Effect of TiO₂ on the microstructure and phase composition of Al₂O₃ and Al₂O₃–TiO₂ APS sprayed coatings

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Abstract. Plasma sprayed ceramic coatings serve as protective layers and are frequently exposed to aggressive wear, corrosion, or high-temperature environment. Currently, alumina and alumina-titania are some of the most popular protective ceramic composite coatings used in the industry. The present work deals with the investigation of the influence of TiO₂ content in the feedstock powder on the resulting microstructure and properties of Al₂O₃, Al₂O₃₃ 3 wt% TiO₂, Al₂O₃₅ 13 wt% TiO₂ and Al₂O₃₇ 40 wt% TiO₂ coatings developed via atmospheric plasma spraying (APS). Specifically, the phase composition, morphology, and microstructure, as well as the mechanical and tribological performance of the coatings were examined. Results revealed that higher content of TiO₂ induced the transformation of phases, leading to the formation of intermediary Al₂TiO₅ and Al₂Ti₃O₇ phases. Also, the dominant α-Al₂O₃ to γ–Al₂O₃ transformation confirmed the formulation of well-melted lamellae within the coating structure. It was also shown that the increase in TiO₂ content decreased the micro-hardness of the coatings due to the formation of the intermediary phases as mentioned above and thus, affected their tribological performance. The lowest volumetric wear, equal to 7.2×10⁻⁹ (N·m), was reported for Al₂O₃ + 13 wt% TiO₂ coating.

Key words: coating; atmospheric plasma spraying; Al₂O₃; TiO₂.

1. Introduction

Atmospheric plasma spraying (APS) is one of the thermal spraying techniques which allows us to produce protective coatings, typically of the thickness of 100–500 µm [1]. The method requires the feedstock in the form of micrometer-sized solid powder [2], which is transported by the carrier gas, and then injected into a hot plasma jet [3]. Subsequently, the melted powders impinge the substrate surface, forms splats, and then build the lamellar coating structure [4]. Coatings may be deposited on metallic, ceramic, or even polymeric substrates [5]. Due to the variety of available substrates and coating materials, the APS method provides a wide range of possible applications, including wear components, biomedical parts, mechanical tools, and many others [6–8].

Importantly, many of the coatings are frequently exposed to high mechanical and thermal loads [9]. But regardless of the harsh conditions they are exposed to, the coatings must preserve satisfactory mechanical, tribological and other properties [6, 10]. Such performance depends on the spraying conditions, which determine the final microstructural properties of the coatings. In our previous study [11], it was shown that the final microstructure of the coating is influenced by the powder characteristic [12]. Dense, homogeneous coatings, which provide good mechanical properties, should be sprayed with the use of powders characterized by monomodal and narrow particle size distribution.

Al₂O₃ and Al₂O₃ + TiO₂ [13] are known for their good mechanical properties and favorable tribological behavior (Fig. 1) [7, 14–17]. Specifically, pure Al₂O₃ coatings are commonly

![Selected properties of Al₂O₃ and Al₂O₃ + TiO₂ coatings](image-url)

Fig. 1. Selected properties of Al₂O₃ and Al₂O₃ + TiO₂ coatings [7, 14–17]
used due to their high hardness and corrosion resistance [6]. To provide increased toughness and wear resistance, TiO₂ is added to pure Al₂O₃ [18,19]. For about 50 years, the chemical composition of Al₂O₃+TiO₂ composite powders commercially available in the market is limited to 3 wt.% [20, 21], 13 wt.% [22, 23], and 40 wt.% [13, 24] of TiO₂. Some other compounds were also investigated but limited only to the individual work. Some authors mixed two separate kinds of particles and studied Al₂O₃+8 wt.% TiO₂ [25] and Al₂O₃+50 wt.% TiO₂ [26].

According to the Al₂O₃–TiO₂ phase diagram proposed by Freudenberg in 1988 [27], changes in the chemical composition of powder feedstock significantly influence the formation of coatings. Firstly, with the increased content of TiO₂, the melting temperature is lowered, which privileges the melting of the powders and provides the homogeneity of the structure [28]. Secondly, the amount of TiO₂ closed to 44%, induces the formation of the Al₂TiO₅ phase, in addition to well-known α-Al₂O₃ to γ-Al₂O₃ phase change [1,29].

