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Article

The microstructure and selected mechanical properties of Al_2O_3 + 3 wt.% TiO_2 plasma sprayed coatings

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Abstract: The Al_2O_3+TiO_2 coatings are of the interest of surface engineering due to their high hardness and wear resistance but also increased toughness, when compared to pure Al_2O_3 ones. This article describes the deposition of Al_2O_3+3 wt.% TiO_2 coatings by Atmospheric Plasma Spraying (APS) technique. The commercial AMI 6300.1 powder (-45 + 22 μm) was used as a feedstock. The 2^k+1 spraying experiment, based on two variables, namely spray distance and torch velocity, was designed. The samples were characterized in the terms of morphology, microstructure, microhardness and roughness. It was observed that the shorter spray distance resulted in lower porosity, higher microhardness and lower roughness of coatings.

Keywords: plasma spraying; coating; microstructure; porosity; microhardness; roughness

Introduction

The surface of engineering element plays often a key role, affecting its operation reliability and service time. Coatings technology develops dynamically, especially over the last 30 years, which is caused by the implementation of technologically advanced spraying devices, as well by further understanding of the nature of the spraying process itself [1]. Thermal spraying global market (revenue from materials, equipment and coatings manufacturing) was assessed at USD 7.58 billion in 2015 and is expected to reach USD 11.89 billion by 2021 [2].

Nowadays, thermally sprayed ceramic coatings have gained a wide range of applications. One of the most extensively studied material combination for spraying is Al_2O_3 and TiO_2. Due to their high hardness and wear resistance, Al_2O_3+TiO_2 based coatings are applied e.g. in butterfly valves in hydraulic systems and textile industry tools [3,4,7,8].

The powders of Al_2O_3+TiO_2 are commercially available only with some specific chemical compositions, including: Al_2O_3+3 wt.% TiO_2, Al_2O_3+13 wt.% TiO_2 and Al_2O_3+40 wt.% TiO_2 [3,5,6]. This work focuses on Al_2O_3+3 wt.% TiO_2 (AT3) coatings obtained by Atmospheric Plasma Spraying (APS). An addition of TiO_2 causes that the coatings are less brittle than those produced by pure alumina [3]. The aim was to deposit several AT3 coatings, according to the 2^k+1 Design of Experiment (DoE) and analyze the influence of spray process parameters on coating morphology, microstructure, microhardness and roughness [9].

Materials and Methods

A commercially available powder (AMI 6300.1) of Al_2O_3+3 wt.% TiO_2 was used as the feedstock for spraying. SEM images (JEOL JSM-6610A, Jeol, Japan) showed blocky, angular morphology of powder, as it was delivered in crushed and blended form (Fig. 1). The nominal powder particle size range was equal to -45+22 μm, according to the powder datasheet [10].

Powder Plasma Spraying (Fig. 2) was carried out at Plasma Spraying Laboratory at Wrocław University of Science and Technology. During the process, particles of micrometer size are heated in plasma jet and are moved towards the substrate with high velocity, together with plasma gases [11].

Spraying experiments were carried out with the use of one cathode – one anode plasma torch SG-100 (Fig. 3 and Fig. 4), at constant electric power of 35 kW. The feed rates of plasma gases were following: Ar – 45 slpm and H_2 – 5 slpm. The torch was mounted on 6-axis robot, whilst the substrates were fixed on rotating carousel. 2^k+1 design of experiment included the following variables: spray distance and relative velocity between torch and substrates. The deposition variables are given in table I.

