Hard, wear resistant coatings deposited on tool materials

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**Aim of the work**

The aim of this work is to investigate the structure and properties of multilayer coatings type of TiC, Ti(C,N), Al$_2$O$_3$, TiN obtained in the CVD and PVD processes and manufactured in the PVD process complex multi-component coatings of TiN+TiAlSiN+TiN, TiN+multiTiAlSiN+TiN and TiN+TiAlSiN+AlSiTiN onto silicon nitride (Si$_3$N$_4$) ceramics tool and compare them with commercial tool materials, coated in the CVD processes by multilayer coatings of Al$_2$O$_3$+TiN wear resistantce.
Thesis work

Producing of multi-layer coatings type of \( \text{Al}_2\text{O}_3 + \text{TiN} \) and \( \text{TiN} + \text{Al}_2\text{O}_3 \) in the CVD processes onto silicon nitride (\( \text{Si}_3\text{N}_4 \)) ceramics can increase utility properties of cutting tools coated by them machining tools in comparison with multilayer coatings with an intermediate of TiC and Ti (C,N) are produced in the CVD process and the multi-component coatings type of TiAlSiN obtained in the PVD process, especially destined for the implementation of modern machining technologies, including high-speed machining with high speeds, precision machining and dry cutting without employment of any cutting fluids.
# Materials

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Coatings</th>
<th>Coating thickness, µm</th>
<th>Process type</th>
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</thead>
<tbody>
<tr>
<td>Si$_3$N$_4$ nitride tool ceramics</td>
<td>TiN</td>
<td>0.8</td>
<td>PVD</td>
</tr>
<tr>
<td></td>
<td>TiN+multiTiAlSiN+TiN</td>
<td>4.0</td>
<td>PVD</td>
</tr>
<tr>
<td></td>
<td>TiN+TiAlSiN+TiN</td>
<td>2.0</td>
<td>PVD</td>
</tr>
<tr>
<td></td>
<td>TiN+TiAlSiN+AlSiTiN</td>
<td>2.5</td>
<td>PVD</td>
</tr>
<tr>
<td></td>
<td>Ti(C,N)+TiN</td>
<td>4.2</td>
<td>CVD</td>
</tr>
<tr>
<td></td>
<td>Ti(C,N)+Al$_2$O$_3$+TiN</td>
<td>9.5</td>
<td>CVD</td>
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<td></td>
<td>TiC+TiN</td>
<td>5.4</td>
<td>CVD</td>
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<tr>
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<td>CVD</td>
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<td>CVD (1)*</td>
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<tr>
<td></td>
<td>Al$_2$O$_3$+TiN</td>
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<td>CVD (2)*</td>
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<td>Al$_2$O$_3$+TiN</td>
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<td>CVD (3)*</td>
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<td>TiN+Al$_2$O$_3$+TiN+Al$_2$O$_3$+TiN</td>
<td>4.5</td>
<td>CVD (4)*</td>
</tr>
</tbody>
</table>

*(1) to (4) commercially available inserts from various manufacturers
Chemical Vapour Deposition (CVD)

based on Sandvik Baildonit
Methodology of researches

- Examinations of the structure:
  - The Scanning Electron Microscopy (SEM);
  - The Transmission Electron Microscopy (TEM).

- The study of chemical composition and phase:
  - X-ray of qualitative and quantitative microanalysis;
  - Optical emission spectrometry (GDOS);
  - X-ray qualitative phase analysis;
  - Texture analysis;
Methodology of researches

➢ Research of the mechanical properties:
  ✓ Roughness;
  ✓ Dynamic hardness;
  ✓ Internal stresses;
  ✓ Adhesion - „scratch test”;

➢ Research of the exploitative properties:
  ✓ Wear resistance – „pin-on-disc”;
  ✓ Try cutting cast iron and nickel alloy
Structure of nitride ceramics tool $\text{Si}_3\text{N}_4$

