

Article

Wear Resistance of (Ti,Al)N Metallic Coatings for Extremal Working Conditions

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Abstract: The article includes research results for the functional properties achieved for a wide range of sintered tool materials, including sintered carbides, cermets and three types of Al₂O₃ oxide tool ceramics ((Al₂O₃ + ZrO₂, Al₂O₃ + TiC and Al₂O₃ + SiC_(w)) with (Ti,Al)N coating deposited in the cathodic arc evaporation (CAE-PVD) method and comparison with uncoated tool materials. For all coated samples, a uniform wear pattern on tool shank was observed during metallographic analysis. Based on the scanning electron microscope (SEM) metallographic analysis, it was found that the most common types of tribological defects identified in tested materials are: mechanical defects and abrasive wear of the tool side, crater formation on the tool face, cracks on the tool side, chipping on the cutting edge and built-up edge from chip fragments. Deposition of (Ti,Al)N coating on all tested substrates increases the wear resistance and also limits the exceeding of critical levels of permanent stresses. It even increases the tool life many times over. Such a significant increase in tool life results, among other things, from a large increase in microhardness of PVD coated materials compared to uncoated samples, increased resistance to thermal and chemical abrasion, improved chip formation and removal process conditions. Use of hard coatings applied to sintered tool materials is considered to be one of the most important achievements in improving the functional properties of cutting tools and can still be developed by improving the coating structure solutions (sorted and nanocrystalline structures) and extending the range of coating applications (Ti,Al)N in a variety of substrates.

Keywords: sintered tool materials; PVD; coatings; metallic coatings; ceramic coatings; wear resistance; cutting



Citation: Mikula, J.; Pakula, D.; Żukowska, L.; Gołombek, K.; Kříž, A. Wear Resistance of (Ti,Al)N Metallic Coatings for Extremal Working Conditions. *Coatings* **2021**, *11*, 157. <https://doi.org/10.3390/coatings11020157>

Received: 23 September 2020

Accepted: 27 January 2021

Published: 29 January 2021

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1. Introduction

Among ceramic materials, ceramics based on Al₂O₃ occupy a special place due to their functional properties, but also a relatively low share in the market of cutting tool materials. The high hardness of Al₂O₃ combined with the high strength of crystalline silicon carbide allows the treatment of heat-treated alloy steels, pressed steels, overlay layers or hardening welds. Alumina ceramics (Al₂O₃), even at very high sinter density, is characterized by high hardness and resistance to abrasion at high temperatures. The technologies for obtaining alumina materials require the introduction of numerous additives into the aluminum oxide that activate or inhibit the sintering process in strictly controlled proportions in order to modify the properties of the finished sinter [1–3]. This applies to additives leading to improvement of sinterability and supporting grain growth of Al₂O₃ (TiO₂, TiO₃, MnO, CuO), having a limited effect on sinterability and recrystallization (Fe₂O₃, ZrO₂), negatively influencing sinterability and limiting grain growth (Cr₂O₃, SiO₂, NaF, CaF₂) and having a positive effect on sinterability and limiting grain growth (MgO, MgF₂). Functional additives such as TiC [4] or SiC_(w) [5] were also introduced.

However, this does not eliminate the drawbacks limiting the applicability of the oxide ceramics. Therefore, the paper discusses additional methods of increasing the durability of ceramic tool materials (referring the wide spectrum of Al_2O_3 -based materials to typical types of cermets and sintered carbides) with the use of a highly efficient (Ti,Al)N coating.

The deposited (Ti,Al)N coatings may exhibit a monophasic cubic structure at a low Al content ($<0.55\%$) and a monophasic wurtzite structure at a high Al content ($<0.69\%$). At intermediate Al contents ($0.55\text{--}0.69\%$), a mixed cubic-wurtzite phase [6] was observed. The mechanical strength and oxidation resistance of the deposited (Ti,Al)N increase with increasing Al content, but decrease for the mixed cubic-wurtzite structure. Strong chemical stability and good resistance to oxidative wear is also ensured at an elevated temperature of $900\text{ }^\circ\text{C}$ [7]. High thermal stability in dry machining at high temperatures is possible due to the formation of a hard and oxidation-resistant thin layer of mixed oxides of Al_2O_3 and TiO_2 . The generated passive layer dissipates heat by removing chips, which allows for increased production at higher cutting speeds [8]. Tool application of (Ti,Al)N coating increases wear resistance and eliminates a number of limitations and drawbacks observed in the use of TiN and Ti(C,N) coatings [9,10]. This article is a continuation of the research conducted by the authors in the field of increasing the functional properties of cutting tools [3,4,11–15].

