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EVALUATION OF THE MECHANICAL BEHAVIOUR OF Al_2O_3 AND Al_2O_3 -TiO_2 COATINGS USING A BENCH DRILL

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RESUMEN

Las propiedades mecánicas de recubrimientos cerámicos medidas por micro-indentación Vickers y Knoop a partir de las huellas residuales fueron relacionadas con los resultados de perforación usando una broca sin rotación. Tres polvos micrométricos fueron utilizados para elaborar los recubrimientos: el primero de ellos fue de Al_2O_3 Sulzer-Metco 105 SPF™, el segundo fue de Al_2O_3 -13 % en peso de TiO_2 Saint Gobain 107™, y el último fue de Al_2O_3 -43 % en peso de TiO_2 Saint Gobain 109™. Los recubrimientos fueron elaborados por proyección térmica con llama de oxi-combustible sobre sustratos de acero AISI-SAE 1020, usando una torcha Terodyn 2000 modificada. Los ensayos de perforación fueron realizados con una broca con punta piramidal de carburo de tungsteno montada en un taladro, que aplicó lentamente una fuerza gradual hasta una carga máxima de aproximadamente 100 N y luego la fuerza fue reducida hasta cero, para obtener una curva de hystéresis (fuerza versus profundidad deindentación), siendo la velocidad de carga y descarga de 0.15 mm/s. Los resultados muestran una buena correlación entre las propiedades mecánicas medidas por ensayos de micro-indentación Knoop y los obtenidos a partir de la curva de hystéresis, permitiendo concluir que la dureza y el módulo de Young de los recubrimientos cerámicos elaborados por proyección térmica podrían ser determinados a partir de ensayos de perforación.

Palabras Clave: *Resistencia a la perforación, Recubrimientos de alúmina y titania, Comportamiento mecánico, Proyección con llama de oxi-combustible*

ABSTRACT

The mechanical properties of ceramic coatings measured by Vickers and Knoop micro-indentation from residual tracks were correlated with the results of indentation depth measured using a non-rotary drill bit. Three micrometric feedstock powders were used to manufacture coatings: the first one was Al_2O_3 Sulzer-Metco 105 SPF™, the second one was Al_2O_3 -13 wt.% TiO_2 Saint Gobain 107™ and the last one was Al_2O_3 -43 wt.% TiO_2 Saint Gobain 109™. Coatings were elaborated by oxy-fuel flame spraying process onto AISI-SAE 1020 steel substrates using a modified Terodyn 2000 torch. The drill tests were realized with a pyramidal tungsten carbide drill bit using a instrumented bench drill, which applied slowly an incremental force to a maximal load (approximately 100 N) and then the force was decreased to zero (being the load and unload speed 0.15 mm/s), in order to obtain the hysteresis graphics (Force Vs Indentation Depth). The results show a good relationship between mechanical properties measured by Knoop micro indentation tests and the force vs indentation depth ratio, obtained from hysteresis graphics, allowing to conclude that the hardness and Young's modulus of thermal sprayed ceramic coatings can be predicted from drilling tests.

Keywords: *Drilling resistance, Alumina titania coatings, Mechanical behaviour, oxy-fuel flame spraying*

1. INTRODUCTION

Drilling resistance was previously used to measure the hardness of surface heat treated steels, finding a good relationship between the results of Vickers micro-indentation and those of drilling tests [1]. Studies on alumina-titania coatings manufactured by atmospheric plasma spraying suggested a good relationship between the drilling depth obtained from rotary drill tests on the surface of the coatings and its micro-hardness Vickers, in function of the load applied and the drill bit diameter [2, 3]. The aim of this study is to continue the previous researches, by correlating mechanical properties of alumina and alumina-titania coatings measured by Vickers and Knoop micro-indentation, with the results of the indentation tests realized using a non-rotary drill bit.

