 31-38
8. K. Jia and T.E. Fisher, Abrasion Resistance of Nanostructured, Conventional Cemented Carbides, *Wear*, 1996, **1-2**(200), p 206-214
9. K. Jia and T.E. Fisher, Sliding Wear of Conventional, Nanostructured Cemented Carbides, 11th International Conference on Wear of Mat, *Wear*, 1997, **203-204**, p 310-318
10. J. Landa, I. Illarramendi, J.M. Montalban, A. Igartua, and G. Mendoza, Application of Nano-HVOF to Piston Rings, *11th Nordic Symposium on Tribology*, Jun 1-5 2004 (Norway), p 115-127
11. M. Ignatiev and E. Kovalev, Nanoparticles Processing for Fabrication of Multi-functional Nanostructured Coatings, *Plastic Metal Forming*, 2006, **4**(17), p 37-41
12. G. Matthäus, M. Kostecki, and O. Dau, The Fully Automatic, Computer Controlled C-CJS (Computerised Carbide Jet System) HVOF System with 25 bar Combustion-Chamber pressure by Thermico, *5th Colloquium on HVOF Flame Spraying*, Nov 16-17 2000 (Germany), GTS e.V., 2000, p 147-158
13. G. Matthäus and A. Sturgeon, Application of the HVOF Process to Internal Coatings of Cylinders, and Hard-and Software for the Internal Coating of Complex Components, *7th HVOF Spraying Colloquium*, Nov 9-10 2006 (Germany), GTS e.V., 2006, p 199-205



14. G. Matthaeus and G. Stevens, New HVOF Equipment and Technology for the Use of Superfine Powders—10  $\mu\text{m}$  and Internal Coating Applications, *6th HVOF Spraying Colloquium*, Nov 27-28 2003 (Germany), GTS e.V., 2003, p 167-176
15. M.H. Li, D. Shi, and P.D. Christofides, Modeling, Control of HVOF Thermal Spray Processing of WC-Co Coatings, *Powder Technol.*, 2005, **2-3**(156), p 177-194
16. M.H. Li and P.D. Christofides, Multi-scale Modeling, Analysis of an Industrial HVOF Thermal Spray Process, *Chem. Eng. Sci.*, 2005, **13**(60), p 3649-3669
17. H. Kreye, F. Gärtner, and H.J. Richter, High Velocity Oxy-fuel Flame Spraying-state of the Art, New Developments and Alternatives, *6th HVOF Spraying Colloquium*, Nov 27-28, 2003 (Germany), GTS e.V., 2003, p 5-17
18. E. Turunen, "Diagnostic tools for HVOF process optimization," Ph.D. Thesis, Helsinki University of Technology, 2005
19. P.H. Shipway, D.G. McCartney, and T. Sudprasert, Sliding Wear Behavior of Conventional, Nanostructured HVOF Sprayed WC-Co Coatings, *Wear*, 2005, **7-12**(259), p 820-827
20. A. Wank, A. Schwenk, B. Wielage, T. Grund, E. Friesen, and H. Pokhmurska, Untersuchungen zur Beständigkeit thermisch gespritzter Schichten bei durch Abrasion dominierter tribologischer Beanspruchung (Investigation on the Resistance of Thermally Sprayed Coatings Under Abrasion Dominated Tribologic Stress), Sep 07-08, 2006 (Germany), Werkstoffe und werkstofftechnische Anwendungen **24** (2006), p 301-306 (in German)
21. M.L. Lau, H.G. Jiang, W. Nuchter, and E.J. Lavernia, Thermal Spraying of Nanocrystalline Ni Coatings, *Phys. Stat. Sol.*, 1998, **166**, p 257-268
22. M. Magnani, P.H. Suegama, N. Espallargas, S. Dosta, C.S. Fugivara, J.M. Guilemany, and A.V. Benedetti, Influence of HVOF Parameters on the Corrosion, Wear Resistance of WC-Co Coatings Sprayed on AA7050 T7, *Surf. Coat. Technol.*, 2008, **202**, p 4746-4757
23. L. Fedrizzi, L. Valentinelli, S. Rossi, and S. Segna, Tribocorrosion Behaviour of HVOF Cermet Coatings, *Corros. Sci.*, 2007, **49**, p 2781-2799
24. V.V. Sobolev and J.M. Guilemany, Dynamic Processes During High Velocity Oxyfuel Spraying, *Int. Mater. Rev.*, 1996, **1**(41), p 13-32
25. O.P. Solonenko, *Advanced Thermophysical Fundamentals of Melt-Droplet-Substrate Interaction and its Application in Thermal Spraying*, Novosibirsk, Russia, 2003
26. H. Du, W. Hua, and J. Liu, Influence of Process Variables on the Qualities of Detonation Gun Sprayed WC-Co Coatings, *Mater. Sci. Eng.*, 2005, **1-2**(408A), p 202-210
27. R.C. Eucker, *Technologies for Films and Coatings, Developments and Applications*, Noyes Publications, 1982
28. Z.G. Ban and L.L. Shaw, Characterisation of Thermal Sprayed Nanostructured WC-Co Coatings Derived from Nanocrystalline WC-18wt.%Co Powders, *J. Therm. Spray Technol.*, 2003, **1**(12), p 112-119