The need to determine the range of possible and reasonable application of the (Ti,Al)N coating was the main reason for undertaking the research. The goal of the paper is to present the investigation results of functional properties of the wide range of sintered tool materials including: cemented carbides, cermets and three types of Al_2O_3 oxide tool ceramics ($\text{Al}_2\text{O}_3 + \text{ZrO}_2$, $\text{Al}_2\text{O}_3 + \text{TiC}$ and $\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$) with (Ti,Al)N coating deposited in the cathodic arc evaporation (CAE-PVD) method and compare them with the uncoated tool materials. The paper is continuation of research carried out by the authors in the area of (Ti,Al)N functional properties.

2. Materials and Methods

Material in the usable form of multi-point inserts was used for the investigations. A wide range of Al_2O_3 oxide tool ceramics ($\text{Al}_2\text{O}_3 + \text{ZrO}_2$, $\text{Al}_2\text{O}_3 + \text{TiC}$ and $\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$) were used as substrate, and typical cemented carbide and cermet materials were used to compare the results. Composition of Al_2O_3 -based ceramic materials was as follows: $\text{Al}_2\text{O}_3 + \text{ZrO}_2$ ceramics with 10 wt.% fraction of ZrO_2 , $\text{Al}_2\text{O}_3 + \text{TiC}$ ceramics with 35 wt.% fraction of TiC and $\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$ ceramics with 15 wt.% fraction of SiC. In case of SiC whiskers, an average diameter (d) of $d < 1\text{ }\mu\text{m}$ and a slenderness (s) of $s < 10\text{ }d$ was applied. In each case, a relative density of over 98% of the theoretical density was achieved. All sinters were characterized by a fine-grained structure. Oxide tool ceramics $\text{Al}_2\text{O}_3 + \text{TiC}$ has a fine-grained structure, with the smallest grain size among the tested types of oxide tool ceramics, with predominantly a number of grains less than $0.5\text{ }\mu\text{m}$.

The samples were coated with (Ti,Al)N coating in the CAE-PVD process. Table 1 shows the characteristics of the tested materials.

The coatings were deposited in laboratory conditions in the Department of Engineering Materials and Biomaterials of the Silesian University of Technology in Gliwice. The samples were prepared for coating deposition in a two-step process. First, chemical cleaning was carried out using washing and rinsing in ultrasonic cleaners and cascade washers, and then the samples were dried in a stream of hot air. In the second stage, the substrate was heated directly in the vacuum chamber to the temperature of $400\text{ }^\circ\text{C}$ in an argon atmosphere at reduced pressure, and then the samples were ion-cleaned with Ar ions with a substrate polarization voltage of -300 V for 25 min. Water-cooled discs with a diameter of 65 mm were used in the deposition process. The discs contained 50: 50% at. TiAl. The process was carried out at a pressure of 10^{-4} Pa . Coatings were deposited in the atmosphere of inert gas Ar and reactive gases N_2 in order to obtain nitrides (gas flow ratio Ar/ N_2 : 25/75) Parameters of deposition: polarization voltage of the substrate: -100 V ;

current intensity at the cathode: 80 A; time of deposition: 60 min; substrate temperature, 400 °C.

Table 1. Characteristics of the investigated materials.

Substrate	Coating	Coating Thickness, μm	Roughness, R_a , μm	Microhardness, HV	Critical Load, L_c (AE) N
Cemented carbide *	uncoated	–	0.14	1810	–
	(Ti,Al)N	2.4	0.18	2940	48
Cermet **	uncoated	–	0.07	1960	–
	(Ti,Al)N	2.7	0.15	3100	55
$\text{Al}_2\text{O}_3 + \text{TiC}$ ***	uncoated	–	0.10	2030	–
	(Ti,Al)N	2.5	0.17	3250	68
$\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$ ****	uncoated	–	0.24	1890	–
	(Ti,Al)N	2.1	0.25	3180	66
$\text{Al}_2\text{O}_3 + \text{ZrO}_2$ *****	uncoated	–	0.19	1870	–
	(Ti,Al)N	2.2	0.24	3150	59

* phase composition: WC, TiC, TaC, Co; ** phase composition: TiCN, WC, TiC, TaC, Co, Ni; *** phase composition: Al_2O_3 , TiC; **** phase composition: Al_2O_3 , SiC; ***** phase composition: Al_2O_3 , ZrO_2 .

Metallographic examinations were carried out in a SUPRA 35 scanning electron microscope (SEM, Carl Zeiss, Oberkochen, Germany). Appropriately prepared samples in the form of transverse fractures were used for observation. In the research, the method of detecting secondary electrons (SE) with an accelerating voltage in the range of 15–20 kV was used. The maximum magnification was $50,000\times$.