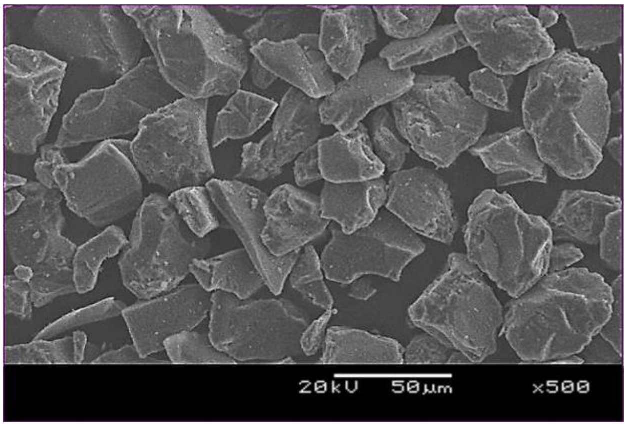


Fig. 1. SEM micrograph of Al_2O_3 + 3 wt% TiO_2 powder in the delivery condition

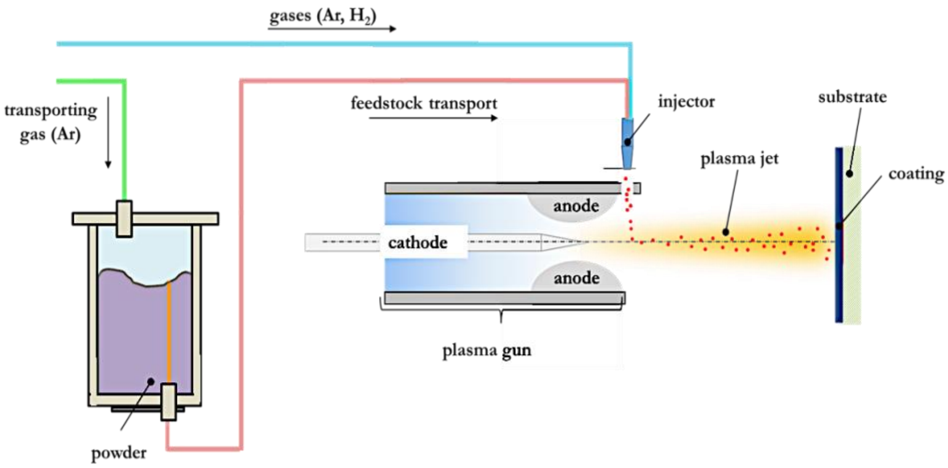


Fig. 2. Scheme of Atmospheric Plasma Spraying (APS), inspired by [12]

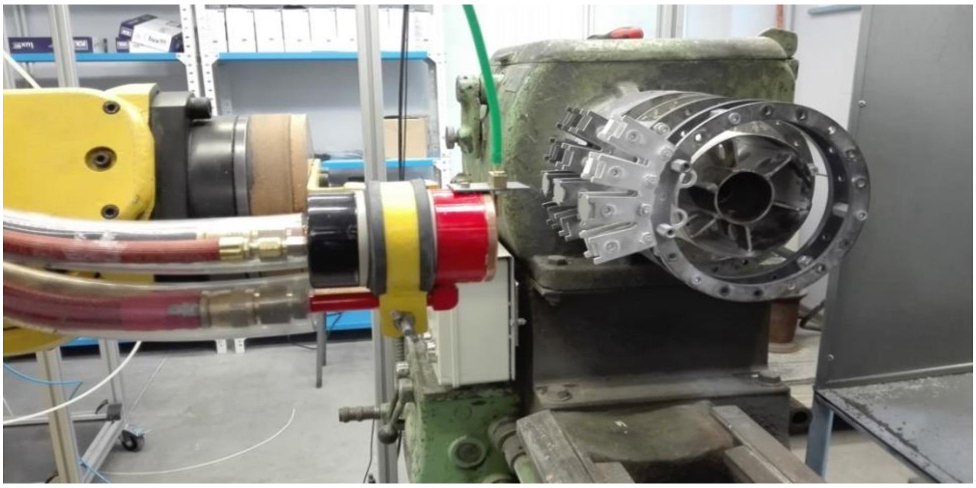


Fig. 3. SG-100 plasma torch and rotating carousel

Table I. The process parameters

Labels	Spray distance, mm	Spray velocity, mm/s	Electric power, kW
AT3-1	80	300	35
AT3-2		500	
AT3-3	90	400	
AT3-4	100	300	
AT3-5		500	