So far, a multitude of studies devoted to APS coatings of the Al₂O₃–TiO₂ system has already been carried out [11,30]. However, many papers regarding the microstructure, the mechanical and tribological performance of the coatings are limited solely to one specific chemical composition. Therefore, in this work, attention is given to the influence of TiO₂ on the phase composi-

tion, morphology, microstructure, mechanical and tribological performance of the coatings. The comparison of pure Al₂O₃ and Al₂O₃+TiO₂ coatings was also highlighted in this work.

2. Materials and methods

2.1. Powders. Four different feedstock powders were used in this study: (i) Al₂O₃, (ii) Al₂O₃+3 wt.% TiO₂, (iii) Al₂O₃+13 wt.% TiO₂ and (iv) Al₂O₃+40 wt.% TiO₂. They are labelled in the paper as: AT0, AT3, AT13 and AT40, respectively.

The following commercial micrometer-sized powders were sprayed utilizing atmospheric plasma spraying:

- Al₂O₃ Metco 6103 (Oerlikon Metco, Pfäffikon, Switzerland), agglomerated and sintered, −45+15 µm.
- Al₂O₃+3 wt.% TiO₂ AMI 6300.1 (Amil Werkstofftechnologie GmbH, Berlin, Germany), fused and crushed, −45+22 µm.
- Al₂O₃+13 wt.% TiO₂ Metco 6221 (Oerlikon Metco, Pfäffikon, Switzerland), agglomerated and sintered, −45+15 µm.
- Al₂O₃+40 wt.% TiO₂ Metco 131VF (Oerlikon Metco, Pfäffikon, Switzerland), agglomerated, −45+5 µm.

The SEM observations confirmed the results of laser diffraction (LD), see Figs. 2 and 3. Microscopic investigations were

![Fig. 2. Top view and cross-section images of powders used for spraying: AT0 (a), AT3 (b), AT13 (c), and AT40 (d)](image)

![Fig. 3. Particle size distributions of powders used for spraying: AT0 (a), AT3 (b), AT13 (c), and AT40 (d)](image)
carried out by a scanning electron microscope Phenom G2 Pro (Phenom World BV, The Netherlands). The powder particle size was evaluated by Partica LA-950V2 (Horiba, Kyoto, Japan), in accordance with the standard [31].

2.2. Spraying. The austenitic stainless steel 1.4301 (2 mm in thickness, with a diameter of 25 mm) was used as a substrate. Prior to the plasma spraying, the coupons were sand-blasted (final $R_a = 3 \mu m$) and cleaned in an ethanol bath. The Ni-Cr 80/20 (Amperit 250, Höganäs GmbH, Laufenburg, Germany) APS sprayed bond coats with the surface roughness of $R_a \approx 5 \mu m$ and the thickness of around 70 $\mu m$ were deposited prior to the deposition of the alumina and alumina-titania topcoats.

A conventional APS was carried out using an SG-100 torch (Praxair, Indianapolis, USA). The deposition of the coatings was accompanied by the optimization of the spraying set-up [11]. Powders were injected with a radial, external mode. The spraying parameters are given in Table 1.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Electrical power (kW)</td>
<td>35</td>
</tr>
<tr>
<td>Gas flow rate Ar/H$_2$ (l·min$^{-1}$)</td>
<td>45/5</td>
</tr>
<tr>
<td>Stand-off distance (mm)</td>
<td>100</td>
</tr>
<tr>
<td>Torch scan speed (m·s$^{-1}$)</td>
<td>0.3</td>
</tr>
<tr>
<td>Powder feed rate (g·min$^{-1}$)</td>
<td>20</td>
</tr>
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</table>

Table 1 Spraying parameters of ceramic topcoats

2.3. Characterization of coatings. The coatings were investigated by a scanning electron microscope Phenom G2 Pro (Phenom World BV, The Netherlands). The porosity of the samples was assessed in ImageJ free software, in accordance with ASTM E2109-01 standard [32]. The analysis was carried out for at least 20 SEM images made at 1000x magnification for each coating. The average thickness of deposits was measured on the micrographs at 500x magnification, in random areas at the coating cross-section.