The thickness of coatings was measured on the rake face using the calotest method and was determined by measuring 5 craters on each sample.

Roughness of R_a (both surface of the coatings and surface of the base material before coating) was measured in two perpendicular directions. Test length, $L_C = 0.8$ mm, and measurement accuracy of ± 0.02 μm was assumed.

Hardness of the samples was determined by the Vickers method. Hardness measurements were made with a load of 2.94 N (HV 0.3) in accordance with the PN-EN ISO 6507-1: 2018-05 standard [16]. The microhardness of the coatings was measured using the dynamic Vickers method. Measurements were carried out in the “load-unload” mode, in which the tester, a loading indenter with a given force, maintains load for some time and then unloads it. This allows for observation of change in plastic and elastic deformation of the sample in loading and unloading mode, and the measuring system allows to record the depth of the created impression during the test. The essence of this test is to measure the impression depth in order to eliminate the influence of the substrate on the result of the coating hardness measurement. Initial hardness tests were carried out at load from 49 mN (HV 0.005). Results of microhardness of the tested coating are similar and do not differ from those found in the literature [17].

Adhesion of deposited coatings to the substrate was tested using the scratch test. A diamond penetrator was moved over surface of the tested coating under a gradually increasing load. For measurement of the critical load L_C (corresponding to the loss of coating adhesion to the substrate), the recorded increase in the value of acoustic emission (AE) was used.

Comparative cutting ability tests were performed with the use of cutting tools (multi-edge inserts) made of the tested materials. Wear resistance of tools made of uncoated base materials and tools with a (Ti,Al)N coating was tested. Tests were carried out in the form of technological cutting tests of gray cast iron EN-GJL-250 with a hardness of approximately 250 HB as the workpiece. The wear criterion (measured as the width of the wear track on the surface of the tool used for machining) was established at the level of

flank wear (VB) = 0.20 mm. Measurements were carried out with the following processing parameters: feed rate $f = 0.1$ mm/rev, depth of cut $a_p = 1$ mm, cutting speed $v_c = 150$ m/min. The exception was the Al_2O_3 ceramics reinforced with SiC whiskers, which shows a much higher wear resistance in comparison with other tested base materials. It is closely related to its microstructure. The addition of approximately 15 wt.% of whiskers with a diameter of $d = 0.1\text{--}0.5$ μm and slenderness $l = (5\text{--}10)$ into the material structure leads to a change in the fracture model of the material from brittle fracture along the grain boundaries in the case of pure aluminum oxide to cracking through grain in the case of the $\text{Al}_2\text{O}_3 + \text{SiC}$ composite, and consequently leads to a significant increase in hardness, fracture toughness and bending strength. This uncoated material exceeds the functional properties of the other tested materials with a coating, however i.e., economic (price) and environmental (carcinogenic effects of whiskers) aspects limit its production and use range. In comparative tests, Al_2O_3 ceramics reinforced with SiC whiskers were used, however, after reaching the assumed cutting time (60 min) without reaching the wear criterion, cutting tests with EN-GJS-400 spheroidal cast iron were carried out with parameters allowing for comparison durability of the material without and with a coating (feed rate $f = 0.2$ mm/rev, depth of cut, $a_p = 2$ mm, cutting speed, $v_c = 250$ m/min) only in case of the mentioned material. The character of the developed damages was evaluated basing on observations on light microscope and on scanning electron microscope and analysis of the chemical composition of the tool wear using the X-ray energy dispersive spectrograph (EDS).