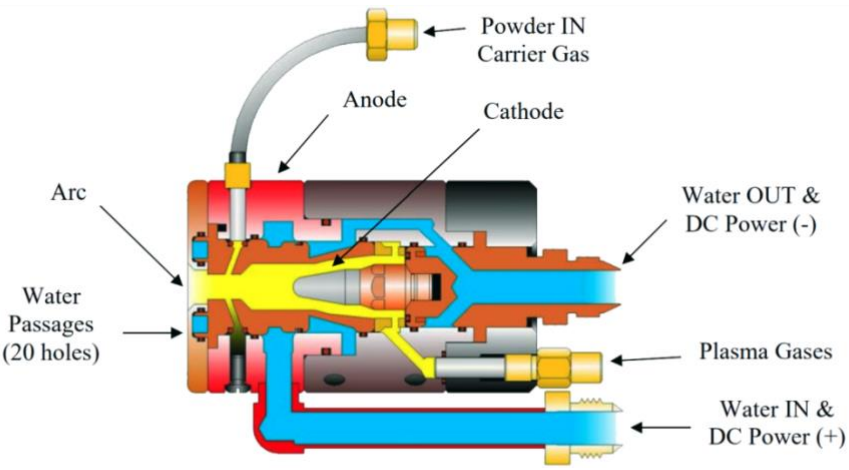


Fig. 4. Scheme of SG-100 plasma torch with internal injection [13]

The powder was injected radially into the plasma jet with the feed rate of 20 g/min. Prior spraying powder was dried at 120 °C for 2 hours, in order to avoid clogging during powder feeding. Coatings were deposited on stainless steel X5CrNi18-10 coupons, having thickness of 2 mm and diameter of 25 mm. Prior the spraying, substrates were grit blasted by corundum (F40 according to FEPA standard) and cleaned with ethanol.

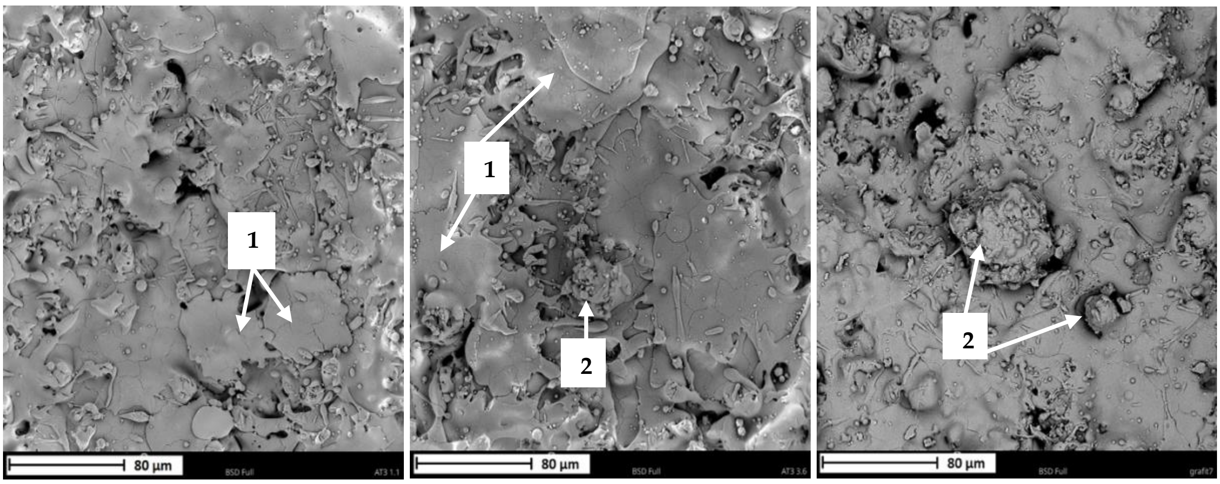
Coatings' free surfaces and cross-sections were observed by using Phenom G2 Pro (Phenom-World, the Netherlands). Based on those images, porosity and thickness of coatings were evaluated. Porosity was calculated according to ASTM E2109-01 standard [14], with the use of ImageJ open source software. The porosity was assessed at 1000x magnification (average taken from 20 images), whilst thickness – at 500x magnification (average from 5 measurements). The roughness of the coatings was measured by MarSurf (PS 10, Mahr, Germany) profilometer and mean Ra, Rz, Rt were determined. Three measurements were made for each coating, then the average values and standard deviations were calculated.

The microhardness of powder and coatings was measured with Vickers penetrator under the load of 1.96 N (HV0.2) using the HV-1000 hardness tester (Sinowon, China). Ten imprints at each case were made.

