

# Effect of nitride nano-scale multilayer coatings on functional properties of composite ceramic cutting inserts

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**Abstract.** A short literature survey which justifies coating of ceramic cutting inserts is presented. The results reported are on selected nitride coatings, in particular nanoscale multilayer, with layers of type Ti-Zr-N, TiN, ZrN and (TiAl)N, deposited by the arc PVD method on oxide-carbide ceramic cutting inserts of type TACN and TW2 produced at the Institute of Advanced Manufacturing Technology. Measurements and quality assessments were made, including of thickness of the coatings and of their constituent micro and nanolayers, microhardness of the coating and of the substrate, surface roughness of the inserts and of the cylindrical workpieces turned with these tools. Lifetimes of the coated and uncoated inserts were compared in turning an alloy tool steel. A significant increase in lifetime of the coated TW2 cutting tools was shown.

**Key words:** PVD coating, nanoscale multilayer, cutting insert, ceramics, tool life.

## 1. Introduction

Despite progress in non-conventional manufacturing technologies, metal cutting still plays an important role in the production of machines, apparatus and goods for everyday use; it makes possible achievement of the highest geometric accuracy of products and produces the best surface properties. Achievement of high requirements with regards to cutting tools is made possible through the development of novel tool materials and wear resistant coating deposition technology. In the Institute of Advanced Manufacturing Technology, R&D are carried out on special cutting tools, especially those designed for cutting hard-to-machine materials [1].

Alumina-based ceramic cutting tools have been used for over sixty years, but only in the last two decades have they found appropriate applications, first of all in turning and milling processes of cast iron and nickel based alloys, and also in finishing operations of hardened steels. Aluminium oxide and silicon nitride-based tools exhibit better cutting properties than coated cemented carbides in the above areas of application [2]. At present the following groups of ceramic tool materials can be distinguished [3,4]:

- $\text{Al}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3 + \text{ZrO}_2$  oxide ceramics,
- $\text{Al}_2\text{O}_3 + \text{TiC}$ ,  $\text{Al}_2\text{O}_3 + \text{TiN}$ ,  $\text{Al}_2\text{O}_3 + \text{Ti(C,N)}$  composite ceramics,
- SiC whiskers-reinforced  $\text{Al}_2\text{O}_3$  oxide ceramics,
- $\text{Si}_3\text{N}_4$ , SiAlON nitride ceramics,
- diamond and cubic boron nitride, super-hard materials .

Ceramic materials are characterized by high or very high hardness and by abrasive wear resistance; but also by relatively low fracture toughness [3]. This group of ceramic tool materials exhibits differentiation of properties such as: thermal and electrical conductivities, hardness, abrasive wear re-

sistance and chemical corrosion resistance, resistance to temperature change and chemical inertness towards the workpiece material, all of which determine applications of these materials [2].

R&D work concerning ceramic cutting tools concentrates on two main issues [3]:

- development of novel ceramic grades of higher fracture toughness and reliability, designed for tools used in machining in conditions of high feed and large depth of cut (e.g. in machining of pearlitic cast iron),
- development of grades of high hardness and chemical resistance, designed for precision machining of hardened steels and hard cast iron.

High-efficiency turning of large workpieces is a further important area of application of ceramic tools [2].

Appropriate coatings deposited on tools made of oxide, nitride and super-hard ceramics can be beneficial, especially for hard turning [1,2]. As a consequence of recent developments in coating deposition technology, many new ceramic tools with PVD and CVD coatings have appeared in the marketplace [3–7]. Up to a recent time, there was a predominant opinion that coating of ceramic inserts was pointless, because of the high or very high hardness of the ceramics [5]; this opinion has changed recently, when beneficial influence of coatings on functional properties of ceramic tools was found. The influence of coating on the increase in tool life is interpreted by a decrease in heat emission while machining, due to a decrease in the coefficient of friction and also due to a lower probability of chipping, by eliminating their initiation sites [3,6,7].

Review of the literature regarding coated ceramics presented in paper [2] has shown that, so far, only simple titanium nitride coatings have been deposited on a ceramic substrate.