3. Results and Discussion

The fractographic examinations carried out allows to state that the coatings were deposited uniformly onto investigated substrate materials. It was found, that investigated materials reveal the dense, compact structure, and their fracture surface topography attests to their high brittleness. The multicomponent (Ti,Al)N were deposited uniformly onto the investigated substrate materials. Coatings present a characteristic columnar, fine-graded structure and adhere tightly to the substrates (Figure 1). The structural and tribological advantages of the (Ti,Al)N coating have been described in previous works of the authors [11–14] and in cited papers [18–20]. The roughness, R_a , parameter measured for substrates is within the range of 0.07 μm in case of cermet substrate to 0.24 μm in case of Al_2O_3 ceramics reinforced with SiC whiskers. Depositing multicomponent (Ti,Al)N coatings onto the investigated substrates causes an increase of the roughness parameter from $R_a = 0.15$ μm in case of coating deposited on cermet substrate to $R_a = 0.25$ μm in case of coating deposited on cermet substrate Al_2O_3 ceramics reinforced with SiC whiskers. Coatings' roughness is largely independent of the degree of substrates' surface development. R_a parameter of coatings is mainly related with the columnar structure of coatings and also with the observed inhomogeneity of coatings, resulting from occurrences of multiple drop shaped micro-particles on the coating surface, which is connected with the nature of the employed coating deposition process character—cathodic arc evaporation CAE-PVD (Figures 1 and 2). Sizes of these micro-particles are differentiated and vary from several tenths of a micrometer to more than 1 μm , and can be a reason for the little increase of coatings' roughness, but the influence of this inhomogeneity on the functional properties, such as tooling quality and tool life, is inconsiderable [3,4]. The occurrence of these morphological defects is related to the essence of the cathodic electric arc evaporation process. During this process, as a result of the phenomenon of sublimation, the evaporated target material is formed, and, as a side effect of arc spraying, micro-droplets of evaporated material are formed, which are deposited in the form of such inhomogeneous particles on the coated surface. This is confirmed by tests of the chemical composition of particles made with the use of an EDS scattered X-ray energy spectrometer, indicating that they are made of pure metal, knocked out of a TiAl target, which settles and solidifies on the surface of the substrate [12]. Probably, as suggested by the authors of ref. [21], the presence of such metal micro droplets and their number are related to the increase in pressure of working gases and the voltage of substrate polarization. The occurrence of these defects

can be eliminated or minimized, e.g., by improving the configuration of cathodes and the macromolecule filtering system using a magnetic field.

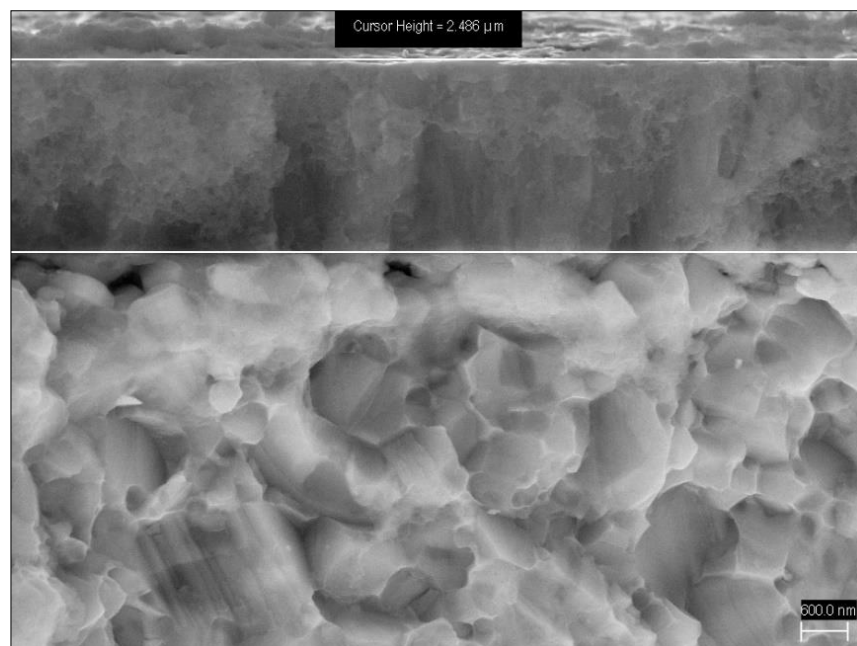


Figure 1. Fracture surface of the (Ti,Al)N coating deposited on cermet substrate.

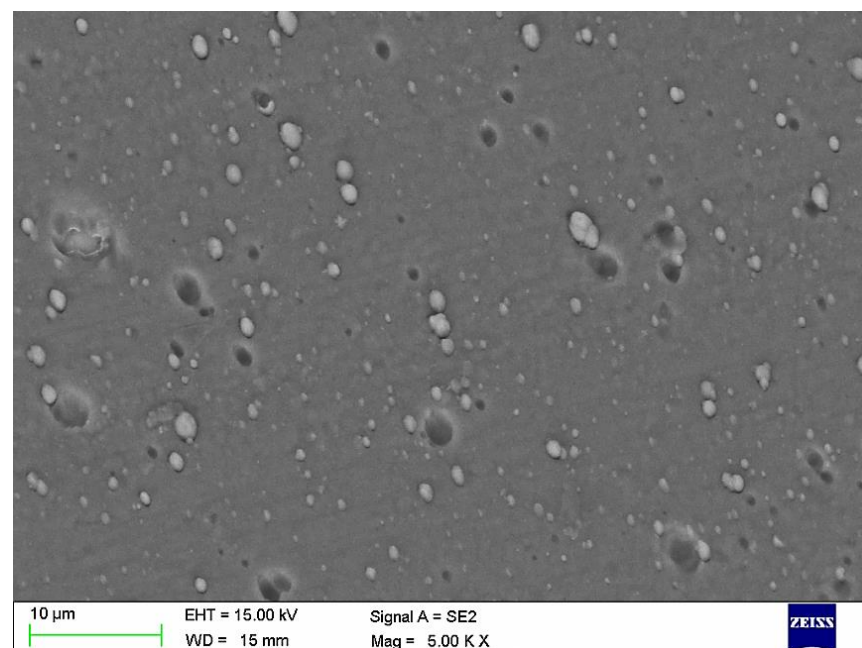


Figure 2. Topography of the (Ti,Al)N coating surface, deposited on the cemented carbide substrate.