Results and discussion

Morphology and microstructure

The SEM images of selected coatings are collected in figure 5 (top surfaces) and figure 6 (cross sections). It can be noticed that the morphology of deposited coatings is dependent on spray process parameters. The shortest spray distance of 80 mm results in coatings with well molten lamellas in their top surfaces (labelled with arrow in Fig. 5a). On the contrary, the specimens sprayed from 90 and 100 mm, have less homogeneous top surfaces – not fully melted particles are observed (Fig. 5b and Fig. 5c). This suggests that some of powder particles reach the substrate in semi-molten state.



(a) (b) (c)

Fig. 5. Free surfaces of produced AT3 coatings: a) AT3-1, b) AT3-3, c) AT3-5. 1 – well molten splats, 2 – non melted particles

All coatings exhibit typical lamellar structure, which is characteristic for the plasma spraying [15]. As observed at the cross-sections, coatings (Fig. 6) reveal cracks, pores and voids, typical for APS process. In each case, substrate-coating interface is well adhered and do not show any discontinuities. The rough free surface of substrate (resulted from grit blasting) is well bonded with the lamellas. All coatings are of the thickness above 200 μm (Fig. 7). Each coating was sprayed with 8 passes; this means that thickness per pass is in the range of 25 to 35 μm.

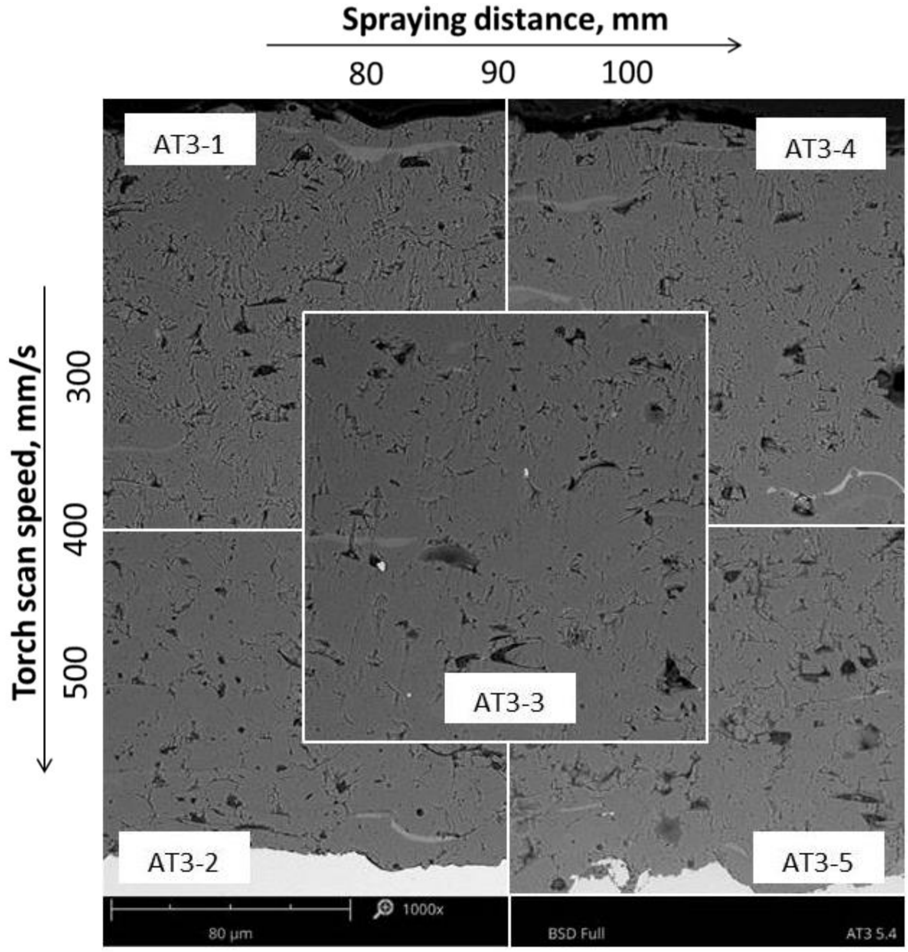


Fig. 6. The relationship between spraying variables and cross-section of AT3 coatings