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However, due to increasing requirements and progress in machining processes such as dry machining, hard machining, high speed cutting (HSC) and high performance cutting (HPC), new concepts are needed for high performance coatings [8]. Development of PVD coatings in the area of cutting tool applications has taken place mainly with reference to steel (especially HSS) or cemented carbide substrates [8-23], and only in few cases referring to the ceramic substrate, including cBN [21,24,25].

Main trends in the development of PVD coatings were presented, amongst others, by Luo et al [9], Jehn [26], Donnet and Erdemir [27] and Musil [28]; they considered changes in the chemical composition and structure of the coatings, systems involving nitrides and other compound layers, starting from two-component titanium nitride (TiN), through three-component nitride (e.g. TiAlN), up to multi-component nitrides (e.g. TiAlCrYN) and multilayer nitride systems [10,23, 26–31], including coatings consisting of nitride nanolayers, such as for example TiN/NbN, TiN/VN, TiAlN/NV, TiAlN/CrN [9]. Wierchoń [32] characterized structure and properties of multicomponent and composite layers produced by combined surface engineering methods, among which PVD and plasma assisted CVD methods played key roles; the latter methods have been applied to the deposition of composite coatings of ultra-high Vickers hardness (80–105 GPa) [11].

Reduction in the thicknesses of the layers in multi-layered coatings and their structure refinement led to the production of a new generation of coatings, which were characterised by nanoscale compositionally modulated multilayer structures of miscellaneous chemical compositions. These coatings, in general, consist of nitrides, carbides, carbonitrides, and sometimes of oxides, constituting binary, ternary or quaternary systems of the following chemical elements, in various combinations: Ti, V, Cr, Zr, Nb, Mo, Hf, Ta, W, Al, Si, and also sometimes B and C (e.g. cBN and CN<sub>x</sub>). In the compositions of such coatings there are also often chemical elements which do not form either nitrides or carbides, such as Cu, Ag, Au or Pd, and which are added with the aim of the structure refinement.

Coatings are described as “nano-layer” or nano-structured when the thicknesses of layers in the coatings or their grain sizes are below 200 nm. Formation of phases, and their distribution in multi-phase and multilayer coatings, occurring during the process of coating deposition, leads to the exceptional properties of such coatings, e.g. their very high hardness, which could reach even a value of 70 GPa, and which is much higher than the hardness of layers or phases present in the coating; this usually occurs in nano-scale coatings [10–12].

Substances that are present in the composition of micro-scale and nano-scale multi-layer coatings are usually characterised by crystalline structures (thermodynamic equilibrium state), or they are amorphous (non-equilibrium state); the coatings can also be composed of a mixture of crystalline and amorphous components. When hard coatings are produced by the PVD method, various phases are formed, amongst which different relations occur, namely:

- complete solid solubility (e.g. TiN-TiC, TiC-WC),
- complete immiscibility, e.g. TiC/Ag, WC/Ag,

- coexistence of crystalline-grain phases with intergranular amorphous phase (e.g. nano-crystalline (nc) TiN grains with an intergranular amorphous a-SiN<sub>x</sub> phase),
- coexistence of crystalline and amorphous phases (e.g. WC-SiC thin films, showing various degree of crystallization, depending on the SiC content [33].

Tribological properties of multiphase coatings can be achieved by the appropriate design of structure of functional layers and by modifying the phase compositions of multiphase coatings. Typical configuration of layers in complex micro-scale multilayer wear resistant coatings was presented in Reference [13]. Coatings deposited on specially prepared substrates of relevant hardness and toughness consisted of several functional layers, namely: “adhesive” metallic layer (e.g. Ti, Cr, Mo, Zr), basic wear resistant layer of high hardness and possibly low internal stress (TiN, CrN, ZrN, TiCN), layer blocking heat flow to the tool (TiAlN, TiZrN), and a layer of low coefficient of friction (Cr, CrN, TiN) [13].

Nano-scale multilayer coatings deposited by the PVD method should ensure that the cutting tools possess [34]:

- optimum hardness to internal stress ratio (high stability of geometry of cutting edge, uniform wear),
- higher thermal strength and chemical stability (possibility of dry and high speed machining, lower “grooved” wear),
- better sliding properties (better chip formation and higher quality of machined surface),
- increased wear resistance (decreased tool costs).