<table>
<thead>
<tr>
<th>Element</th>
<th>Mass concentration of elements, %</th>
<th>Atomic concentration of elements, %</th>
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<tbody>
<tr>
<td>N</td>
<td>60</td>
<td>57.04</td>
</tr>
<tr>
<td>Si</td>
<td>40</td>
<td>42.96</td>
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<tr>
<td>Sum</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

$\beta$-Si$_3$N$_4$[001]
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**X-ray qualitative phase analysis**

![X-ray diffractogram](image)

Reflection angle $\Theta$

Radiation intensity

- TiC
- Ti(C,N)
- Al₂O₃
- TiN
The chemical composition of the coatings

Ti(C,N) + Al₂O₃ + TiN

<table>
<thead>
<tr>
<th>Element</th>
<th>Mass concentration of elements, %</th>
<th>Atomic concentration of elements, %</th>
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</thead>
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<tr>
<td>C</td>
<td>9.72</td>
<td>20.21</td>
</tr>
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<td>N</td>
<td>22.03</td>
<td>40.29</td>
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<tr>
<td>Ti</td>
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<td>39.50</td>
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<tr>
<td>Sum</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

area X1

area X2

<table>
<thead>
<tr>
<th>Element</th>
<th>Mass concentration of elements, %</th>
<th>Atomic concentration of elements, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>42.86</td>
<td>55.89</td>
</tr>
<tr>
<td>Al</td>
<td>57.14</td>
<td>44.11</td>
</tr>
<tr>
<td>Sum</td>
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<td>100</td>
</tr>
</tbody>
</table>
Structure of PVD coatings

- TiN + multiTiAlSiN + TiN
- TiN + TiAlSiTiN + TiN
- TiN + TiAlSiN + AlSiTiN
- TiN
Structure of CVD coatings

- TiC+Ti(C,N)+Al₂O₃+TiN
- TiC+TiN
- Ti(C,N)+TiN
- TiN+Al₂O₃
Texture of coatings

Exemplary inverse for TiN layer representing the distribution of normal to the TiN+Al₂O₃+TiN+-Al₂O₃+TiN coating surface in the (001)-(011)-(111) base triangle (KN - normal direction)

Exemplary inverse for TiN layer representing the distribution of normal to the TiN+TiAlSiN+TiN coating surface in the (001)-(011)-(111) base triangle (KN - normal direction)
Structure of the TiN+Al<sub>2</sub>O<sub>3</sub>+TiN+Al<sub>2</sub>O<sub>3</sub>+TiN coating

Cross-section perpendicular to a layer surface
Structure of the \( \text{TiN} + \text{Al}_2\text{O}_3 \) coating

The transition zone

Cross-section perpendicular to a layer surface
Structure of the TiN+Al$_2$O$_3$ coating

Cross-section perpendicular to a layer surface

Dark field of reflexes (113) Al$_2$O$_3$
Structure of the TiN+Al$_2$O$_3$+TiN+Al$_2$O$_3$+TiN coating

Cross-section perpendicular to a layer surface
The chemical composition of coatings

TiN+multiTiAlSiN+TiN

TiC+Ti(C,N)+Al₂O₃+TiN

Analysis depth, µm
Mass concentration, %
Coatings adhesion

Coating:

- TiN+TiAlSiN+AlSiTiN
- TiN+TiAlSiN+TiN
- TiN+multiTiAlSiN+TiN
- TiN
- TiN+Al2O3+TiN+Al2O3+TiN
- Al2O3+TiN (Sandvik)
- Al2O3+TiN (Iscar)
- TiN+Al2O3+TiN
- TiN+Al2O3
- TiC+Ti(C,N)+Al2O3+TiN
- TiC+TiC
- Ti(C,N)+Al2O3+TiN
- Ti(C,N)+Ti

Critical load, Lc, N

Load range 0-100 N
Load increase rate (dL/dt) 100 N/min
Penetrator’s travel speed (dx/dt) 10 mm/min
Acoustic emission detector’s sensitivity AE 1