The fracture morphology of the coatings was observed by a JEOL JSM-5800LV-Oxford scanning electron microscope (JEOL, Tokyo, Japan). The coatings were pre-cut and manually broken to get the fracture surface.

The X-ray diffraction data of the feedstock powders and coatings were obtained using a D8 Advance diffractometer under the Bragg–Brentano configuration with Cu $K_\alpha = 0.15406$ nm radiation. A continuous scan mode with an increment of 0.02° in the scanning range of $10°-80°$ ($2\theta$) was used. The content of the phases was assessed by the reference intensity ratio (RIR) technique, given in detail in [33, 34].

The roughness of the coatings was measured according to the standard [35] by profilometer MarSurf PS10 (Mahr, Göttingen, Germany). For each coating, 5 measurements of $R_a$, $R_z$, $R_p$ parameters were carried out and then the average and the standard deviation were determined.

The microhardness of the coating was estimated with a Vickers indenter under the load of 1.96 N (HV0.2) on a Sinowon HV-1000 apparatus (Sinowon Innovation Metrology, China), according to the standard [36]. Ten (10) indentations in random areas of the coatings were made for each time. After the measurements, the average values and standard deviations were specified.

Dry sliding wear tests were carried out in a rotating unidirectional configuration, with the use of a T-01 device (ITe-PIB, Radom, Poland), according to the standard [37]. The coatings were ground and polished before the tests to keep the $R_a$ of free-surface below 0.8 $\mu m$. As a counterpart, polished 100Cr6 bearing steel balls with a diameter of 6 mm were used. The tests were carried out under ambient conditions, over a distance of 500 m. The other parameters used for testing were the following: a relative sliding speed of 0.1 m/s, a wear track radius of 6 mm and a normal load of 5 N. Wear traces were observed by SEM Phenom G2 Pro (Phenom World BV, The Netherlands).

3. Results and discussion

3.1. Microstructure and phase composition. The observations of coatings topography (Fig. 4) revealed a classical morphology of conventional plasma sprayed coatings [38]. On the surface, well-melted micron-sized splats, as well as semi- and even non-melted powder particles were distinguished. The last one was especially observed in the AT40 coating but some single original feedstocks were observed in AT0, probably due to the highest melting temperature among all tested powders.

Furthermore, it is supposed that AT40 powder fragmented and disaggregated during spraying – many fine, non-aggregated particles were observed in the top surface of AT40 coating. This was not found for AT0, AT3 and AT13 particles, characterized by different production conditions: AT0 and AT13 – agglomerated and sintered; AT3 – fused and crushed (this powder is not available on the market in the agglomerated state); AT40 – only agglomerated. The agglomerating and sintering process provided stronger binding and thus, easier transport of the material during AT0 and AT13 APS spraying. A similar trend of good bond strength (and consequently – unproblematic feeding) was observed also for AT3 powder. Currently, commercially available spheroidal AT40 powders are manufactured only in the agglomerated form, without sintering. Thus, AT40 particles debonded the most easily and were easily directed to-
towards the periphery of plasma jet, characterized by lower temperature. Hence, some re-solidified or even non-melted powder particles were observed.

The cross-section micrographs (Fig. 5) showed that all coatings well adhered to the bond coat layer. The interface between alumina or alumina-titania topcoats and NiCr bond coats is free of any cracks, pores, or discontinuities. Coatings revealed a similar thickness, in the range between 186 µm and 201 µm, which provided similar deposition efficiencies as the coatings were sprayed with a similar number of spray cycles each time (Table 2). The microstructure of coatings was relatively homogeneous, free of inter- or intra-lamellar cracks, and with some content of porosity, which is typical for plasma-sprayed coatings. It was observed that the pores were of various sizes (ranging from a micrometer to a nanometer) and were homogeneously distributed along the coating cross-section.