The purpose of the investigations carried out in Institute was to produce good quality PVD coatings on cutting inserts made of oxide and composite ceramics and cBN; these coatings should produce an increase of tool lives of inserts and should meet further requirements at the same time, e.g. regarding the quality of machined surface. Tool life and tool wear tests performed at Institute, reported in publications [1,2,35], based on turning of the NC6 grade tool steel of hardness 50 ±2 HRC with uncoated and PVD TiN coated inserts, made of composite oxide-carbide ceramics and cBN, have shown that coating deposition resulted in an increase of 50–90% in tool lives of the inserts. As a consequence of good functional properties achieved for ceramic inserts with single layer coating, further research work was carried out at Institute on ceramic coated inserts, regarding micro-scale and nano-scale multilayer coating deposition [36]. The present paper focuses on the results of investigations carried out in Institute, referring to the influence of nano-scale multi-layer nitride coatings on the properties of the coated inserts of composite oxide-carbide ceramics.

## 2. Experimental procedures

The coated and uncoated cutting inserts investigated, manufactured by Institute to the specification SNGN 120408 T02020, were of mixed ceramic types:

- TACN, comprising Al<sub>2</sub>O<sub>3</sub> with minor additions of ZrO<sub>2</sub> and up to 30% Ti(C,N),
- TW2, consisting of Al<sub>2</sub>O<sub>3</sub> and up to 30% TiC.

Using the Institute NNW-6.6 apparatus (type IIUHL4), the above-mentioned inserts were coated by the arc PVD method with nano and micro-layers of nitrides of the metals Ti and Zr and the alloy TiAl. The multilayer coatings on the inserts of type TACN and TW2 had the following structures:

- microlayer of Ti-Zr-N, alternately 10 nanolayers of TiN and ZrN,
- microlayer of (TiAl)N, alternately 10 nanolayers of TiN and (TiAl)N.

In order to determine the structures of the coatings, fractured inserts and metallographic sections, transverse and taper low-angle ( $5-6^\circ$ ), were observed optically and on the Japanese Jeol JSM-6460LV scanning electron microscope. The thicknesses of the multilayer coatings and of their constituent micro and nanolayers were determined on the fractured inserts using the SEM. To determine the total thickness of the coatings, the method of grinding a spherical indentation, followed by the microscopic examination of the resultant crater (PN-EN 1071-2:2004) was also employed.

Vickers microhardnesses of the coatings and substrates were determined on oblique metallographic sections using a digital microhardness tester type FM-7 (Future-Tech Corp.) using a load of 0.2452 N. Measurements of surface roughness were made using a Hommel Profilografometry Tester T1000.

For the cutting tests, the inserts were clamped in the CSRNL 3225-12 (Pafana Company) lathe tool holders giving tool angles: clearance angle  $\alpha_0 = 6^\circ$ , rake angle  $\gamma = -6^\circ$ . Inserts had also additional 0.2 mm wide chamfers of negative angle  $20^\circ$ . A PK420 type chip breaker was used. The workpiece was a 850 mm long cylindrical bar of 85 mm diameter of hardness  $50 \pm 2$  HRC. Workpiece material was the alloy tool steel NC6 with the following chemical composition: C 1.38%, Mn 0.6%, Si 0.25%, Cr 1.45%, V 0.18%, P and S max 0.03% and Fe balance. Bars were dry turned on a TZC-32N Universal type lathe, power of 50 kW at a cutting speed  $v_c = 150$  m/min, feed  $f = 0.10$  mm/rev, cutting depth  $a_p = 0.5$  mm.

Plan of the tool wear measurements encompassed several turning tests for each of the uncoated and coated inserts tested. It was assumed that the  $VB_{B_{max}}$  flank wear land length would be measured at time intervals of 5 min (according to PN-ISO 3685:1996).

### 3. Results and discussion

**3.1. Structure of the deposited coatings.** Scanning electron micrographs of fractures and metallographic cross-sections, of which examples are presented as Figs. 1–3, revealed the structures of multilayer coatings and enabled the evaluation of micro- and nano-layer thicknesses.