2. EXPERIMENTAL SETUP

Three micrometric feedstock powders were used to manufacture coatings: the first one was Al_2O_3 Sulzer-Metco 105 SPF™, the second one was Al_2O_3 -13 wt.% TiO_2 Saint Gobain 107™ and the third one was Al_2O_3 -43 wt.% TiO_2 Saint Gobain 109™. The chemical composition and the size particle distribution of feedstock powders were determined by Wavelength-Dispersive X-Ray Fluorescence (WD-XRF) using an ARL Optim´X spectrometer and by Laser Diffraction using a Master Sizer 2000 equipment, respectively.

Coatings were elaborated by oxy-fuel flame spraying process onto AISI-SAE 1020 steel substrates using a Modified Terodyn 2000 Torch. Substrates were previously blasted using corundum particles according to the ASTM D7055-09 [4] in order to obtain an average roughness surface (R_a) higher than 5 µm, which improve the coating adherence, then they were cleaned in a sonicated alcohol bath to eliminate the blast process impurities. The oxy-fuel flame produced from 22.5 L/min of acetylene and 94.1 L/min of oxygen was used to preheat the substrates to 240 ºC and then, to spray 12 g/min of feedstock powders toward substrates located to 9 cm from nozzle torch, obtaining a thickness coating between 400 and 500 µm.

The phases in coatings were identified by X-Ray Diffraction using a Rigaku Miniflex diffractometer from the JCPDS standard card 00-029-0063 for the γ-Al_2O_3 phase and 01-070-1434 for the Al_2TiO_5. The surface of manufactured coatings was grinded and polished using abrasive papers and diamond paste (3 and 1 µm) respectively to obtain a smooth surface and then the micro-indentation and drilling tests were realized. The surface structure of coatings was observed before and after polishing using a JEOL JSM-6490LV Scanning Electron Microscope (SEM). The Vickers micro-hardness of coatings was measured from twenty indentations realized applying a 3N load during 15 seconds according to ASTM C1327-08 standard [5], while to Knoop micro-indentations a 5N load was applied during 15 seconds in order to determine the Young's Modulus. Both Vickers micro-hardness and Young's modulus were calculated from residual tracks produced using a Wilson Instrument 401MVD onto the surface of coatings.

On the other hand, the drilling tests were realized with a pyramidal tungsten carbide drill bit using the instrumented bench drill showed in Figure 1, which applied slowly an incremental force to a maximal load (approximately 100 N) and then the force was decreased to zero using a load and unload speed of 0.15 mm/s in order to obtain the hysteresis graphics (Force Vs Indentation Depth).

Figure 1. Instrumented bench drill.

3. RESULTS

3.1 Feedstock powders characterization

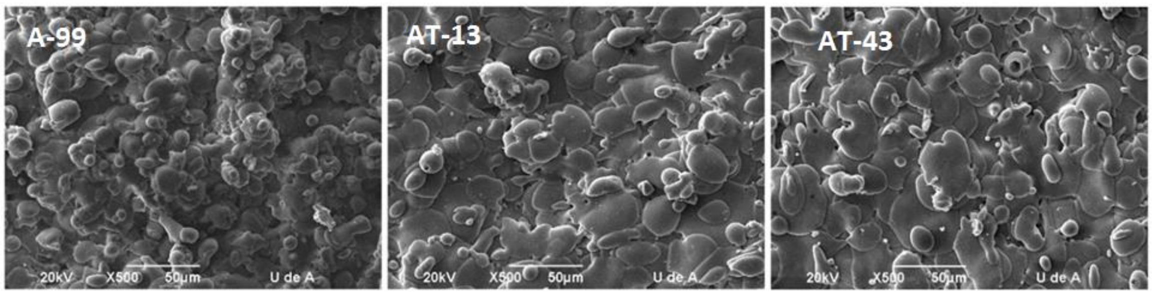
The results of chemical analysis and size particle distribution realized to feedstock powders are shown in Table 1.

Table 1. Chemical composition and size particle distribution of feedstock powders.

