

# A comparative study on thermally sprayed alumina based ceramic coatings

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Aluminium oxide is relatively cheap material, abundant almost everywhere and therefore it is widely used for thermal spray applications. Various alumina based powder containing 13 wt. Titania, two different 40 wt.% Zirconia and three different compositions of alumina-zirconia-chromia were deposited by atmospheric plasma spraying (APS) and high power plasma spraying (HPPS). The coatings obtained were evaluated by optical microscopy, microhardness measurements, X-ray diffraction and porosity measurements. Moreover, abrasion and friction wear resistance were evaluated by using Pin-on-Disc machine. Microhardness values of APS coatings are relatively high as compared to HPPS coatings except in alumina-zirconia-chromia coatings. HPPS have higher hardness values. APS coatings are much coarser and show higher porosity values than HPPS coatings. The best wear/friction behaviour exhibited coating  $\text{Al}_2\text{O}_3$ -40 wt.%  $\text{ZrO}_2$  that deposited from agglomerated and sintered powder type. © 2000 Kluwer Academic Publishers

## 1. Introduction

With the development of science and technology, materials are required to have good resistance to high temperature, wear and corrosion in many applications. The traditional metals are not able to be used at high temperatures and in corrosive environments. The excellent properties of oxide ceramics ( $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ ) like wear resistance, high temperature and chemical stability, high hardness as well as electrical isolation make those materials very challenging for many applications [1].

In the area of antiwear coatings oxide ceramics coatings ( $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{TiO}_2$ , etc.) are applied using different thermal spray processes in the form of individual layers. In many industries these coatings have become technically significant on components where wear and friction can cause critical damage in the form of abrasion, erosion and scuffing together with corrosion [2].

## 2. Experimental work

The specimens for microscopic observations ( $50 \times 20 \times 5$  mm made of steel 37) and wear tests specimens (cylindrical specimens of 14 mm diameter and 40 mm length made of steel 37) were sand blasted before spraying. Cooling was achieved with compressed air jets ( $2 \times 6$  Bar) disposed orthogonal to the substrate and moved with the spraying torch.

The as received powder used in this study were commercial powders, details of which are given in Table I.  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  powders used in this study were manufactured by spray drying in the Material Science Institute of Aachen University of Technology (Germany) [3]. Atmospheric plasma spraying was performed with Plasma-Technik PT 3000 S and Plasma-Dyne SG-100 systems with F4 burner and 6 mm nozzle diameter using argon and hydrogen as plasma gases. High power plasma spraying was performed with Plazjet gun system TAFE model 7070 using nitrogen and hydrogen as plasma gases. The APS and HPPS were performed at the Materials Science Institute of Aachen University of Technology (Germany) and the DGS at the Institute of Materials Science in Tampere University of Technology (Finland). The powders used in this study and their spraying parameters are given in Tables II–IV.

The specimens for microscopic observations, ( $50 \times 20 \times 5$  mm) were cut into smaller parts. Care was taken to ensure that the cutting wheel engaged first into the coating surface and then substrate. Otherwise a coating of less adhesion might detach from the substrate. The sectioned specimens were embedded in low viscosity, cold-hardening resin (Epofix<sup>TM</sup>). Typical mounts have a diameter 20–30 mm. Then the specimens were ground and polished on an Abramin type machine for optical microscopic research.

TABLE I Spray powder details

Powder	Commercial designation	Particle size	Production process
Al <sub>2</sub> O <sub>3</sub>	Amperit 740.0	-22.5 + 5.6	Fused
	Amperit 740.1	-45 + 22.5	Fused
	Amperit 740.8	-20 + 5	Fused
Al <sub>2</sub> O <sub>3</sub> -13 wt.% TiO <sub>2</sub>	Amperit 744.0	-22.5 + 5.6	Fused + Sintered
	Amperit 744.1	-45 + 22.5	Blended
Al <sub>2</sub> O <sub>3</sub> -40 wt.% ZrO <sub>2</sub>	Amperit 750.0	-22.5 + 5.6	Fused
	Amperit 751.1	-45 + 22.5	Agglomerated + Sintered
65Al <sub>2</sub> O <sub>3</sub> -30ZrO <sub>2</sub> -5Cr <sub>2</sub> O <sub>3</sub>	—	>45	Agglomerated
55Al <sub>2</sub> O <sub>3</sub> -30ZrO <sub>2</sub> -15Cr <sub>2</sub> O <sub>3</sub>			
40Al <sub>2</sub> O <sub>3</sub> -30ZrO <sub>2</sub> -30Cr <sub>2</sub> O <sub>3</sub>			