Figure 1 presents some examples of fractures of the coating deposited on the TACN grade insert; the following nitride layers (in order, starting from the substrate) can be observed:

- Ti-Zr-N micro-layer of thickness of  $1.8 \mu\text{m}$ ,
- ten nanolayers of TiN and ZrN, placed alternately, where these nanolayers were approximately 100 nm thick in the case of TiN and 108 nm in the case of ZrN.

In the Ti-Zr-N micro-layer many very thin sublayers can be observed; this structure was formed as a result of the coating

deposition technology applied, where two oppositely placed Ti and Zr cathodes were used and the substrates were placed on the rotating planetary table. These thin TiN and ZrN alternate sublayers (of thicknesses of about few nm) were seen on the metallographic low angle taper sections.

Figure 2 shows an example of SEM micrographs of fractures of coatings deposited on the TACN grade insert. This coating consisted of the following nitride layers:

- TiN micro-layer,  $0.8 \mu\text{m}$  thick,
- (TiAl)N micro-layer,  $0.7 \mu\text{m}$  thick,
- ten nanolayers of TiN and (TiAl)N deposited alternately, where each of the nanolayers was about 148 nm thick (in case of TiN) or 145 nm (in case of (TiAl)N).

In Fig. 3 the SEM micrographs are examples of the fracture of TiN coating deposited on the TW2 grade insert; the coating thickness is approx.  $3.2 \mu\text{m}$ . Before all the above coatings were deposited, a metallic titanium layer of several tens nm was deposited directly on the substrate surface. Total thicknesses of the coatings deposited on the ceramic inserts used in the cutting tests are given in Table 1. Differences in the coating thicknesses, especially in the case of the same type of inserts, resulted from the way in which the inserts have been clamped in the prototype instrument used in the coating deposition process; surfaces of the inserts were differently placed towards the cathodes and accordingly were coated with different intensities.

**3.2. Hardness of coatings and substrates.** In Table 2 the results of microhardness measurements are given; these measurements were performed on the metallographic taper sections of coated inserts of grades TACN and TW2. Statistical analysis, using the Student t-test for testing the hypothesis of the equality of average values, indicates that at the significance level  $\alpha = 0.05$ , the Ti-Zr-N/  $10 \times$  (TiN/ZrN) coating on TACN insert had a small, but significantly larger, microhardness value (mean 2872 HV 0.025) than the same type of coating on the TW2 insert (mean 2984 HV 0.025). At the same time the uncoated TACN insert had a much lower microhardness (mean 1896 HV 0.025) than the uncoated TW2 insert (mean 2305 HV 0.025). The microhardness of the TiN/(TiAl)N/  $10 \times$  (TiN/(TiAl)N) coating on the TW2 insert, 2966 HV 0.025, did not differ significantly from the microhardness of the coating of Ti-Zr-N/  $10 \times$  (TiN/ZrN) on the same type of insert.

**3.3. Surface roughness of coated inserts.** An increase in surface roughness of inserts was observed due to the deposition of the coatings, which is typical for arc PVD methods and is caused by the presence of micro-droplets (see Figs. 1c, 2 and 3). The results obtained are presented as Fig. 4. Since there was a large scatter in the values of the roughness parameter  $R_a$  for inserts TW2, only values for TACN inserts are reported. There was a large increase in the surface roughness of the smooth TACN inserts (mean  $R_a = 0.11 \mu\text{m}$ ), after coating the value increased by several times. For the case of TW2 inserts, whose surfaces were less smooth (mean  $R_a = 0.27 \mu\text{m}$ ), the roughness increased by 1.5–2.0 times (e.g. for TiN coated TW2, the mean  $R_a = 0.46 \mu\text{m}$ ).

