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*Article*

# Investigations of microstructure and selected mechanical properties of $Al_2O_3$ + 40 wt.% $TiO_2$ coatings deposited by Atmospheric Plasma Spraying (APS)

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**Abstract:** Atmospheric Plasma Spraying (APS) enables deposition of coatings from different materials, including those based on $Al_2O_3$ and $TiO_2$. In this work, $Al_2O_3$ + 40 wt.% $TiO_2$ coatings were tested. The relationships between mechanical properties, microstructure and spraying parameters (namely: spraying distance and torch scan velocity) were investigated. Commercial -45 + 5 μm powders in agglomerated as-produced state were sprayed onto the stainless steel 1.4301 substrates. The aim of the study was to determine the adhesion, microhardness and roughness of coatings but also to characterize their microstructure. It was observed that coatings sprayed from shorter distance were well melted and revealed good adhesion, but at the same time they were more porous and of lower microhardness than those deposited from the longer spraying distance.

**Keywords:** plasma spraying; coating; microstructure; porosity; hardness; roughness; adhesion

## Introduction

Among the various methods of producing coatings, plasma spraying, APS (Atmospheric Plasma Spraying) is one of the techniques of surface engineering. In this process, the powder particles in the plasma stream are melted and accelerated, and then they hit the ground and solidify very quickly ($10^5 \div 10^6$ K/s) [1,2]. The joining mechanism occurs as a result of intensive deformation of the molten material in contact with the substrate [3,4]. The quality of the obtained coating, including its adhesion, is influenced by many parameters, including type and preparation of the substrate, sprayed material, preheating the substrate before spraying, as well as process parameters [5,6]. However, there is still little information in the literature regarding the relationship between coating adhesion and process parameters, type of substrate and applied material. Therefore, the article focuses on examining the relationship between spraying parameters and adhesion, microhardness, roughness and morphology, on the example of $Al_2O_3$ + 40 wt.% $TiO_2$ coatings.

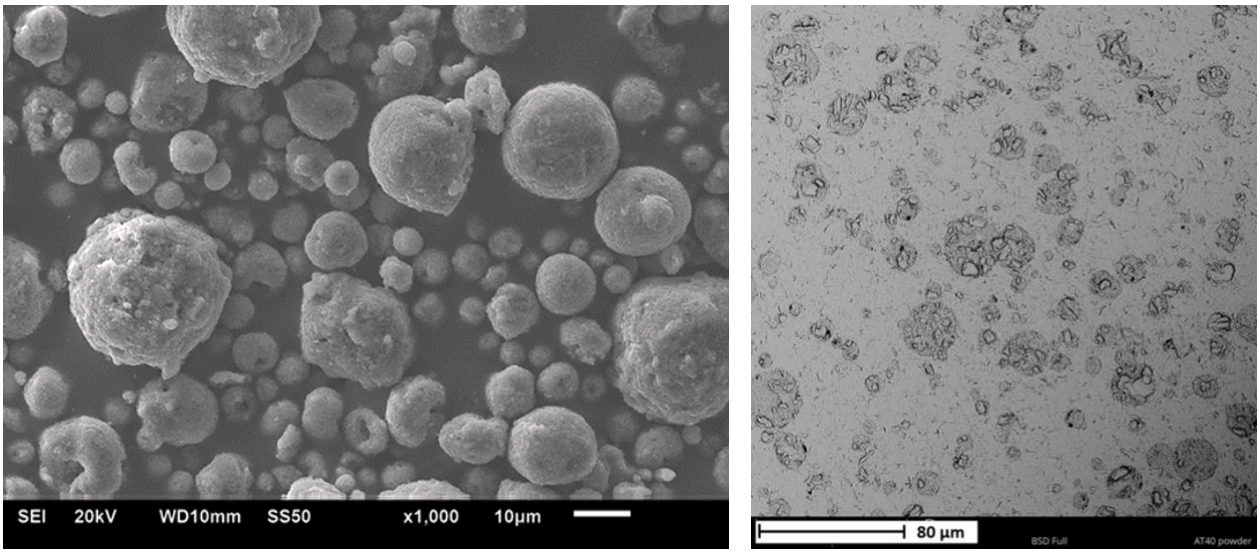
Coatings based on $Al_2O_3$ + 40 wt.% $TiO_2$ are characterized by an excellent combination of properties such as: resistance to brittle fracture, wear resistance, corrosion resistance and relatively high fatigue strength [7÷9]. These coatings are used, among others as an equipment for the production of textiles, pump and compressor components, exhaust shaft sleeves and mechanical seals as well as electron emitters [3,10÷13].

