The Tribological Properties of Titanium Carbonitride TiCN Coating Lubricated with Non-Toxic Cutting Fluid

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Abstract: This paper presents the results of tribological studies of TiCN coatings formed on the HS6-5-2C steel by plasma-assisted chemical vapour deposition (PVD). The coating thickness was measured with the optical microscope. The surface texture analysis was performed before and after the tribological tests with a Talysurf CCI Lite optical profiler. The tribological properties were investigated using a T-01M tribotester for a ball-on-disc configuration in the sliding contact. The tests were carried out under lubricated friction conditions using lubricants. Cutting fluids and coatings have contributed to the reduction of resistance movement. The non-toxic cutting fluid has reached comparable coefficient of friction with a coolant containing mineral oil.

Introduction

Technological developments have created the need for coatings that meet demanding applications of advanced materials. Single layer and multilayer coatings, widely used especially on cutting tools [1-3], increase the quality of machined parts and enable high speed cutting [1]. Titanium carbonitride (TiCN) coating is characterized by high hardness and low friction, can be applied by physical deposition (PVD) at temperatures ranging from 300 to 450°C [3], exhibits high melting temperature [4], anti-wear behaviour, chemical stability and corrosion resistance [5, 6].

The effect of tools with coatings on the quality of surface treated and durability of the tool were examined [7, 8] to find that coated tools had lower wear than conventional high-speed steel tools. Two studies [9-11] describe experiments that involved machining with biodegradable, non-toxic coolants based on plant oils and oil-containing cutting fluids, in which the roughness of work materials and tool wear were compared. The results showed that the properties of plant oil coolants were comparable to or better than the properties of coolants containing mineral oil.

To reduce tool wear and improve the surface quality of a workpiece, cutting tool lubricants are used during machining for cooling, lubricating, transporting chips from the machining zone, reducing the coefficient of friction, and temporary protection against corrosion [12-14]. Lubricants are applied in over 80% of metalworking operations [15]. Widely used mineral oil-based coolants are usually toxic. Plant oils have become a renewable, hazard-free and easily biodegradable alternative, characterized by good lubricity, low volatility, higher fire resistance and good thermal stability. Currently, plant oil based metal working fluids are the area of extensive research [16-18].

Materials and methods

The tests were performed on a TiCN coating deposited on the HS6-5-2C high-speed tool steel by the PVD. The surface and cross-sections of the TiCN coating as well as its chemical analysis in the
micro-regions were carried out using a Jeol JSM 7100F field emission scanning electron microscope with an EDS analyser. A Talysurf CCI Lite interferometer was used to analyse the geometric structure of the specimen and counter-specimen surfaces before and after the tribological tests [16].

The tribological tests were carried out on a T-01M ball (100Cr6 steel, Ø = 10 mm)-on-disk (HS6-5-2C steel without coating or with the TiCN coating, Ø = 38 mm) testing machine in accordance with the requirements of ASTM G 99. Friction tests were performed at a load of P = 50 N, a sliding speed of v = 0.1 m / s, along the friction path s = 1000 m, at the constant relative humidity of 40 ± 5% and ambient temperature T0 = 23 ± 1°C under technically dry friction, lubricated friction with a mineral oil-based coolant and with a non-toxic coolant, consisting of alkanolamine borate, a biodegradable polymer - zinc aspartate and DEMI demineralised water.

**Test results**

Figure 1a shows an image of the TiCN coating surface morphology. Figures 1b and 1c show the results of elemental analysis in the micro-regions of the coating.

![Image of TiCN coating surface morphology](image1.png)

**Fig. 1. SEM: a) view of the disk area and the EDX radiation spectrum with the result of the chemical analysis for the TiCN coating: b) at point 1, c) at point 2.**

The elemental analysis in the micro-region of the TiCN coating (Fig. 1b) showed the presence of titanium, nitrogen and carbon as components of the coating and iron from the substrate. In the image of the surface morphology (Fig. 1a), droplet shaped particles of various sizes were observed. They were most likely formed during the process of coating deposition as a result of titanium droplets splashing on the surface of the substrate. The analysis of the chemical composition showed that in addition to elements such as titanium, nitrogen and carbon, oxygen and silicon were also present (Fig. 1c). In the following testing stage, metallographic specimens were made, cross-sections of the coating were observed and its thickness of approx. 2 μm, was measured.
Figure 2 shows the force-penetration depth changes and the view of the impression in the TiCN coating on the HS6-5-2C steel after the nanohardness measurement. The nanohardness results are summarized in Table 1.

![Figure 2](image.png)

**Fig. 2. Force - penetration depth changes in the TiCN coating and the microhardness impression.**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>$H_{IT}$, [GPa]</th>
<th>$E_{IT}$, [GPa]</th>
<th>$E^*$, [GPa]</th>
<th>$H_{VIT}$, [HV]</th>
<th>$C_{IT}$, [%]</th>
<th>$F_{max}$, [mN]</th>
<th>$h_{max}$, [mm]</th>
<th>$S$, [mN/mm]</th>
<th>$A_p$, [mm$^2$]</th>
<th>$W_{elast}$, [nJ]</th>
<th>$W_{plast}$, [nJ]</th>
<th>$W_{total}$, [nJ]</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiCN</td>
<td>38.18</td>
<td>311.57</td>
<td>342.38</td>
<td>3 535.70</td>
<td>0.36</td>
<td>49.99</td>
<td>0.32</td>
<td>352</td>
<td>1.31</td>
<td>4</td>
<td>1</td>
<td>5</td>
</tr>
</tbody>
</table>

The analysis of the loading-unloading curve indicates elastic properties of the TiCN coating, as can also be deduced from the values of the elastic modulus $E_{IT} = 311.57$ GPa and the reduced elastic modulus $E^* = 342.38$ GPa. Hardness measured by instrumental indentation according to the Oliver and Pharr method equals 38.18 GPa and the Vickers hardness equals 3,535.70 HV. The TiCN coating is characterized by a small creep module $C_{IT}$ - 0.36%, a small contact area $A_p$ - 1.31 mm$^2$, plasticity - 1 nJ and 4 times higher elasticity - 4 nJ.