- 18.3±22.4 N
- 26.5±47.7 N
- 26.7±83.1 N
Coatings adhesion

TiN+Al₂O₃
Structure of the TiN+multiTiAISiN+TiN coating

Cross-section parallel to a layer surface

Dark field of reflexes (111) TiN
Structure of the TiN+Al₂O₃ coating

Cross-section parallel to a layer surface

Dark field of reflexes (111) TiN
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Structure of the TiN+Al$_2$O$_3$+TiN+Al$_2$O$_3$+TiN coating

Cross-section parallel to a layer surface
Surface topography of PVD coatings

**Element** | **Mass concentration of elements,%** | **Atomic concentration of elements, %**
---|---|---
N | 16.24 | 39.86
Ti | 83.76 | 60.14
Sum | 100 | 100

TiN+multiTiAlSiN+TiN

area X1
Surface topography of CVD coatings
Coatings roughness

Coating:

- TiN+TiAlSiN+AlSiTiN
- TiN+TiAlSiN+TiN
- TiN+multiTiAlSiN+TiN
- TiN+Al2O3+TiN+Al2O3+TiN
- Al2O3+TiN (Sandvik)
- Al2O3+TiN (Iscar)
- TiN+Al2O3+TiN
- TiN+Al2O3
- TiC+Ti(C,N)+Al2O3+TiN
- TiC+TiN
- Ti(C,N)+Al2O3+TiN
- Ti(C,N)+TiN
- uncoated

**Roughness Ra, µm**

- TiN+TiAlSiN+AlSiTiN: 0.3 µm
- TiN+TiAlSiN+TiN: 0.4 µm
- TiN+multiTiAlSiN+TiN: 0.35 µm
- TiN+Al2O3+TiN+Al2O3+TiN: 0.6 µm
- Al2O3+TiN (Sandvik): 0.25 µm
- Al2O3+TiN (Iscar): 0.25 µm
- TiN+Al2O3+TiN: 0.25 µm
- TiN+Al2O3: 0.6 µm
- TiC+Ti(C,N)+Al2O3+TiN: 0.25 µm
- TiC+TiN: 0.25 µm
- Ti(C,N)+Al2O3+TiN: 0.25 µm
- Ti(C,N)+TiN: 0.25 µm
- uncoated: 0.06 µm

*measuring length l = 0.25 mm, measuring accuracy = 0.01 µm*
Coatings hardness

Coating:

- TiN+TiAlSiN+AlSiTiN
- TiN+TiAlSiN+TiN
- TiN+multiTiAlSiN+TiN
- TiN
- TiN+Al2O3+TiN+Al2O3+TiN
- Al2O3+TiN (Sandvik)
- Al2O3+TiN (Iscar)
- TiN+Al2O3+TiN
- TiN+Al2O3
- TiC+Ti(C,N)+Al2O3+TiN
- TiC+TiN
- Ti(C,N)+Al2O3+TiN
- Ti(C,N)+TiN
- uncoated

Hardness, GPa

Load, F = 70 mN

Coatings hardness:
- TiN+TiAlSiN+AlSiTiN: 35.24 GPa
- TiN+TiAlSiN+TiN: 32.57 GPa
- TiN+multiTiAlSiN+TiN: 18.50 GPa
- TiN: 18.50 GPa
- TiN+Al2O3+TiN+Al2O3+TiN: 35.24 GPa
- Al2O3+TiN (Sandvik): 32.57 GPa
- Al2O3+TiN (Iscar): 18.50 GPa
- TiN+Al2O3+TiN: 35.24 GPa
- TiN+Al2O3: 32.57 GPa
- TiC+Ti(C,N)+Al2O3+TiN: 18.50 GPa
- TiC+TiN: 18.50 GPa
- Ti(C,N)+Al2O3+TiN: 35.24 GPa
- Ti(C,N)+TiN: 32.57 GPa
- uncoated: 18.50 GPa

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Coatings stresses

Coating:

- TiN+Al2O3+TiN+Al2O3+TiN
- Al2O3+TiN (Iscar)
- TiN+Al2O3+TiN
- TiN+Al2O3
- TiC+TiN
- Ti(C,N)+Al2O3+TiN
- Ti(C,N)+TiN

Stress, MPa

- TiN+Al2O3+TiN+Al2O3+TiN: 1008 MPa
- Ti(C,N)+Al2O3+TiN: 299 MPa
Coatings wear resistance

- Ti(N,C)+TiN
  - Number of cycles: 15000

- TiN+Al₂O₃+TiN
  - Number of cycles: 11000
Coatings wear resistance

Coating:

- TiN+TiAlSiN+AlSiTiN
- TiN+TiAlSiN+TiN
- TiN+multiTiAlSiN+TiN
- TiN
- TiN+Al2O3+TiN+Al2O3+TiN
- Al2O3+TiN (Sandvik)
- Al2O3+TiN (Iscar)
- TiN+Al2O3+TiN
- TiN+Al2O3
- TiC+Ti(C,N)+Al2O3+TiN
- TiC+TiN
- Ti(C,N)+Al2O3+TiN
- Ti(C,N)+TiN

Number of cycles

Pressure force, $F_N = 5$ N
Linear velocity, $v = 0.1$ m/sec
Friction radius, $r = 5$ mm
Counter-specimen – WC

Number of cycles:
- TiN+TiAlSiN+AlSiTiN: 500\(\pm\)2000
- TiN+TiAlSiN+TiN: 5000\(\pm\)11000
- TiN+multiTiAlSiN+TiN: 10000\(\pm\)25000
Coatings wear resistance

Ti(C,N)+Al₂O₃+TiN
number of cycles 25000

TiN
number of cycles 2000
Cutting properties

Machining time, min

Tool live VB, mm

Si3N4  TiN+Al2O3

TiN+Al2O3 machining time $t = 8$ min
VB = 0.15 mm
Cutting properties

- TiN + Al₂O₃ + TiN + Al₂O₃ + TiN
- Ti(C,N) + TiN
- TiN + multiTiAlSiN + TiN
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Hard, wear resistant coatings deposited on tool materials

Cutting properties

Coating:
- TiN+TiAlSiN+Al2O3+TiN
- TiN+TiAlSiN+TiN
- TiN+multiTiAlSiN+TiN
- TiC+TiN
- TiN+Al2O3+TiN+Al2O3+TiN
- Al2O3+TiN (Sandvik)
- Al2O3+TiN (Iscar)
- TiN+Al2O3+TiN
- TiN+Al2O3
- TiC+Ti(C,N)+Al2O3+TiN
- TiC+TiN
- Ti(C,N)+Al2O3+TiN
- Ti(C,N)+TiN
- uncoated

Cutting speed, \( v_c = 400 \text{ mm/min} \)
Feed rate, \( f = 0.2 \text{ mm/rev} \)
Depth of cut, \( a_p = 2 \text{ mm} \)
Criterion of the cutting edge consumption evaluation, \( VB = 0.3 \text{ mm} \)
Machining time = 8 min

Tool live VB, mm
Cutting properties

Coating:
- TiN+TiAlSiN+AlSiTiN
- TiN+TiAlSiN+TiN
- TiN+multiTiAlSiN+TiN
- TiC+TiN
- TiC+Ti(C,N)+Al2O3+TiN
- TiN+Al2O3
- TiN+Al2O3+TiN
- Al2O3+TiN (Iscar)
- Al2O3+TiN (Sandvik)
- TiN+Al2O3+TiN
- TiN+Al2O3+TiN+Al2O3+TiN
- TiN
- TiN+multiTiAlSiN+TiN
- TiN+TiAlSiN+TiN
- TiN+TiAlSiN+AlSiTiN
- uncoated