## Materials and methods

$Al_2O_3$ + 40 wt.% $TiO_2$ (Metco 131VF, Oerlikon Metco, Germany) with a particle size of -45+5 μm was used as the coating material for the spraying process. SEM pictures (Fig. 1) showed agglomerated powder morphology, according to the data declared by the producer [10].

The spraying process was carried out using an SG-100 gun (one cathode and one anode). The flow of plasma-forming gases was: 45 slpm for Ar and 5 slpm for $H_2$. The gun was mounted on the arm of a 6-axis robot, while the substrates were mounted in a holder in the welding rotor. The spraying process was designed assuming a variable spraying distance and torch scan speed. Process parameters are presented in table I.

The powder was introduced radially with a flow rate of 20 g/min. Before spraying, the powder material was dried at 120 °C for 2 hours to ensure stable powder feed to the plasma torch and to avoid problems associated with clogging during feeding. The coatings were applied to a substrate made of 1.4301 austenitic stainless steel, 2 mm thick and 25 mm in diameter. Prior to spraying, the substrates were grit blasted (using F40 corundum according to the FEPA standard) and cleaned with ethanol.




(a) (b)

**Fig. 1**. SEM image of the $Al_2O_3$ + 40 wt.% TiO2 powder in delivery state: a) top view, b) cross-section

**Table I.** Parameters of the APS process

| Sample's designation | Spraying distance, mm | Torch scan velocity, mm/s | Power, kW |
|---|---|---|---|
| AT40-1 | 80 | 500 | |
| AT40-2 | 90 | 400 | 35 |
| AT40-3 | 100 | 500 | |

Microscopic observations of the coatings were made on JEOL JSM-6610A and Phenom G2 Pro microscopes. Based on microscopic images, the porosity and thickness of the coatings were estimated. The porosity assessment was carried out in accordance with ASTM E2109-01 (Standard Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings, 2014), based on digital image analysis in ImageJ. Porosity was assessed at 1000x magnification (results averaged based on measurements of 20 images), while thickness – at 500x magnification (results averaged for 5 measurements). Coating roughness was measured using a MarSurf PS10 profilometer (Mahr, Germany) – Ra, Rz and Rt parameters were determined. Three measurements were made for each coating, and then the mean value and standard deviation were determined.

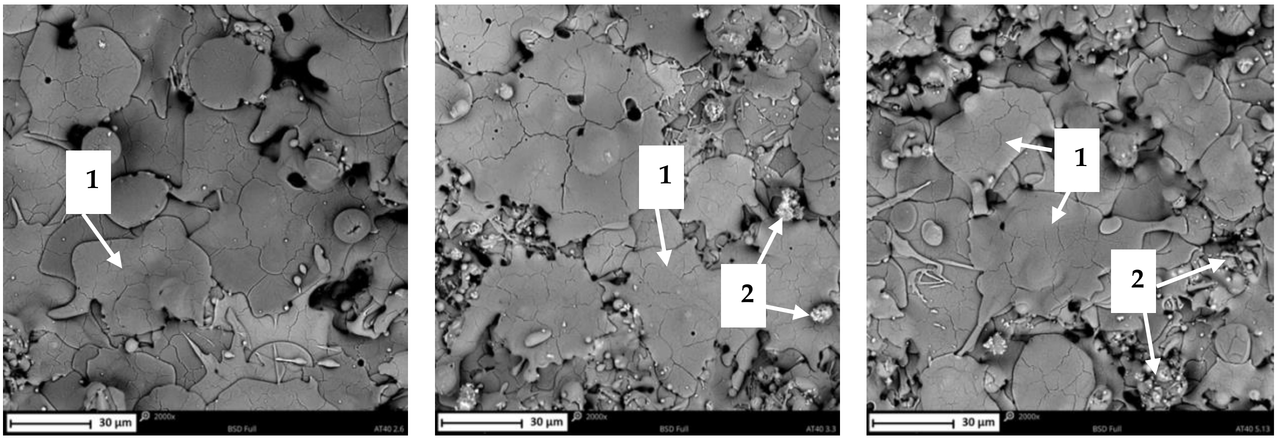
The microhardness of coatings was measured with a Vickers indenter at a given load of 1.96 N (HV0.2), using a HV-1000 hardness tester (Sinowon, China), in accordance with PN-EN ISO 4516: 2004 (Metal and other inorganic coatings. Vickers and Knoop's microhardness tests). 10 measurements were made for each sample.

Determination of coating adhesion was carried out in accordance with PN-EN ISO 14916: 2017-05 (Thermal spraying. Determination of adhesion by peeling). Distal Classic cold-setting adhesive with an average strength of 50 MPa was used for sample preparation.