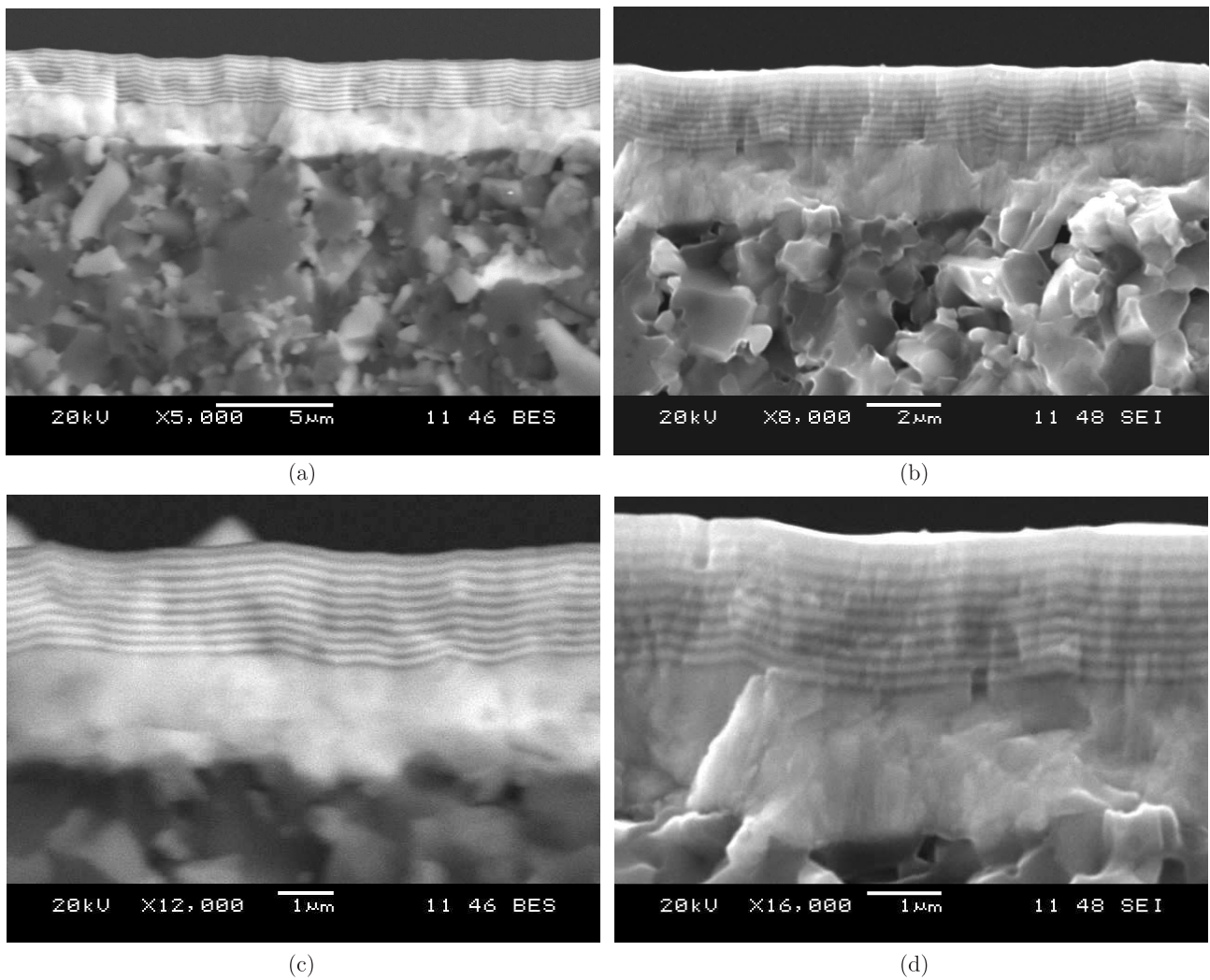


Fig. 1. SEM micrographs showing the fractured multilayer Ti-Zr-N/ 10× (TiN/ZrN) coating deposited on TACN grade ceramic cutting insert at different magnifications and types of electron image: 5000×, BES (a), 8000×, SEI (b), 12000×, BES (c) oraz 16000×, SEI (d)