The highest microhardness of investigated uncoated materials was stated in the case of Al_2O_3 ceramics reinforced with TiC (2030 HV0.3) and the lowest for cemented carbide (1810 HV0.3). Deposition of (Ti,Al)N coatings onto specimens causes the surface microhardness increase reaching from 2940 HV0.005 to 3250 HV0.005, that is over 50% more in comparison with the substrate hardness and in case of (Ti,Al)N coating is connected with the occurrence of the metallic-covalent character of bonds [15].

All investigated coatings are characterized by good adhesion in the range of 48–68 N, measured by acoustic emission registration during the scratch test. The best adherence to

the substrate, is demonstrated by coating deposited onto the substrate of Al_2O_3 ceramics reinforced with TiC (Figure 3). Good adhesion, especially in the case of Al_2O_3 ceramics reinforced with TiC can be caused among others by advantageous internal stress distribution in the area of coating/substrate connection, resulting largely from material constants and process condition. This was confirmed in the case of testing materials corresponding to those tested in this paper [12].

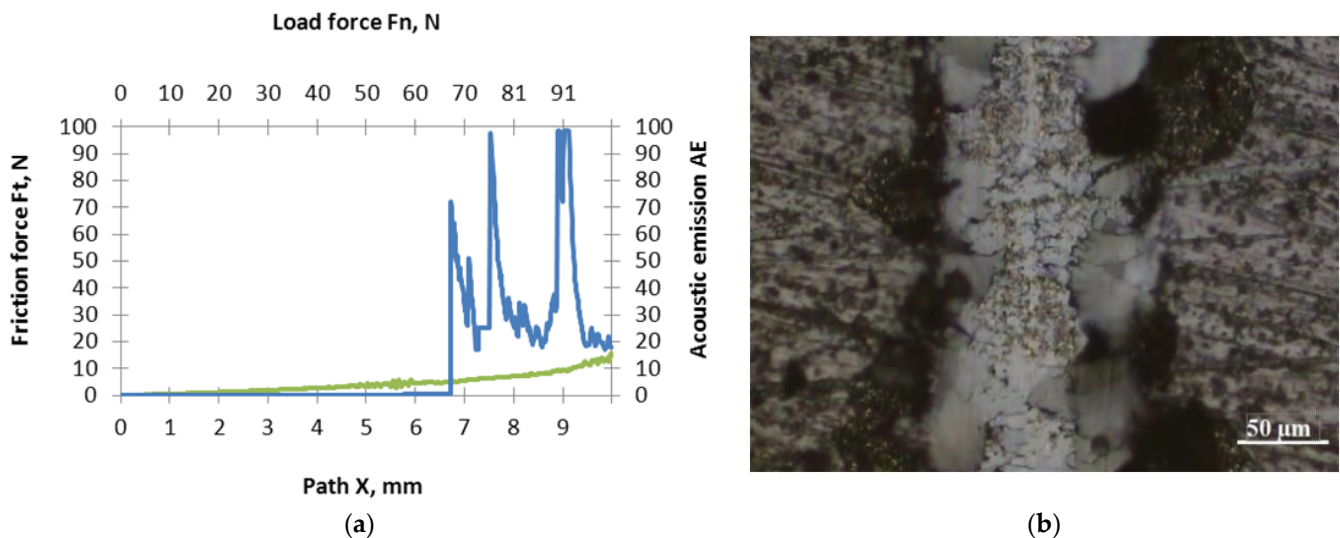


Figure 3. Scratch test results of the (Ti,Al)N coating surface deposited on $\text{Al}_2\text{O}_3 + \text{TiC}$ oxide ceramics substrate (a) diagram of the dependence of the acoustic emission (AE) seen as blue line and friction force F_t seen as green line, (b) characteristic damages.