TABLE II HPPS (Plazjet) Spraying parameters

Sample	Powder	Current (A)	Voltage (V)	Power (kW)	N <sub>2</sub> slpm	H <sub>2</sub> slpm	Carrier slpm	Dist mm	Cooling + sub. temp.
A10	Al <sub>2</sub> O <sub>3</sub> 740.8	500	301	150	180	20	11	180	Air
H50	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub> 744.1	500	351	175	208	57	7	190	Air + CO <sub>2</sub> 250°C
AZ8 FC	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> 750.0	500	285	142.5	200	—	8	190	Air + CO <sub>2</sub>
AZ13 AS	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> 751.1	500	342	171	200	65	5	190	Air + CO <sub>2</sub>
FF11-1	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> -Cr <sub>2</sub> O <sub>3</sub> 65-30-5	500	332	166	200	80	5	190	Air + CO <sub>2</sub> 230°C
FF13-2	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> -Cr <sub>2</sub> O <sub>3</sub> 40-30-30	500	337	168.5	200	80	5	190	Air 330°C
FF14-1	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> -Cr <sub>2</sub> O <sub>3</sub> 55-30-15	500	335	167.5	200	80	5	190	Air + CO <sub>2</sub> 380°C

TABLE III APS Spraying parameters

Sample	Powder	Current (A)	Voltage (V)	Power (kW)	H <sub>2</sub> slpm	Ar slpm	Carrier slpm	Dist mm	Substrate temperature
H11	Al <sub>2</sub> O <sub>3</sub> 740.1	600	72	43	14	41	4.3	120	
S2	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> 750.0	700	60	42	6.4	41.8	3.8	120	Preheat 462 310 °C
SS2	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> 751.1	700	60	42	6.4	41.8	3.8	120	Preheat 461 270 °C
K1	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> -Cr <sub>2</sub> O <sub>3</sub> 65-30-5	700	60.5	42.4	6.4	41.8	3.8	120	177 °C
K7	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> -Cr <sub>2</sub> O <sub>3</sub> 55-30-15	700	60.5	42.4	6.4	41.8	3.8	120	Preheat 425 280 °C
K4	Al <sub>2</sub> O <sub>3</sub> -ZrO <sub>2</sub> -Cr <sub>2</sub> O <sub>3</sub> 40-30-30	700	61	42.7	6.4	41.8	3.8	120	Preheat 446 283 °C
L4	Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub> 744.1	600	62	37.2	6.4	41.8	3.5	100	Preheat 390 425 °C

An optical microscope type Axiophot, was used for the specimens evaluation. The specimens were examined and evaluated to 200 and 500 magnification. The system is coupled to a 35 mm film camera.

An interactive image analysing system, IBAS 2000 of the firm Kontron, is used to estimate the porosity in the coating. This system evaluates the prepared microscopic specimens by estimating the total surface percentage of a marked phase. This marking is done manually by the user.

The Vickers microhardness test is used for all types of coatings and has now become a routine technique [4]. To determine Vickers microhardness a Micronet 1 tester of Buehler Ltd., was used. The magnification used

was 400× for the measurements. The test forces were 25 and 300 Pounds (0.981 N) and the testing time was 12 seconds. Twenty measurements were made for each load on the cross-section of every specimen. The testing body is a 4-side diamond-pyramid which locks-in an angle 136°. On the sample a quadrangle impression is made by the plastic deformation caused by the diamond. With a video graphic system the user marks the diagonals of the quadrangle and a computer calculates the Vickers hardness.

Wear resistance was tested with Pin-on-Disk method; Pins are cylindrical specimens with 14 mm diameter and length of 40 mm. The wear is abrasively tested on 400 SiC grinding paper. The pressure is 5 N (per

sample) and 4 specimens, three coated and one uncoated, can be tested simultaneously. Every 25 m (69 sec.), the test was stopped in order to measure the weight loss of specimens, and to change SiC grinding paper. The test distance was 200 m. the diagrammatic sketch of the tester is presented in [5].