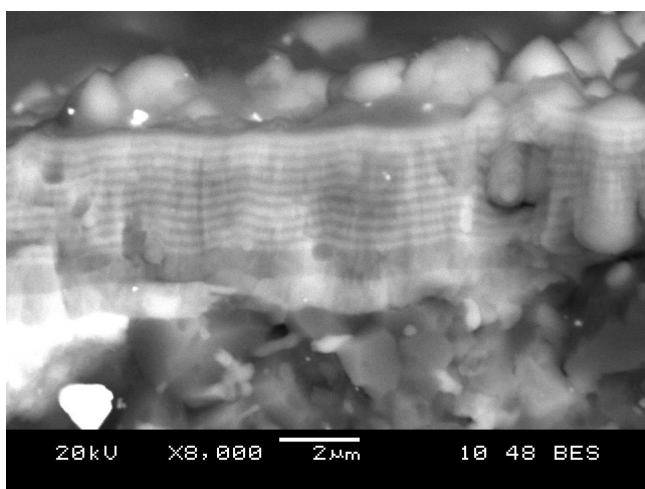


Fig. 2. A SEM micrograph showing the fractured multilayer TiN/(TiAl)N/ 10× (TiN/(TiAl)N) coating deposited on the TACN grade ceramic cutting insert

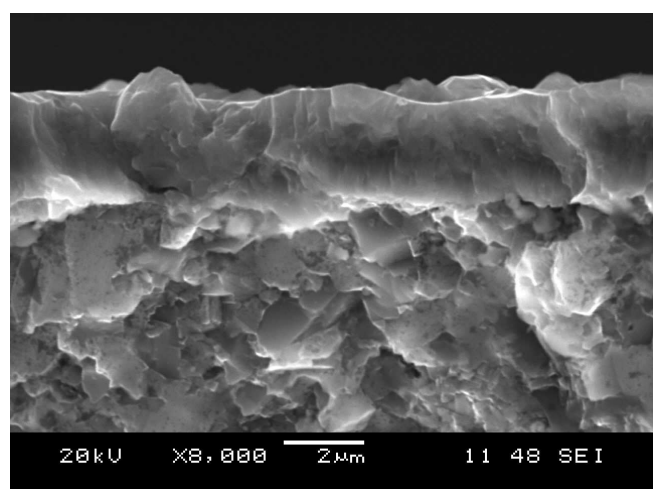


Fig. 3. A SEM micrograph showing the fractured TiN coating deposited on the TW2 grade ceramic inserts

Table 1  
Total thickness of the tested coatings on the substrates of oxide-carbide composite ceramics

Coated insert (insert, coating)	Measurements number	Coating thickness (μm)				
		Minimum value	Maximum value	Average value	Standard deviation	Confidence level at α = 0.05
TACN, Ti-Zr-N/ 10× (TiN/ZrN)	24 <sup>1,2</sup>	3.8	6.6	5.1	0.9	±0.4
TACN, TiN/(TiAl)N/ 10× (TiN/(TiAl)N)	21 <sup>1,2</sup>	4.3	6.1	5.0	0.5	±0.2
TW2, TiN	14 <sup>1,2</sup>	2.6	3.9	3.2	0.4	±0.2
TW2, Ti-Zr-N/ 10× (TiN/ZrN)	24 <sup>2</sup>	4.9	6.3	5.6	0.4	±0.4
TW2, TiN/(TiAl)N/ 10× (TiN/(TiAl)N)	24 <sup>2</sup>	4.5	6.3	5.4	0.6	±0.5

<sup>1</sup>Measurement from the fracture image using the SEM

<sup>2</sup>Microscopic measurement of spherical crater according to PN-EN 1071-2:2004

Table 2  
Results of microhardness measurements performed for coated and uncoated inserts of TACN and TW2 grades

Coated insert (insert, coating)	Measurements number	Microhardness HV 0.025				
		Minimum value	Maximum value	Average value	Standard deviation	Confidence level at α = 0.05
TACN, Ti-Zr-N/ 10× (TiN/ZrN)	65	2544	3480	2984	210.1	±52
TW2, Ti-Zr-N/ 10× (TiN/ZrN)	61	2507	3128	2872	166.1	±43
TW2, TiN/(TiAl)N/ 10× (TiN/(TiAl)N)	4	2692	3297	2966	265.2	±422
TACN, uncoated	4	1724	2019	1896	139.8	±222
TW2, uncoated	3	2116	2434	2305	167.3	±416