![Fig. 5. SEM images showing cross-sections of deposited coatings](image)

### Table 2
Average thickness and porosity of deposited Al2O3 and Al2O3–TiO2 coatings

<table>
<thead>
<tr>
<th></th>
<th>AT0</th>
<th>AT3</th>
<th>AT13</th>
<th>AT40</th>
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<tbody>
<tr>
<td>Thickness (µm)</td>
<td>189 ± 9</td>
<td>201 ± 7</td>
<td>198 ± 6</td>
<td>186 ± 5</td>
</tr>
<tr>
<td>Thickness per pass (µm/pass)</td>
<td>24</td>
<td>25</td>
<td>25</td>
<td>23</td>
</tr>
<tr>
<td>Porosity (vol. %)</td>
<td>14 ± 1</td>
<td>9 ± 1</td>
<td>13 ± 1</td>
<td>12 ± 1</td>
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</table>

The values of porosity are given in Table 2. No direct relationship was observed between the TiO2 content and porosity. However, the influence of the manufacturing route of the powders cannot be neglected. It was reported that powder feedstock of the same chemical composition, but made by different techniques, may influence the properties of the final coating [13]. It was shown that the melting of the powder depends not only on the melting point (resulting from the chemical composition) but is also influenced by a thermal conductivity of the particle itself. Wang et al. [39] has shown that the fused and crushed feedstock powder, which consisted of mechanical blends of separately fused Al2O3 and TiO2 particles, was characterized by better thermal conductivity than the Al2O3 + TiO2 powder in the agglomerated form. The poor thermal conductivity led to the increased porosity of the coating [40]. As a result, an AT3 coating exhibited a higher melting and lower porosity than AT13 and AT40 coatings. Nevertheless, when the porosity of the coatings is compared based on the agglomerated powders, it could be seen that with the increased content of TiO2, the porosity of the coatings is slightly decreased.

Fig. 6 shows SEM micrographs of fracture surfaces. Although all of the investigated samples were built of finely structured powders, the observed surfaces showed some differences. For AT0, the lamellar structure was clearly visible, and the columnar grains were obviously observed, which was caused by rapid cooling rates upon the particle deposition. AT3 revealed the smoothest fracture surface when compared with AT0, AT13, and AT40, with well-flattened splats. This may be attributed to a different powder morphology of AT13. The fractures of AT13 and AT40 also exhibited a typical structure for plasma spraying. However, some single fine pores for AT13 were observed. Finally, the fracture surface of AT40 consisted of regions with a different melting degree. Unlike in the AT0 and AT3 lamellar fractures, a bimodal morphology, containing melted and non-melted fine powder residues were retained in the coating. Additionally, single micro-sized voids were observed, which were induced by the presence of loosely bonded powders.

![Fig. 6. SEM micrograph of the fractured surfaces in deposited coatings](image)

Fig. 7 presents the phases identified in powders (Fig. 7a) and APS sprayed coatings (Fig. 7b). AT0 particles contained 100% of thermodynamically stable α-Al2O3. In the case of...
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AT3 powder, a diffraction analysis also revealed only peaks of α-Al₂O₃, and the results were confirmed by a quantitative evaluation, given in Fig. 8. It is assumed that in this powder, some TiO₂-related phases might be present, but their concentration was not enough to be detected by XRD. AT13 and AT40 powders, in addition to α-Al₂O₃ phase, also contained: rutile-TiO₂ (AT13, AT40), tialite Al₂TiO₅ (AT13, AT40), and Al₂(−x)Ti₁+(x)O₅ (AT40). Similar phase compositions were found in other studies [3, 6, 41].

After spraying the powders, phase changes occurred. In the case of AT0 coatings, the transformation of α-Al₂O₃ into γ-Al₂O₃ was reported, due to lower nucleation energy of γ-Al₂O₃ and the rapid cooling of the splats [42, 43]. The α-Al₂O₃ remained in the coating due to the presence of semi-melted powder particles, as confirmed by the previous SEM observations (Fig. 4). In AT3 coatings, the content of α-Al₂O₃ (25 vol.%) was significantly reduced with an appearance of γ-Al₂O₃ (72 vol.%), accompanied by a formation of a small amount of Al₂TiO₅ (3 vol.%). The results confirmed then the high degree of powders melting [44], as already discussed in the context of porosity evaluation. In AT13 coatings, the changes were even more complex. Beyond the common transformation of α-Al₂O₃ into γ-Al₂O₃ (before spraying – 83 vol.%, after – 53 vol.% of α-Al₂O₃), it was observed that tialite (Al₂TiO₅) was reduced significantly (from initial 10 vol.% to 5 vol.%). Additionally, rutile-TiO₂ was not observed in the coating (which was found in the as-produced powder). This resulted probably from the use of Ar/H₂ gases for the spraying process, which led to the reduction of TiO₂ phases [13]. Also, the AT40 coating revealed different phase composition when compared with the feedstock powder. The dominating phase was tialite Al₂TiO₅ (40 vol.%), and that was clearly related to the Al₂O₃–TiO₂ diagram [45]. Tialite originated from a reaction between Al₂O₃ and TiO₂ in the hot plasma plume. Additionally, its formation was promoted by the fine size of AT40 particles, characterized by a larger surface area [27, 46, 47]. In the agglomerated powder, the total contact surface area between Al₂O₃ and TiO₂ is very high due to the small size of the primary particles. In that case, Al₂O₃ and TiO₂ react together and form Al₂TiO₅. A significant reduction of the intermediate phase Al₂(−x)Ti₁+(x)O₅ was also found, due to the oxidization and decomposition into Al₂TiO₅, [13].