Figure 3 shows the coefficients of friction recorded during technically dry friction and lubricated friction with a mineral oil-based coolant and coolants with zinc aspartate for the HS6-5-2C steel - 100Cr6 steel, and HS6-5-2C steel with the TiCN coating - 100Cr6 steel pairs.

![Figure 3](image.png)

**Fig. 3. Mean coefficients of friction for the friction pairs tested under technically dry friction and with the use of coolants.**
The use of coolants reduced the values of mean coefficient of friction. The lowest mean friction coefficients of 0.11 were recorded for the TiCN coating under lubricating conditions both with mineral oil and zinc aspartate based working fluids. For the HS6-5-2C steel disk, the mineral oil-containing coolant turned out to be better. It can therefore be concluded that the use of zinc aspartate as a lubricant in combination with the TiCN coating does not reduce the coefficient of friction but reduces the effect of toxic agents contained in mineral oils.

Figures 4 ÷ 7 show the topography and primary profiles of disks after friction tests.

**Fig. 4.** Geometric structure of the HS6-5-2C steel disk: a) isometric image, b) primary profile after friction using a coolant containing mineral oil.

<table>
<thead>
<tr>
<th>Max. depth, [nm]</th>
<th>Max. height, [nm]</th>
<th>Hole field, [μm²]</th>
<th>Area outside, [μm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>80,717</td>
<td>21,014</td>
<td>4,012</td>
<td>2,176</td>
</tr>
</tbody>
</table>

**Fig. 5.** Geometric structure of the HS6-5-2C steel disk with the TiCN coating: a) isometric image, b) primary profile after friction using a coolant containing mineral oil.

<table>
<thead>
<tr>
<th>Max. depth, [nm]</th>
<th>Max. height, [nm]</th>
<th>Hole field, [μm²]</th>
<th>Area outside, [μm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>262,740 nm</td>
<td>226,250 nm</td>
<td>13,876 μm²</td>
<td>14,072 μm²</td>
</tr>
</tbody>
</table>

**Fig. 6.** Geometric structure of the HS6-5-2C steel disk: a) isometric image, b) primary profile after friction using a coolant containing zinc aspartate.

<table>
<thead>
<tr>
<th>Max. depth, [nm]</th>
<th>Max. height, [nm]</th>
<th>Hole field, [μm²]</th>
<th>Area outside, [μm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>219,087 nm</td>
<td>10,152 nm</td>
<td>51,979 μm²</td>
<td>0,164 μm²</td>
</tr>
</tbody>
</table>
Fig. 7. Geometric structure of the HS6-5-2C steel disk with the TiCN coating: a) isometric image, b) primary profile after friction using coolant containing zinc aspartate.

The analysis indicates that the wear of friction pair material was lower when the coolant containing mineral oil was used. After the tribological tests under lubricated conditions with the mineral oil coolant, build-ups and recesses formed on the disks, whereas after the tests with the zinc aspartate coolant only recesses were observed.

Table 2 shows the disk and ball surface geometric structure parameters before and after the tribological tests with the working fluids used.

Table 2. Parameters of the surface geometric structure

<table>
<thead>
<tr>
<th>Test</th>
<th>DISC</th>
<th>Surface roughness parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Sa, [μm]</td>
</tr>
<tr>
<td>BEFORE TESTS</td>
<td>HS6-5-2C</td>
<td>0,04</td>
</tr>
<tr>
<td></td>
<td>TiCN</td>
<td>0,23</td>
</tr>
<tr>
<td>AFTER WET FRICTION WITH</td>
<td>HS6-5-2C</td>
<td>0,02</td>
</tr>
<tr>
<td>CUTTING FLUID CONTAINING MINERAL OIL</td>
<td>TiCN</td>
<td>0,07</td>
</tr>
<tr>
<td>AFTER WET FRICTION WITH</td>
<td>HS6-5-2C</td>
<td>0,03</td>
</tr>
<tr>
<td>ZINC ASPARTATE</td>
<td>TiCN</td>
<td>0,04</td>
</tr>
</tbody>
</table>

The analysis of the results shows that the uncoated HS6-5-2C steel disk has the lowest values of amplitude Sa, Sq, Sp, Sv, Sz parameters after the tests carried out under lubrication with the mineral oil coolant. This indicates a smooth surface. The distribution of ordinates of this surface is characterized by high asymmetry and steepness. In turn, the lowest values of the amplitude parameters, i.e., a smoother surface of the disk with the TiCN coating, were observed after the tests using the zinc aspartate based coolant.

Summary
The thin, hard, elastic TiCN coating used in the study, obtained in the PVD process and working in tribosystems perfectly fulfills its function, i.e., it contributes to the low coefficient of friction, as was confirmed by the results of tribological tests, where the lowest values were obtained for the configuration with a TiCN coated disk after friction with lubrication using both mineral oil and zinc aspartate working fluids.

In the analysis of the obtained geometric structure of the disk surface after tests with a coolant containing mineral oil, a smoother surface was observed in the HS-5-2C steel disk, and after lubrication with a zinc aspartate coolant in the disc with the TiCN coating.

Considering the results obtained, the coolant containing zinc aspartate works better with the TiCN coating. This proves that the zinc aspartate based coolant is in some cases a better choice than the mineral oil based working fluid, as it can limit the use of toxic, commonly used cutting fluids.
References