## Results and discussion

### *Morphology, microstructure and microhardness*

SEM photos of sprayed coatings are shown in figure 2 and figure 3. The morphology of the coatings depended on the spraying parameters. Coatings sprayed from a distance of 80 mm showed the best degree of melting the particles (Fig. 2a), in contrast to the coating sprayed from a distance of 100 mm, where there were areas with incomplete melting of the powder (Fig. 2c). This indicates that the increased spraying distance does not provide enough heat to completely melt the powder. The torch scan speed, in turn, had a significant impact on the number of cracks in the coating – the coating sprayed at a torch scan velocity of 400 m/s (Fig. 2b) showed more cracks than at a torch scan velocity of 500 m/s (Fig. 2a and Fig. 2c). Therefore, a larger amount of heat delivered caused higher stresses in the coating, which resulted in the formation of the mentioned cracks.

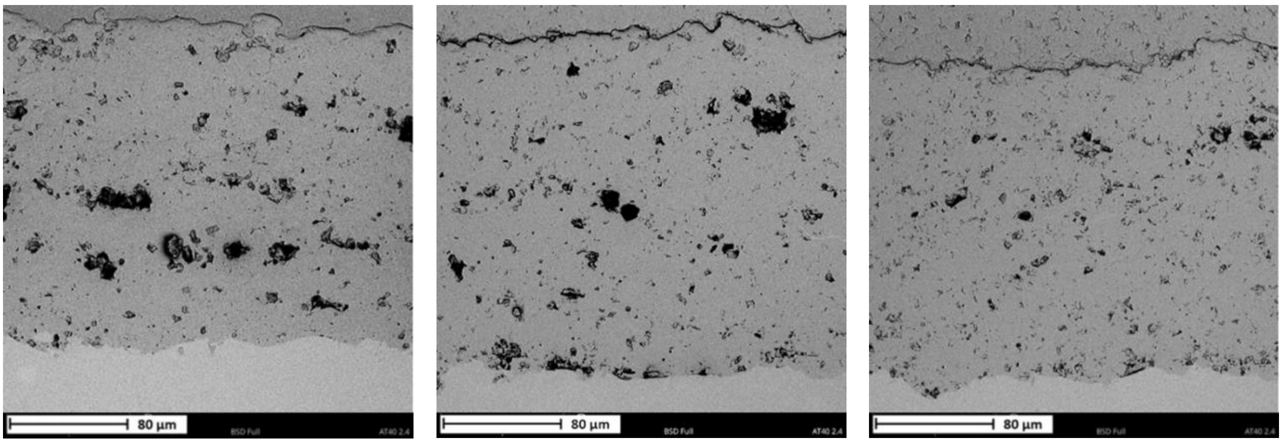



(a) (b) (c)

**Fig. 2.** SEM images of the coatings: a) AT40-1, b) AT40-2, c) AT40-3; 1 – fully melted particles, 2 – non-melted powder particles

All coatings had a lamellar structure, with pores and cracks (Fig. 3) - which is representative of the powder plasma spray method [4÷6]. In each case, on the border between the substrate and the coating, the coherence of the connection was visible – the rough surface of the substrate (previously subjected to grit blasting) was well bonded to the coating material. The thicknesses of all sprayed coatings were 150÷200 µm. Coatings were applied at 8 passes; the average thickness sprayed in one pass was 19÷25 µm. Microscopic images confirmed that the use of powder in the agglomerated delivery state ensured coating homogeneity [8,11].

The porosity values of the coatings (Table II) showed that the spraying distance had a significant impact on the obtained microstructure. The coating applied from the shortest distance had the highest porosity of approx. 13 vol.% (Fig. 3a), while sprayed coatings from 90 mm and 100 mm (Fig. 3b and Fig. 3c) showed a comparable porosity of 11 vol.%. These results are consistent with the porosity obtained in [8], where for the $Al_2O_3$ + 40 wt.% $TiO_2$ coating with a thickness of approx. 200 µm porosity value ranged from 10.5 to 11.6 vol.%. Also, the authors of article [2] obtained the porosity results of $Al_2O_3$ + 40 wt.% $TiO_2$ plasma sprayed coatings at a level of about 12%.