Fig. 7. XRD patterns of powders used for APS spraying (a) and deposited coatings (b)

Fig. 8. Quantitative estimation of the phases composition in powders and coatings
3.2. Roughness. Fig. 9 represents the average surface roughness of the as-sprayed coatings. Amongst all coatings, AT40 revealed the lowest roughness parameters. This stemmed from the deposition of finer particles, which had already broken during feeding to the plasma jet. Smaller powders tend to generate a surface of lower roughness [48]. $R_s$, $R_z$, and $R_t$ parameters obtained for AT0, AT3, and AT13 coatings were similar, which is caused by the factors influencing the roughness most effectively (namely: plasma power, carrier gas flow rate, and rotation velocity of the substrate during spraying [24, 49]), were kept at a constant level. The sizes of AT0, AT3 and AT13 particles were similar to each other and thus resulted in similar roughness of the deposited coatings.

![Fig. 9. Average roughness of sprayed coatings](image)

3.3. Microhardness. The results of microhardness measurements (Fig. 10) clearly indicated that HV0.2 decreased with the increased content of TiO$_2$. At the same time, it was observed that the AT13 coating with the highest content of a hard and brittle $\alpha$-Al$_2$O$_3$ phase in the final deposit was characterized by a low decrease of hardness when compared with an AT3 coating [50]. Thus, the effect of the porosity on the hardness was limited by the effect of the phase composition [30].

![Fig. 10. Average Vickers microhardness of sprayed coatings](image)

The estimated hardness values corresponded well with the results given in the literature. For example, Yilmaz et al. [26] reported the hardness of Al$_2$O$_3 + 13$ wt.% TiO$_2$ coatings equal to 853 HV0.2, whilst Al$_2$O$_3 + 40$ wt.% TiO$_2$ – equal to 566 HV0.2. Similar values were given also by Yugeswaran et al. [21].

3.4. Wear resistance. Ball-on-disk tests revealed that the AT13 coating was displaying the best tribological behavior amongst all the sprayed coatings, with the volumetric wear equal to $7.2 \times 10^{-5}$ mm$^3$/Nm (see Fig. 11). Moreover, this coating was characterized by the lowest scatter of standard deviation.

![Fig. 11. Average volumetric wear of sprayed coatings](image)

The wear mechanism of the coatings was studied by SEM analysis, which is presented in Fig. 12. In the case of AT0 and AT3 coatings (see example in Figs. 12a–12b, respectively), the main wear mechanism was low cycle fatigue, accompanied by splats delamination. This cyclic fatigue was characterized by the cracking of the coating, the delamination of the material, and its smearing through the wear trace. In the case of the AT3 coating, a lot of debris and cracks are visible, as well as some scratches. The wear of the AT13 coating was different (see Fig. 12c) – a more adhesive mode was observed. However, numerous cracks were observed in the wear trace, and therefore, similar low cycle fatigue was observed. It was intensified by the spallation and smearing of the debris material. The highest wear resistance of the AT13 coating was related to its low friction coefficient (Table 3) and hard structure (high content of $\alpha$-Al$_2$O$_3$ phase). For AT40, the friction coefficient was higher, while the hardness and content of $\alpha$-Al$_2$O$_3$ phase were lower than for AT13. Additionally, it was observed that detachment of coatings was mainly initiated at the splat boundaries and areas of uniformities. In Fig. 12d there are some micro-scratching and spallation areas. In places where non-fully melted and re-solidified particles were removed, the relatively soft material (high content of an Al$_2$TiO$_3$ phase) was smeared on the surface and fill these areas. Therefore, the AT40 coating was worn the most intensively. A similar trend was observed also by Szala.