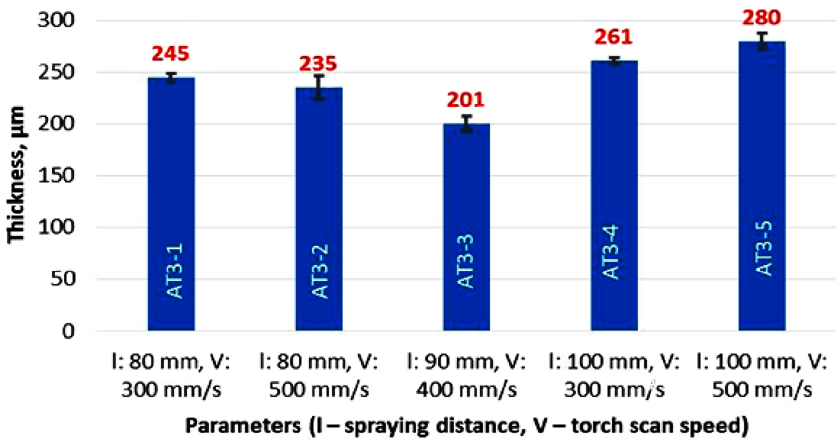


Fig. 7. The thickness of AT3 coatings

Taking into account the porosity, the correlation between spraying parameters and volume porosity is observed (Fig. 8). Longer spray distance (so the "coldest" spraying conditions) results in higher porosity (9.2 vol.% and 10.8 vol.% for samples AT3-4 and AT3-5 respectively). The lowest porosity is observed for shorter spraying distance of 80 mm (8.0 vol.% and 8.6 vol.% for samples AT3-1 and AT3-2). The porosity values are in accordance with [15], where Al_2O_3+3 wt.% plasma sprayed coatings were obtained with the porosity in the range of 6÷8%.

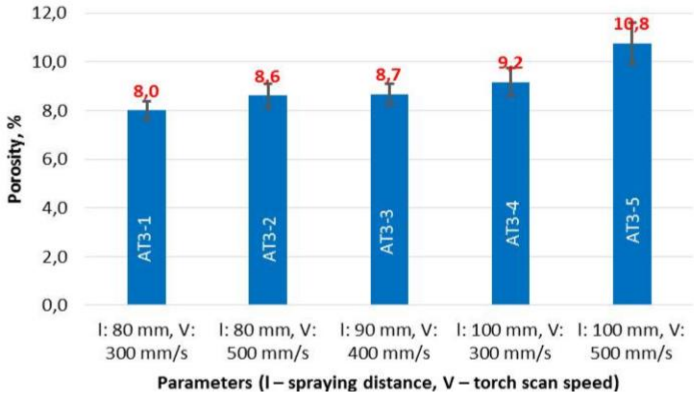


Fig. 8. The mean porosity values of AT3 coatings

Roughness

The roughness values (Ra, Rz, Rt) are collected in figure 9. According to the results, all parameters are about two times greater than those of substrate material, prepared by grit blasting. Nevertheless, such values are typical for APS spraying. Higher roughness is evaluated for coatings sprayed at longer spray distance – what is also typical for conventional thermal spraying.