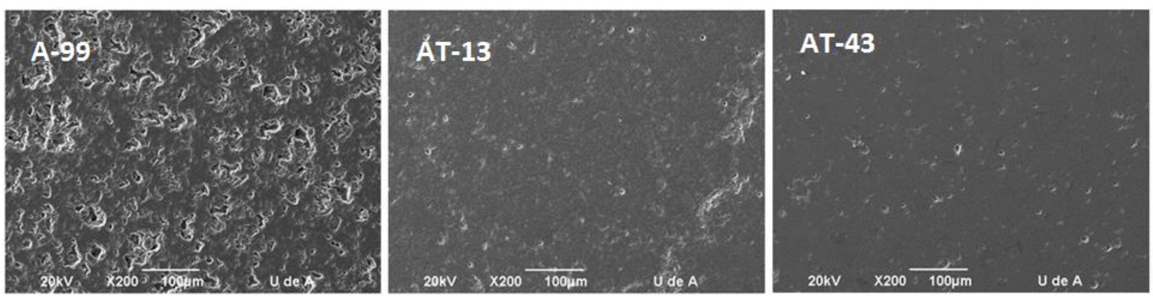
Powder	Sulzer Metco 105 SPF™	Saint-Gobain 107™	Saint-Gobain 109™
Percentage of Al_2O_3 (wt.%)	99.71 ± 0.03	84.74 ± 0.18	55.19 ± 0.25
Percentage of TiO_2 (wt.%)	0	14.32 ± 0.18	43.14 ± 0.25
Other oxide (SiO_2, Fe_2O_3)	0.29	0.94	1.67
Particle size: d(0.1) / d(0.9) [µm]	6.9 / 25.6	6.3 / 22.1	8.9 / 22.4

3.2 Coatings characterization

In order to identify the coating manufactured from Sulzer Metco 105 SPF™ powder, this was called as A-99, while those fabricated from Saint-Gobain 107™ and Saint-Gobain 109™ powders were codified as AT-13 and AT-43 respectively, according to their chemical composition. On the other hand, the surface structure of manufactured coatings is shown in Figure 2.



(a)



(b)

Figure 2. Structure surface of coatings. (a) Before polishing. (b) After polishing.

Despite of the similar distribution size particles of the three feedstock powders used to manufacture the coatings, the splats on the as-sprayed surface of A-99 sample are less flattened than those on AT-13 and AT-43 coatings, the splats being more flattened on AT-43 sample, as it is shown in the last figure. This is due to the higher melting temperature of Al_2O_3 particles (2050 °C) used to manufacture the A-99 coating, while it decreases as the TiO_2 percentage in the powder increases (the melting point of Al_2O_3-13 wt.% TiO_2 and Al_2O_3-43 wt.% TiO_2 used to fabricate the AT-13 and AT-43 coatings are 1980 °C and 1860 °C, respectively). The lowest flattening of splat reduces the stacking up in the structure of coatings, which explains the higher surface porosity of A-99 coatings after polishing and the decreasing of this porosity as the TiO_2 content in the coatings increases.

The phase analysis revealed only the γ-Al_2O_3 phase in A-99 and AT-13 coatings. TiO_2 was not observed, probably because Ti was solubilized in the γ-Al_2O_3 phase [6-8]. Al_2TiO_5 was observed in AT-43 coating. All those observations are in accordance with previous studies [6, 7]. The Table 2 summarizes the results of the Vickers and Knoop indentations realized in order to calculate the micro hardness and Young's modulus of coatings. According to the results of Vickers tests, A-99 coating is less hard than AT-13 coating, in spite of the presence of alumina which is the harder phase identify in the coatings studied. This is due to its higher porosity, which

decreases the hardness of those coatings [9]. Meanwhile, AT-43 coating is the softer due to the presence of the Al_2TiO_5 phase, which has a hardness of 7 GPa, against 14 GPa for the γ-Al_2O_3 and 11 GPa for the rutile [9].
On the other hand, the results of Knoop tests indicate that A-99 coating is the harder. Indeed, Knoop micro-indentations are more superficial than Vickers micro-indentations, so that the results are less affected by the difference of porosity in the coatings.