The results of machining tests carried out with the use of uncoated inserts from sintered carbides, cermets and three types of oxide ceramics indicate that the longest tool life was achieved in case of Al_2O_3 ceramics reinforced with SiC whiskers. The cutting parameters and workpiece material were selected as a compromise that may allow for the carrying out the cutting tests at the same cutting conditions for wide range of sintered tool materials used as substrates, however in case of Al_2O_3 ceramics reinforced with SiC whiskers the tool life criterion flank wear track width of $\text{VB} = 0.2 \text{ mm}$ was not exceeded, even after 50 min of the continuous turning (Figure 4). Al_2O_3 ceramics reinforced with monocrystalline SiC whiskers provide extremely high wear resistance, especially fracture toughness through such mechanisms as crack deflection, crack bridging, and by whisker pull-out from the matrix when the tool is subjected to crack-forming stresses. For this reason it was decided to apply much more loaded cutting conditions only in case of this one ceramic substrate to measure the relative increase of tool life for uncoated and coated tools. In all other cases of substrates, such conditions would have caused a catastrophic damages, so originally assumed parameters were maintained.

Comparison results of machining tests carried out with the use of uncoated and coated materials revealed that depositing the (Ti,Al)N coatings on investigated sintered tool materials results in a significant increase of their wear resistance, irrespective of substrate used. The increase of tool life in case of cemented carbides and cermets is multiple and amounts to 500% and 633%. Several increases of tool life were also observed in case of $\text{Al}_2\text{O}_3 + \text{TiC}$ and $\text{Al}_2\text{O}_3 + \text{ZrO}_2$ ceramics (171% and 209%, respectively). Comparison of functional properties is shown in Figure 5. In case of Al_2O_3 ceramics reinforced with SiC whiskers ($\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$), made in stronger conditions, a 36% tool-life increase was achieved as a result of coating deposition (Table 2). The tool-life increase of all investigated sintered tool materials deposited with (Ti,Al)N coating is most likely associated not only with the microhardness values obtained by coatings, but also with low chemical coating affinity of the coating to the workpiece material (in particular to iron and carbon) and

with the barrier protecting the tool edge against oxidation and overheating. Wear resistant effects of coating are still provided, even in case of coating abrasion or local cracking at the edge zone because of diffusion of hard particles during cutting operations causing reinforcement of the near-surface zone of the edge, furthermore protective functions of coating at not damaged zones (e.g., chip and heat removing) are still fully provided (Figure 6a).

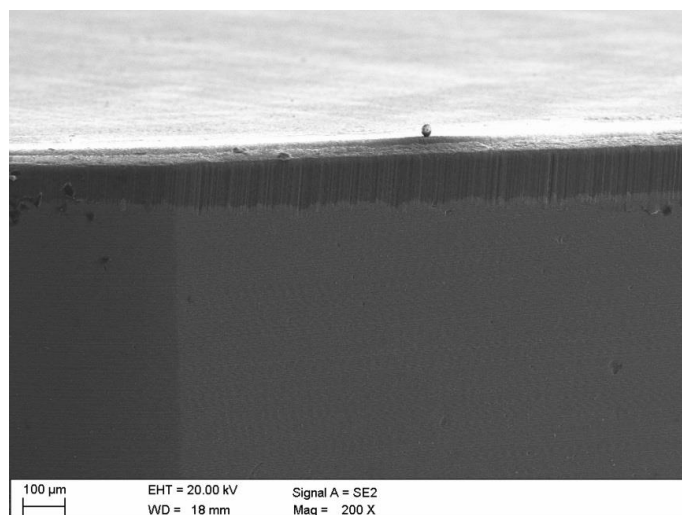


Figure 4. Flank wear character of the Al_2O_3 ceramics reinforced with SiC whiskers after 50 min turning of grey cast iron.