Roughness Ra, μm

5.6 μm
2.2 μm
Cutting properties

TiN+multiTiAlSiN+TiN
machining time = 1.5 min
VB = 0.42 mm

TiN+Al₂O₃
machining time = 0.5 min
VB = 0.98 mm
Conclusions

1. It has been proven stated thesis of the work pointing out that: Producing of multi-layer coatings type of $\text{Al}_2\text{O}_3+\text{TiN}$ and $\text{TiN}+\text{Al}_2\text{O}_3$ in the CVD processes onto silicon nitride ($\text{Si}_3\text{N}_4$) ceramics can increase utility properties of cutting tools coated by them machining tools in comparison with multilayer coatings with an intermediate of $\text{TiC}$ and $\text{Ti (C,N)}$ are produced in the CVD process and the multi-component coatings type of $\text{TiAlSiN}$ obtained in the PVD process, especially destined for the implementation of modern machining technologies, including high-speed machining with high speeds, precision machining and dry cutting without coolants.
Conclusions

2 All PVD and CVD coatings deposited onto the nitride tool ceramics are characterized by a structure without pores and discontinuities and by tight adherence to themselves and of the entire multilayer coating to the substrate. The comprehensive examinations on the transmission electron microscope (thin foil perpendicular to the layer surface) make it possible to reveal the columnar structure for the TiN layer included in the TiN+Al₂O₃.
Conclusions

These examinations have revealed also that there is an interface between the TiN and Al$_2$O$_3$ layers in case of the TiN+Al$_2$O$_3$ coating, where the fine grains of these phases are found. Occurrences of the scarce fine-grained Al$_2$O$_3$ grains with the monoclinic structure were revealed in this zone, unlike the typical structure of the Al$_2$O$_3$ phase with the trigonal lattice, which occurs outside of this border area over the entire layer width. The observed interface zone contributes to good adhesion between these layers, which is also confirmed by high abrasion wear resistance of this coating.
Conclusions

Evaluating differences of textures of the TiN layers obtained with the PVD and CVD methods one can state that – in the most general understanding – the method of physical deposition from the gaseous phase favours development of the relatively strong texture. It is the \{111\} + \{001\} double texture most often; however the \{001\} component is usually stronger.
Conclusions

Surface layer hardness increase, compared to the uncoated substrate hardness can reach even 100%. Hardness of the investigated coatings systems determines their abrasion wear resistance, which was revealed most clearly for the TiN+ Al₂O₃ coating - the hardest one from the CVD coatings, adding simultaneously to the decrease of the cutting tools edge wear intensity during machining.
Conclusions

The CVD coatings, compared to the PVD ones, are characteristic of the very good adherence to the nitride ceramics substrate, which is decided not only by adhesion, but also the diffusion mixing of elements in the interface between the substrate and the coating, and in case of the TiN+Al_2O_3 double-layer coating – mixing of phases in the interface between the particular layers, which – even at the highest load – does not cause total delamination of any coating in the adhesion tests.
Conclusions

The abrasive wear resistance tests made with the „pin-on-disk” method revealed that the PVD coatings have worse tribological properties compared to those put down with the CVD method. In nearly all cases damage of coatings occurs up to the Si₃N₄ nitride ceramics substrate zone. Hardness of the investigated coating systems determines their abrasion wear resistance, which was revealed most clearly for the Al₂O₃+TiN coating – the hardest among the CVD ones, contributing at the same time to decreasing the cutting tool edge wear intensity during machining.
Conclusions

High hardness, good adhesion, and very good abrasion wear resistance - especially noticeable for the nitride ceramics with the Al₂O₃+TiN combination of layers and with the TiN+Al₂O₃ coating, increase the cutting tool flank life and therefore, and also because of the possibility of their use in the pro-ecological dry-cutting processes, without employment of any cutting fluids, make these coatings suitable for many industrial applications on cutting tools.
Acknowledgements

The research was carried out partially in cooperation with Doc. Dr Ing. Antonin Kriz from University of West Bohemia, Plzen and M. Sima from SHM-Šumperk (Czech Republic).
Thank you for your kind attention!