Table 3  
Results of turning tests for determination of tool lives T of coated and uncoated inserts

Coated insert (insert, coating)	Tests number	Tool life T (min)				
		Minimum value	Maximum value	Average value	Standard deviation	Confidence level at α = 0.10
TACN, uncoated	3	38.3	55.0	44.4	9.2	±15.5
TACN, Ti-Zr-N/ 10× (TiN/ZrN)	3	30.0	33.3	31.9	1.7	±2.9
TACN, TiN/(TiAl)N/ 10× (TiN/(TiAl)N)	3	31.4	37.5	33.5	3.5	±5.9
TW2, uncoated	3	32.5	42.6	38.3	5.2	±8.8
TW2, TiN	3	45.0	52.5	49.2	3.8	±6.4
TW2, Ti-Zr-N/ 10× (TiN/ZrN)	3	55.0	72.5	64.3	8.8	±14.8
TW2, TiN/(TiAl)N/ 10× (TiN/(TiAl)N)	3	55.0	77.5	67.5	11.5	±19.3

Notice: The  $VB_{Bmax} = 0.4$  mm was established as the corresponding to the tool life T

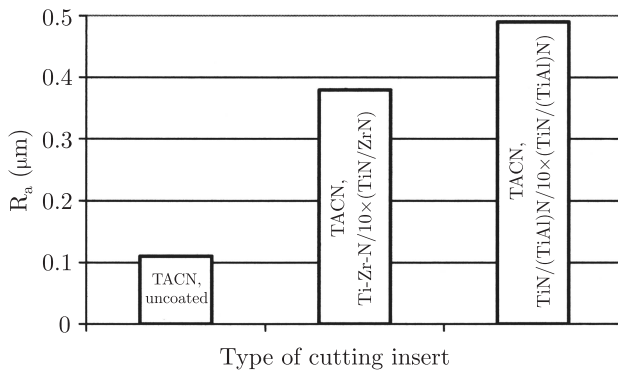


Fig. 4. Average values of roughness parameter  $R_a$  of the surfaces of TACN grade ceramic cutting inserts, coated and uncoated

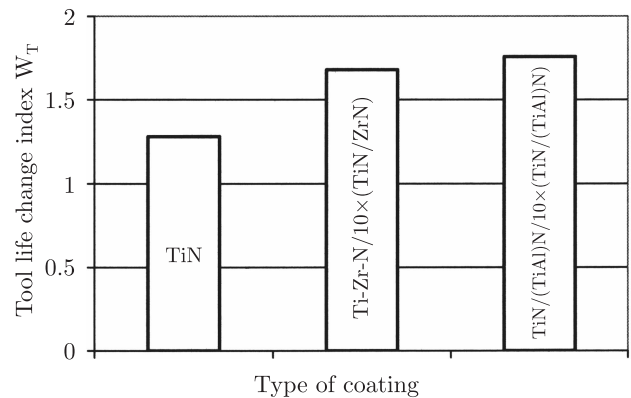


Fig. 5. Tool life change indices  $W_T$  of the coatings deposited on the TW2 grade ceramic cutting inserts