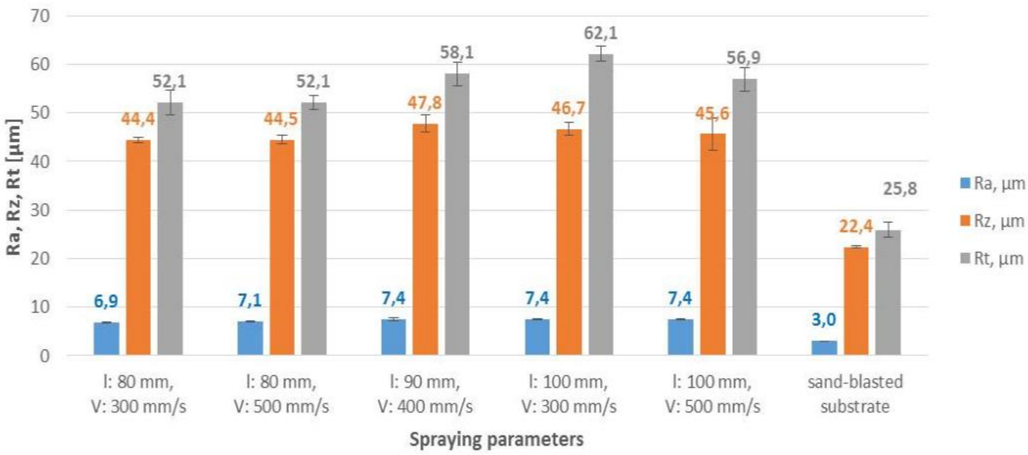


Fig. 9. The comparison of the roughness values of AT3 coatings

Hardness

Finally, Vickers hardness of coatings was investigated (Fig. 10). The temperature-related spraying conditions and porosity have a great influence on microhardness values. Coatings of higher porosity (those sprayed from longer distance AT3-4 and AT3-5), are of lower hardness. The highest microhardness of almost 800 HV0.2 is observed for AT3-1 coating, which was sprayed with the lowest spray distance and lowest torch velocity. The results are consistent with work [16], where the stand-off distance of 90 mm provided sufficient time for proper melting of powder particle, resulting in high hardness of Al_2O_3+3 wt.% TiO_2 coating. It is believed that full melting of powder helps in formulation of homogeneous coatings and increases its hardness.

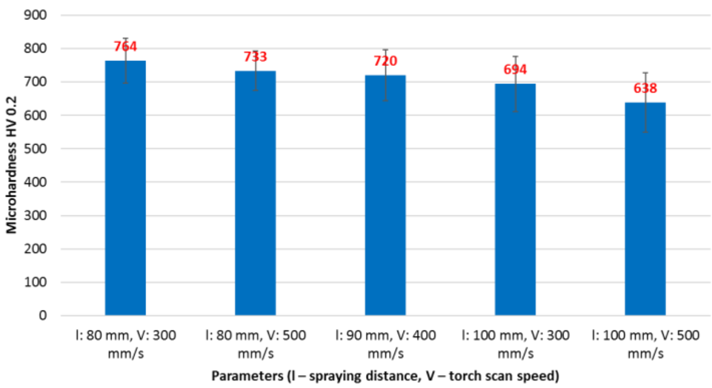


Fig. 10. Hardness HV0.2 of AT3 coatings

Discussion

According to the microstructure of the sprayed coatings, the addition of TiO_2 to the Al_2O_3 caused the melting temperature reduction [17]. Depend on process parameters, the heat flux, which is taken to melt the particles could be not sufficient. It could result in sintering of melted TiO_2, which caused the porosity and worse connection between particles. This phenomenon is related to the aggregation Al_2O_3 particles in the liquid phase [15,18].

The microhardness values are quite typical [16,18]. Moreover, the own results of authors from plasma sprayed Al_2O_3 coatings (850÷900 HV0.2) confirmed that the addition of TiO_2 reduce the microhardness value. What could be interested, coatings which contain unmelted particles have higher microhardness because of greater amount of α-Al_2O_3, than γ-Al_2O_3 [19]. A detailed XRD analysis should be performed to prove it.

The influence of linear torch speed on mechanical properties and microstructure is less than spray distance. Probably the electric power and plasma enthalpy are more important factors [20].

Conclusions

In this article Al_2O_3+3 wt.% TiO_2 coatings were produced by Atmospheric Plasma Spraying (APS). Afterwards, samples were characterized for their morphology, microstructure, roughness and microhardness. It can be summarized that:

- The change of spraying parameters, such as spraying distance and velocity, resulted in different morphology, microstructure and properties of coatings. The shorter spray distance resulted in lower porosity, well melted particles and higher microhardness of the coatings. On the contrary, deposits sprayed from the longer distance were of higher porosity, lower microhardness and revealed non melted powder particles;
- At the same time, all coatings revealed cracks, pores and voids, typical for APS. Their number was dependent on spray distance;
- Roughness of the samples was representative for APS spraying. All measured surface topography parameters of coatings were two times greater when comparing them to the substrate material.

The future studies will focus on the influence of TiO_2 content on Al_2O_3+TiO_2 coatings and more detailed characterization of deposits.

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Conflicts of Interest: The authors declare no conflict of interest.

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