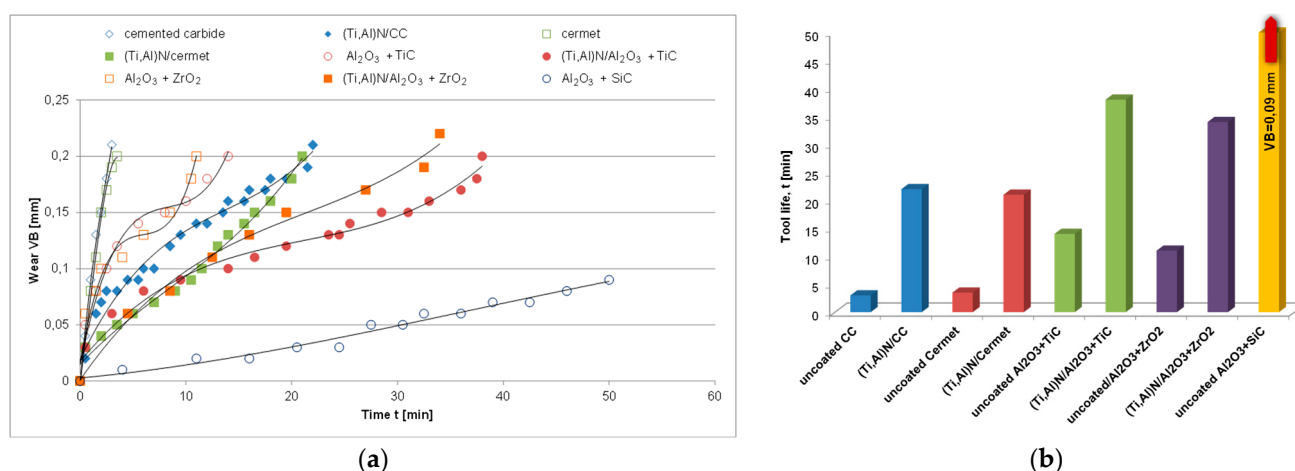


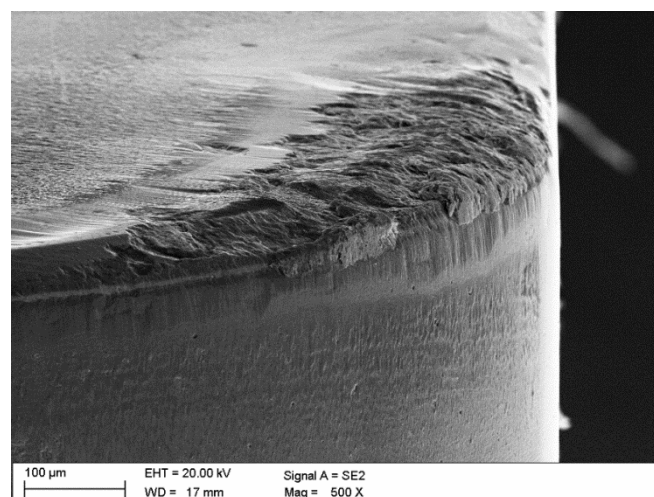
Figure 5. Comparison of the wear tracks (a) and tool life (b) of uncoated and coated sintered tool materials during cutting tests of EN-GJL-250 grey cast iron. VB criterion = 0.20 mm, feed rate, $f = 0.1$ mm/rev, depth of cut, $a_p = 1$ mm, cutting speed, $v_c = 150$ m/min.

In case of Al_2O_3 based ceramics (of low chemical affinity to Fe and high chemical stability), the diffusion wear is very low and characteristic uniform wear of tools with small craters was observed for uncoated tools as well for coated tools (Figure 6b). In this case wear protection of coatings can be mostly related with heat abstraction, chip disposal improvement, thermal shock and fatigue wear protection as well as abrasive wear protection. In case of cemented carbide and cermet substrates, crater wear was significantly decreased after coating deposition, mostly as a result of diffusion and abrasive wear protection (Figure 6c). This has been confirmed by results obtained earlier by the authors, and results of other tests of functional properties carried out for this group of coatings [9–14,22–24].

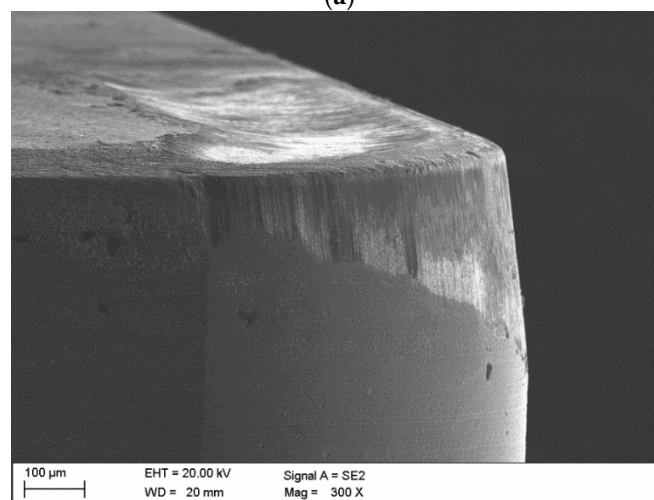
Table 2. Comparison of tool life for tools from cemented carbides, cermets, $\text{Al}_2\text{O}_3 + \text{ZrO}_2$, $\text{Al}_2\text{O}_3 + \text{TiC}$ and $\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$ oxide ceramics with the (Ti,Al)N coatings.