(a) (b) (c)

**Fig. 3.** SEM images of the cross-sections of coatings: a) AT40-1, b) AT40-2, c) AT40-3

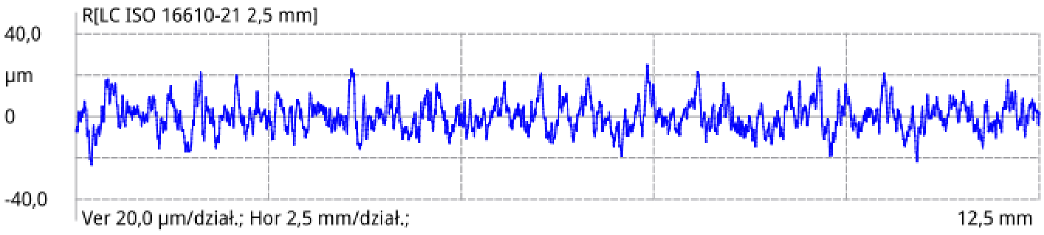
Microhardness values of the coatings are presented in table II. The AT40-1 sample showed lower microhardness (931 HV0.2) than coatings sprayed at 90 mm and 100 mm (1018 respectively and 1002 HV0.2). A smaller value of the standard deviation for AT40-2 and AT40-3 samples is also noticeable, which proves that the coatings are more homogeneous. All microhardness values are comparable to those presented in the literature – average microhardness of $Al_2O_3$ coatings + 40% by weight. $TiO_2$ was estimated at the levels: 910 HV0.1 [7], 797÷852 HV0.3 [8].

**Table II.** Comparison of porosity and microhardness of the sprayed coatings

| Sample's designation | Porosity, % | Microhardness HV0.2 |
|---|---|---|
| AT40-1 | 12.9 ± 0.9 | 931 ± 111 |
| AT40-2 | 11.7 ± 1.1 | 1018 ± 70 |
| AT40-3 | 11.2 ± 0.8 | 1002 ± 70 |

### *Roughness*

Roughness values were comparable for all coatings. An example of a roughness profile is shown in figure 4. The average arithmetic deviation of the profile from the mean Ra line was from 5.5 to 6.5 μm. The total height of the Rt profile was in the range of 45÷55 μm. These results are comparable with those obtained earlier by the authors for coatings with different content of $Al_2O_3$ and $TiO_2$ [6.13]. The roughness of the substrate after grit blasting was lower: Ra = 3.0 μm and Rt = 25.8 μm.




| R[LC ISO 16610-21 2,5 mm] | | |
|---|---|---|
| Ra | 5,786 | µm |
| Rz | 42,445 | µm |
| Rt | 48,350 | µm |

**Fig. 4.** An example of the AT40-2 coating roughness profile

### *Adhesion*

The largest differences in the results for sprayed samples with different parameters were observed in the case of coating adhesion measurements (Fig. 5). All fractures were adhesive. The highest adhesion (almost 27 MPa) was obtained for the coating sprayed from the closest distance of 80 mm, with the best degree of particles melting. The lowest value, in turn, was obtained for the coating sprayed from the farthest distance of 100 mm, in which the local lack of melting of powder particles was observed.

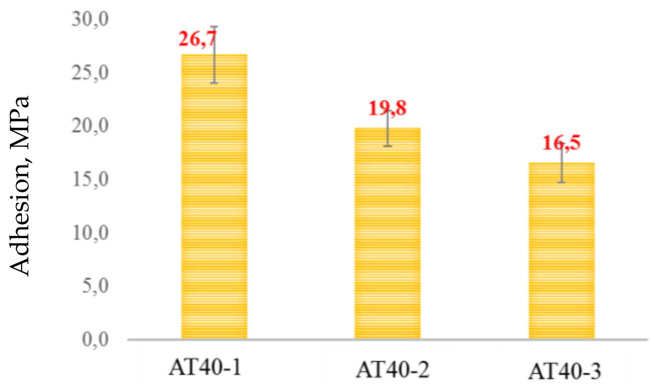



**Fig. 5.** Adhesion values of $Al_2O_3$ + 40 wt.% $TiO_2$ coatings

## Conclusions

The article examined the properties of $Al_2O_3$ + 40 wt.% $TiO_2$ applied by plasma spraying (APS). The morphology, microstructure, microhardness, roughness and coating adhesion were characterized. It has been shown that:

- change of spraying parameters – spraying distance and torch scan velocity relative to the substrate – resulted in different morphology, microstructure and coating properties. Coatings sprayed from a shorter distance of 80 mm were characterized by a better degree of powder particles melting and better adhesion, but at the same time higher porosity and lower microhardness. The coatings applied from the longest distance of 100 mm were characterized by inferior powder melting and lower adhesion, but they were less porous and had higher microhardness;

- all coatings had cracks, pores and voids, which is typical for coatings applied by the APS method. The intensity of the cracks depended on the torch scan velocity – slower movement of the torch resulted in more cracks in the coating;
- all samples showed similar roughness values. The roughness of the surface to which the coatings were applied was twice lower compared to the roughness of the coating.

Further research anticipates the comparison of properties of powder and suspension coatings based on $Al_2O_3$ + 40 wt.% $TiO_2$.

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