### 3.4. Influence of coatings on the tool lives of the inserts in turning.

The  $VB_{Bmax}$  wear land on the insert flank face was measured after every 5 min of turning time, up to the moment that it reached or exceeded 0.4 mm value, which was the established value for the tool life,  $T$ , determination in these tests. Turning test was repeated three times for each type of the insert tested. The average values of tool lives for uncoated and coated inserts are presented in Table 3, together with the confidence level at the significance level  $\alpha = 0.10$ . The analysis, which included testing of the hypothesis of the equality of average values using the Student's  $t$ -test (at  $\alpha = 0.10$ ), shows that, in respect of tool lives, the TW2 grade inserts with multi-layer Ti-Zr-N/ $10 \times$  (TiN/ZrN) and TiN/(TiAl)N/ $10 \times$  (TiN/(TiAl)N) coatings are significantly superior to all the other inserts tested, both coated and uncoated. The tool lives of these inserts were  $T = 55.0$ – $77.5$  min. The TiN coated TW2 grade inserts also show significantly higher tool lives ( $T = 45.0$ – $52.5$  min) than the uncoated TW2 inserts ( $T = 32.5$ – $42.5$  min), as well as the coated TACN inserts ( $T = 30.0$ – $37.5$  min). The negative effect of coating TACN inserts is surprising, especially as these inserts, when uncoated, exhibit similar tool lives ( $T = 38.3$ – $55.0$  min) as the uncoated TW2 grade inserts, or even slightly higher. There can be several reasons for the result obtained in the case of TACN inserts and accordingly some further investigations on the deposition of PVD coatings on ceramics will be carried out. To enable a comparison of the significant influence of different coatings on increases of tool life achieved for TW2 grade inserts, the  $W_T$  indices of tool life changes are presented in Fig. 5. The  $W_T$  indices are the ratios of tool lives of the coated inserts to the uncoated ones.

### 3.5. Workpiece surface quality after turning using uncoated and coated ceramic inserts.

Results of measurements of roughness parameters  $R_a$  of workpiece surface roughness after turning with uncoated and coated composite ceramics are given in Fig. 6. In spite of the large deterioration in the surface smoothness of the inserts resulting from coating (Fig. 4), there was little deterioration in the surface smoothness of the workpiece turned with these inserts.

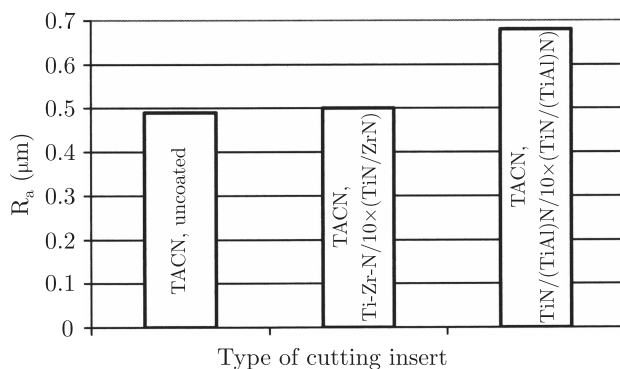


Fig. 6. Average values of roughness parameter  $R_a$  of the rollers machined with the TACN grade ceramic cutting inserts, coated and uncoated

## 4. Conclusions

It was shown that coating of oxide-carbide TW2 ceramic cutting inserts is beneficial regarding tool life, whereas the reverse has been found for ceramic inserts TACN. This is a very important observation, both regarding the economic range of application of coated ceramics and for indication of the direction of further research aimed at understanding the phenomena responsible for this state of affairs, determining the optimum, as regards tool life, method of coating of ceramic inserts (including TACN grade) and the selection of the most appropriate, regarding coating, substrate material.

The beneficial increase in tool life by multilayer coating of ceramic inserts, including nanoscale, provides the basis for further research in this area, including various compositions of the coated layers. The values of the roughness parameter  $R_a$  determined on surfaces of workpieces turned with ceramic inserts, only slightly inferior to those turned with uncoated inserts, indicate that, for a specific range of cutting parameters, it is possible to meet the requirements of the quality of surface finish, even with a much inferior surface smoothness of the coated cutting inserts. It is required simultaneously for Institute to ameliorate the arc PVD method, so as improve the surface smoothness of the deposited coatings.

Use will be made by Institute of the results now presented in manufacturing ceramic cutting inserts; the longer lifetimes resulting from their PVD coating should prove beneficial in specific industrial applications.

**Acknowledgements.** The authors wish to express their thanks to Professor J. Kozak of Warsaw Technical University and to Mr. J. Barcentewicz, Director of the Institute of Advanced Manufacturing Technology, for their encouragement to perform these studies and to prepare the paper. The authors wish to acknowledge the help provided by: Mr. K. Hubner of the Institute of Foundry and Mr. J. Danielak in preparing metallographic sections, Mrs. T. Miller and Mr. M. Korpala for roughness measurements and Mr. A. Fedaczyński for ball-cratering the inserts, of our Institute.

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