![Table 3](image)

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Average values of friction coefficients of deposited coatings</th>
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<tr>
<td>Friction coefficient $\mu$</td>
<td>AT0</td>
</tr>
<tr>
<td></td>
<td>0.68 ± 0.04</td>
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</table>
Effect of TiO$_2$ on the microstructure and phase composition of Al$_2$O$_3$ and Al$_2$O$_3$–TiO$_2$ APS sprayed coatings

The wear resistance of the coatings was related to the phases which remained after spraying the powder feedstocks. The dominant phases in AT0, AT3, and AT13 coatings were $\alpha$-Al$_2$O$_3$ and $\gamma$-Al$_2$O$_3$. The AT3 coating contained only 25 vol.% of $\alpha$-Al$_2$O$_3$, whilst in AT0 one alpha phase content was equal to 50 vol.%, which apparently influenced the microhardness values. On the other hand, the porosity of AT3 was significantly lower than that of AT0, and it could be a reason why similar volumetric wear of the AT0 and AT3 coatings was obtained. In this case, the powders were melted only partially; that is, the outer surface of the powders melted and solidified rapidly (in the form of $\gamma$-Al$_2$O$_3$ phase, of relatively lower hardness and higher toughness than $\alpha$-Al$_2$O$_3$), whilst the core of the powder was unchanged (in the form of hard $\alpha$-Al$_2$O$_3$). Therefore, the coatings revealed the structure that was built of the $\gamma$-Al$_2$O$_3$ matrix, strengthened by $\alpha$-Al$_2$O$_3$ [52]. Additionally, low wear of the AT13 coating influenced by the decrease in the coating hardness with TiO$_2$ is added in Al$_2$O$_3$ powder, as confirmed also by other authors [49]. The AT40 coating was worn the most intensively ($1.4 \times 10^{-4}$ mm$^3$/N·m), which is consistent with having the lowest microhardness amongst all coatings.

4. Conclusions

In this paper, the APS spraying with feedstock powders having chemical compositions: Al$_2$O$_3$, Al$_2$O$_3$ + 3 wt.% TiO$_2$, Al$_2$O$_3$ + 13 wt.% TiO$_2$, and Al$_2$O$_3$ + 40 wt.% TiO$_2$ was carried out. The coatings were studied in terms of the influence of TiO$_2$ content on the microstructure, phase composition, as well as the chosen mechanical and tribological properties. To summarize, the following can be stated:

- All the coatings revealed a topography and microstructure typical for APS sprayed coatings, with cracks, pores, and voids. It was observed that powder melting depends not only on TiO$_2$ content but is influenced by its characteristics resulting from the manufacturing route.
- XRD studies showed that all the powders underwent phase transformations during spraying. With the increased content of TiO$_2$, the number of phases related to pure Al$_2$O$_3$ ($\alpha$-Al$_2$O$_3$ and $\gamma$-Al$_2$O$_3$) was considerably decreased, whilst the number of TiO$_2$-phases (rutile-TiO$_2$, Al$_2$TiO$_5$, Al$_{2(2-x)}$Ti$_{1(1+x)}$O$_5$) increased.
- The clear trend of the decrease in Vickers hardness was observed in the coatings with an increased amount of TiO$_2$, due to the presence of more phases derived from TiO$_2$. The highest hardness of 994 HV0.2 was obtained for Al$_2$O$_3$, characterized also by the highest content of $\alpha$-Al$_2$O$_3$ (50 vol.%) in the final coating.
- The wear resistance of the coatings was influenced both by the decreased hardness of the coatings with increased TiO$_2$ content, as well as different phase compositions of the as-sprayed coatings. The AT13 coating showed the best tribological performance, due to the combined effect of favorable hardness and the microstructure consisting of an $\gamma$-Al$_2$O$_3$ matrix, strengthened by $\alpha$-Al$_2$O$_3$ particles.
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