Table 2. Mechanical properties of the coatings

Coating	A-99	AT-13	AT-43
HV_{3N} (GPa)	10.0 ± 2	12.4 ± 1	6.95 ± 0.3
HK_{5N} (GPa)	10.3 ± 0.9	8.8 ± 0.4	4.7 ± 0.2
Young's modulus (GPa)	168.7 ± 23.9	183.8 ± 38.5	94.7 ± 15.7

Figure 3 presents the drilling tests results. A good reproducibility of the results can be observed. As the Oliver and Pharr study [11], the indentation depth varies according to the hardness of the coating. The ratio of the maximal load P to the square of maximal indentation depth h^2 (average of three tests) is presented for each sample in the Table 3. It can be observed a good correspondence between the hardness Knoop of coatings and the P/h^2 values obtained from the drilling tests, which indicate that to the load applied in these tests the contact produced between the coating surface and the drill tip is shallow as the Knoop indentation.

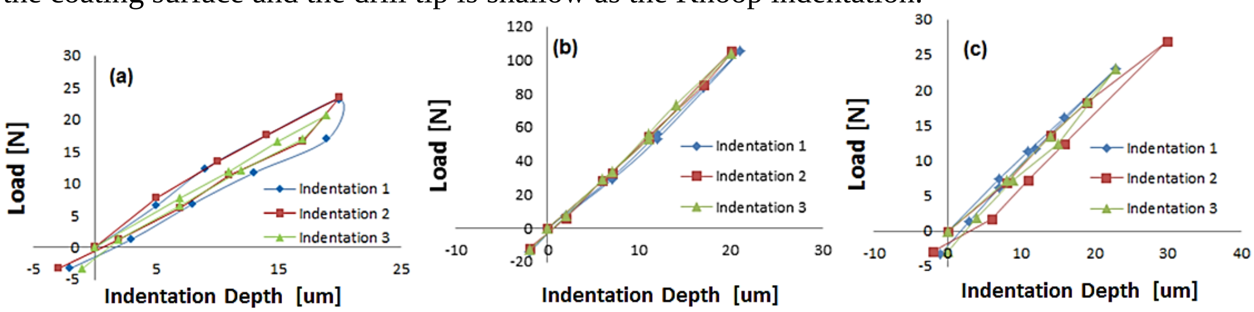


Figure 3. Indentation depth in function of the load applied on the coatings. (a) A-99. (b) AT-13. (c) AT-43.

The Young's modulus was predicted proportional to the slope of the last part of the curve corresponding to the load (80 N to 100N), recorded Δ afterwards. This slope Δ (average of three tests) is presented in the Table 3, for each sample. According to these results, AT-13 coatings should be the most rigid, which corresponds to the Knoop tests results.

Table 3. Ratio of the load applied to the square of indentation depth and the slope of the last part of the curve corresponding to the load (80 N to 100N) **Δ**.

Coating	A-99	AT-13	AT-43
$\frac{P}{h^2}$ [N/μm^2]	0.26 ± 0.0	0.25 ± 0.01	0.17 ± 0.01
Δ	4.4 ± 0.3	5.7 ± 0.9	4.3 ± 0.8

4. CONCLUSIONS

Coatings of Al_2O_3, Al_2O_3-13 wt.% TiO_2 and Al_2O_3-45 wt.% TiO_2 were elaborated by oxy-fuel flame spraying. They were characterized onto their surface by Vickers and Knoop micro-indentations in order to correlate the results of these tests with those of drilling tests performed onto the surface. The results show a good relationship between mechanical properties measured by Knoop micro indentation tests and those obtained from drilling tests, concluding that the hardness and Young's modulus of thermal sprayed ceramic coatings can be predicted from drilling tests using a non-rotary drill bit.

5. ACKNOWLEDGMENTS

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