Friction tests were performed at room temperature of about 20 °C (the relative humidity of the laboratory was about 37%) using a Pin-on-Disc apparatus. The disc was made of 100Cr6 steel of 200 mm track diameter. Lubricant was Shell Tellus 1 32. Only 0.5 ml oil sprayed on the track at the beginning of each sample test. A normal load of 20 N and constant sliding velocity equal 0.5 m/s (by adapting the rotary speed of the disc to the diameter of the track) were selected. During the test the friction torque was recorded in real time by a computerised data acquisition system equipped with analogue-digital (A-D) converter. A PC 286-AT computer received the data, immediately transmitting it into hard disc. The acquisition rate was 10 Hz.

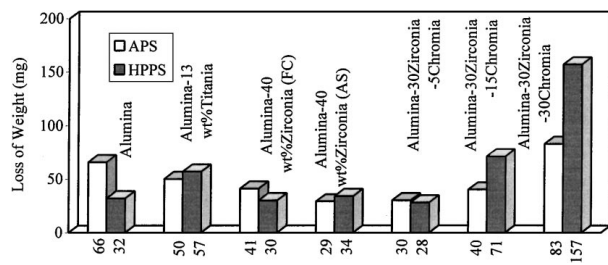


Figure 1 Comparison of wear resistance of different oxide ceramics coatings.

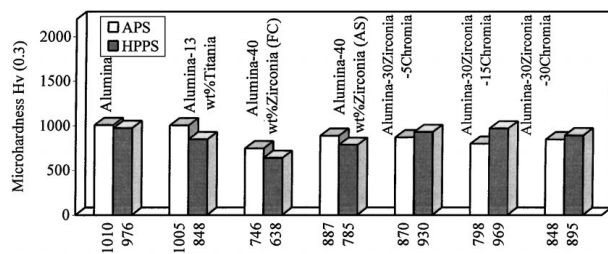


Figure 2 Comparison of microhardness of different oxide ceramics coatings.

X-ray diffractometry with CoK radiation using the Philips X-Ray Diffractometer was performed to identify phases for the deposits. The coatings and powders were examined in continuous scan mode  $20^\circ < 2 > 120^\circ$  at a rate of  $2^\circ$  per minute. The tube voltage was set to 30 kV and the current to 30 mA.

### 3. Results and discuss

Figs 1–4 show the wear resistance, microhardness, porosity and coefficient of friction values different oxide mixtures sprayed by APS and HPPS. These Figs. show that HPPS  $Al_2O_3$  coatings could have better wear resistance than APS  $Al_2O_3$  coatings. APS chromia, alumina-13wt% titania and alumina-40wt% zirconia coatings are as good as or better than HPPS coatings. Alumina-30wt% zirconia-15wt% chromia and Alumina-30wt% zirconia-30wt% chromia coatings have the lowest wear resistance at all.

Microhardness values of APS coatings are relatively high as compared to HPPS coatings except in

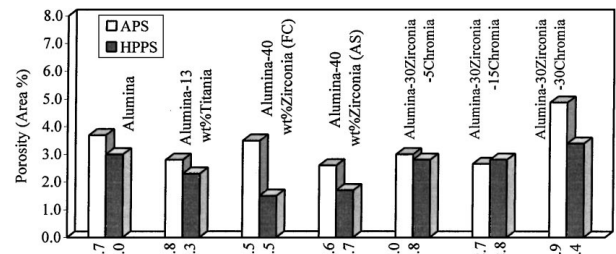


Figure 3 Comparison of porosity of different oxide ceramics coatings.

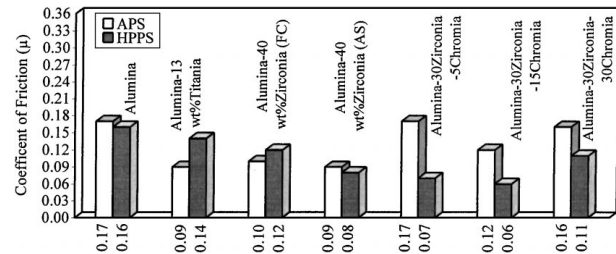


Figure 4 Comparison of coefficient of friction of different oxide ceramics coatings.