Substrate	Coating	Tool Life t , min
Cemented carbide	uncoated	3.0 *
	(Ti,Al)N	22.0 *
Cermet	uncoated	3.5 *
	(Ti,Al)N	21.0 *
$\text{Al}_2\text{O}_3 + \text{TiC}$	uncoated	14.0 *
	(Ti,Al)N	38.0 *
$\text{Al}_2\text{O}_3 + \text{ZrO}_2$	uncoated	11.0 *
	(Ti,Al)N	34.0 *
$\text{Al}_2\text{O}_3 + \text{SiC}_{(w)}$	uncoated	50.0 */5.5 **
	(Ti,Al)N	>60.0 */7.5 **

* EN-GJL-250 grey cast iron, feed rate, $f = 0.1$ mm/rev, depth of cut, $a_p = 1$ mm, cutting speed, $v_c = 150$ m/min; ** EN-GJS-400 spheroidal cast iron, feed rate, $f = 0.2$ mm/rev, depth of cut, $a_p = 2$ mm, cutting speed, $v_c = 250$ m/min.



(a)



(b)

Figure 6. Cont.

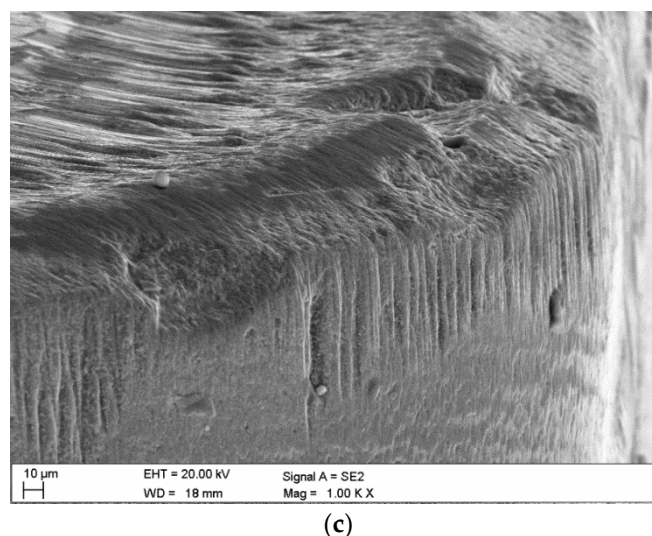


Figure 6. (a) Wear character of the cermet substrate with (Ti,Al)N coating after 15 min turning of grey cast iron; (b) Wear character of the Al_2O_3 ceramics reinforced with SiC whiskers after 60 min. turning of spheroidal cast iron; (c) Wear character of the uncoated cemented carbide after 3 min. turning of grey cast iron.

4. Summary and Conclusions

A wide range of different sintered tool substrates were coated with (Ti,Al)N coating characterized by dense, compact structure, good adhesion and high microhardness. Depositing the (Ti,Al)N coatings on investigated sintered tool materials resulted in significant increase of their wear resistance, for all types of substrates used. This result supplements results obtained in the work of other authors [9–14]. The tool-life increase effect of coating deposition regardless of substrate used should be connected with the low chemical affinity of coating material to the workpiece material, with a barrier that protects tool edge against oxidation and overheating, and with a very high microhardness causing abrasive wear protection. The process of coating wear and process of diffusion of hard particles during machining operations ensure abrasion resistance also after partial wear of the coating. In case of Al_2O_3 based substrates wear protection of coatings can be mostly related with heat abstraction, chip disposal improvement, thermal shock and fatigue wear protection as well as abrasive wear protection. Additionally, in case of cemented carbide and cermet substrates, crater wear was significantly decreased. It can be a result of diffusion and abrasive wear protection provided by the deposited coating. The results achieved on materials based on cemented carbides and cermets as well as on a wide range of oxide ceramics indicate the possibility of significant improvement of functional properties of coated cutting inserts for high speed machining, precision machining and dry cutting without the use of cooling lubricants. The scale of obtained increases in tool life of coated oxide inserts based on ceramic materials is a justification for extending the application of this type of coating to a wide range of ceramic materials, which are currently offered mainly in an uncoated state.

Author Contributions: Conceptualization, J.M. and L.Ž.; methodology, J.M., D.P., L.Ž., K.G., and A.K.; investigation, J.M., D.P., L.Ž., K.G., and A.K.; interpretation of data, J.M., D.P., L.Ž., K.G., and A.K.; data curation, J.M.; writing—original draft preparation, J.M., L.Ž., and K.G.; writing—review and editing, J.M., D.P., and K.G.; visualization, J.M.; supervision, J.M.; project administration, J.M.; funding acquisition, J.M. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the Ministry of Science and Higher Education of Poland as the statutory financial grant of the Faculty of Mechanical Engineering, Silesian University of Technology.

Data Availability Statement: Not applicable.

Acknowledgments: This work was supported by the Ministry of Science and Higher Education of Poland as the statutory financial grant of the Faculty of Mechanical Engineering, Silesian University of Technology.

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

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