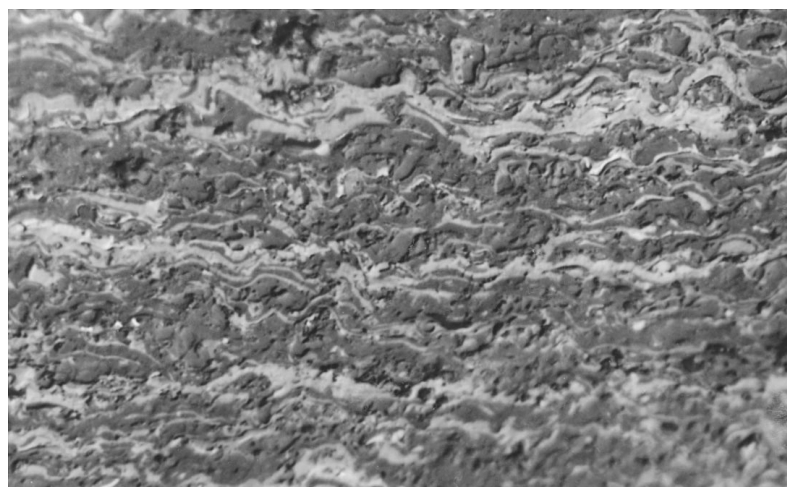


Figure 5 Cross-section of HPPS alumina-13wt.% titania coating 500x.

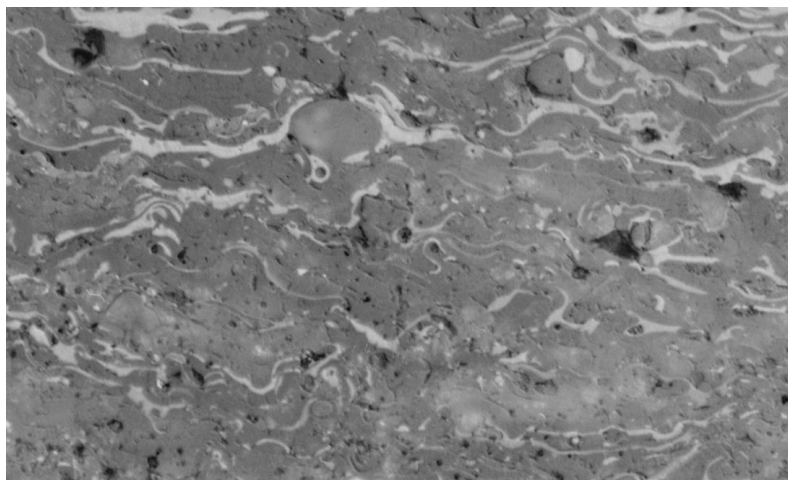


Figure 6 Cross-section of APS alumina-13wt.% titania coating 500 $\times$ .

alumina-zirconia-chromia coatings HPPS have higher hardness values. HPPS coatings show lower porosity values than APS coatings.

It is seen that the microstructures of APS (for examples Figs 5 and 6) coatings are much coarser, i.e. the splats are much thicker. A coarser structure is expected with this process since the molten particle velocities are not as high as in HPPS process.

The results show that by the addition of zirconia to alumina matrix the porosity, hardness and coefficient of friction of the coating are decreased. At the same time the wear resistance is increased. The reason for this improvement must be seen in the higher toughness of this ceramic. The influence of  $ZrO_2$  in  $Al_2O_3$  on the toughness has been shown by Claussen [6]. Also the addition of  $TiO_2$  to  $Al_2O_3$  decreases the porosity, hardness and coefficient of friction and improves the wear resistance only of APS coatings.

By comparing the studied oxide ceramic coatings, the best combined wear/friction behaviour exhibited coating  $Al_2O_3$ -40 wt%  $ZrO_2$  (agglomerated and sintered powder) as well as  $Al_2O_3$ -30 wt%  $ZrO_2$ -5 wt%  $Cr_2O_3$  (HPPS) and  $Cr_2O_3$  coatings.

#### 4. Conclusions

The results show that the wear resistance of sprayed oxide ceramic is not depending only on the hardness

of the coating but also strongly of the toughness of the material. Microhardness values of APS coatings are relatively high as compared to HPPS coatings except in alumina-zirconia-chromia coatings. HPPS have higher hardness values. APS coatings are much coarser and show higher porosity values than HPPS coatings. The best wear/friction behaviour exhibited coating  $Al_2O_3$ -40 wt%  $ZrO_2$  that deposited from agglomerated and sintered powder type.

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