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## **ANALYSIS OF THE TRIBOLOGICAL RESISTANCE OF COATINGS DEDICATED TO IMPROVING THE DURABILITY OF TOOLS USED IN THE GLASS FORMING PROCESS**

### **Key words**

Multilayer coating, tribological resistance, glass forging dies.

### **Abstract**

The article presents an analysis of the possibilities of using modern surface engineering solutions to improve the durability of the tools used in the glass forming process. These tools are exposed to the simultaneous effects of several destructive factors in the operating process, i.e. cyclic thermal shocks, the corrosive effects of molten glass, and the friction caused by the presence of

intensive sludge from molten glass remaining on the tool between the forming processes.

The article presents the results of the material and tribological properties of selected multilayer coatings. The research methods presented in the article focused on the analysis of mechanical properties, surface topography, as well as the wear properties of selected coatings. The studies on mechanical properties included tests on hardness and Young modulus using the nanoindentation method, and tests on adhesion were conducted using the scratch-test method.

## **Introduction**

The expectations of the industry towards new material solutions in the field of the surface engineering are increasingly directed to the manufacture of layers and coatings characterized by increasingly better strength properties at elevated temperatures. The article presents an analysis of the possibilities of using modern surface engineering solutions to improve the durability of the tools used in the glass forming process. Analysis of the operating conditions of these tools made it possible to identify three main destructive factors in the technological process, i.e. cyclic thermal shocks, the corrosive effects of molten glass, and the friction caused by the presence of intensive sludge from molten glass remaining on the tool between the forming processes. The most effective method of improving the durability of the tools used in the process of forming glass are the modern technology of surface engineering, due to the complex operating conditions, which are concentrated in the surface layer of the material. The transition metals' nitrides, such as TiN or CrN, are widely used as coatings that increase the durability tools and elements used in processes such as operations in plastic forming and machining [1,2]. However, the main disadvantage of these materials, considerably limiting their possibilities of application, is the low oxidation resistance. The state of the art indicated that the addition of elements such as Si, Al, and Cr [3], [4] to chemical the composition of the TiN and CrN coatings significantly improve their oxidation resistance at elevated temperatures. The growing use as wear resistant coatings applied in elevated temperatures are multi-layered coatings in the form of multicomponent nitrides produced on the basis of Ti, Al, Cr, and Si [5], [6].

The article presents the results of the properties of different multilayer coatings, i.e. Cr/CrN, Cr/CrN/AlCrTiN, and CrN/AlCrN/AlTiCrSiN. The research methods presented in the article focused on the analysis of mechanical properties, surface topography, and the wear properties of selected coatings. The studies on mechanical properties included tests on hardness and Young modulus using the nanoindentation method, and tests on adhesion were conducted using the scratch-test method.

## 1. Experimental details

### 1.1. Preparation of PVD coatings

Samples for investigations were made of martensitic steel H17N2 according to PN-71/H-08620 (C-0.20, Cr-17.0, Ni-2.0) as typical steel used for making tools in the glass industry. The investigated PVD coatings were created using the Arc Evaporation method carried out using technological devices produced by ITeE – PIB Radom. The Cr/CrN, Cr/CrN/AlCrTiN, and CrN/AlCrN/AlCrTiSiN multilayer coatings deposition process was executed. Using pure Cr targets for the creation of particular layers of Cr and CrN, the following component targets were used:

- 70% Al + 30% Cr + 30%Ti,
- 70% Al + 30% Cr + 30%Ti + 30%Si, and
- 70% Al + 30% Cr.

Parameters of the all surface treatment processes are presented in Table 1.

Table 1. Parameters of the Arc Evaporation technology

Coating	Temperature $T$ [°C]	Current $I$ [A]	$U_{bias}$ [V]	Time $t$ [min]	Pressure $p$ [mbar]	Atmosphere
Cr/ CrN	400	$6 \times 55$	-50	5	$5.0^{-3}$	Ar
	420	$6 \times 55$	-150	90	$3.5^{-2}$	N <sub>2</sub>
Cr/ CrN/ AlCrTiN	390	$6 \times 55$	-50	10	$5.0^{-3}$	Ar
	390	$2 \times 60$	-150	30	$3.5^{-2}$	N <sub>2</sub>
	400	$4 \times 60$	-100	60	$2.0^{-2}$	N <sub>2</sub>
CrN/ AlCrN/ AlTiCrSiN	390	$6 \times 55$	-50	10	$5.0^{-3}$	Ar
	390	$2 \times 60$	-150	30	$3.5^{-2}$	N <sub>2</sub>
	400	$4 \times 60$	-100	60	$2.0^{-2}$	N <sub>2</sub>

### 1.2. Characterization of material properties of PVD coatings

The created composites underwent investigations aimed at defining their materials properties. The hardness and Young's modulus of each investigated PVD coating was measured by means of the nanoindentation method using a Nano-Hardness Tester from CSM Instruments. Measurements were carried out with the Berkovich indenter in a single cycle using the following parameters:  $F = 10$  mN,  $dF/dt = 20$  mN/min.

### 1.3. Characterization of the tribological properties

The tribological tests were executed using the ball-on-disc tribometer designed by DUCOM Instruments with a sodium-calcium ball  $\varnothing = 10$  mm (chemical composition of glass ball:  $\text{SiO}_2$  72%;  $\text{Na}_2\text{O}$  13.8%;  $\text{K}_2\text{O}$  0.1%;  $\text{CaO}$  9.0%;  $\text{MgO}$  4.0%;  $\text{Al}_2\text{O}_3$  1.0%;  $\text{Fe}_2\text{O}_3$  0.1%) according to the following parameters: the ball load forces  $F = 2$  N, temperature  $T_1 = 500^\circ\text{C}$ , the sliding distance  $s = 1000$  m, and the velocity  $V = 0.1$  m/s. During the tests, the friction coefficient was measured. After the tests, the wear tracks were analysed with the use of a MG140 confocal profilometer by DUCOM and Surface Imaging & Metrology Software MountainsMap 7.0. Based on the volume of removed coating material, the wear index  $W$  was calculated, according to the following formula (1):

$$W = V/(F \cdot s) \quad (1)$$

where

$V$  – volume of removed coating material [ $\text{mm}^3$ ],

$F$  – ball load force [N], and

$s$  – length of wear track [km].

## 2. Results

### 2.1. Characterization of PVD coatings

According to the assumptions, three multi-coatings were produced. Figure 1 shows images of the surface and cross sections of the tested coatings. The basic mechanical properties of the deposited PVD coatings are presented in Table 2.

Table 2. Properties of the deposited PVD coatings

Coating	Thickness $g$ [ $\mu\text{m}$ ]	Hardness [GPa]	Young modulus [GPa]	Roughness $Ra/Rz/Rt$ [ $\mu\text{m}$ ]
Cr/CrN	5.73	$27 \pm 1.5$	$299 \pm 18$	0.15/1.86/2.74
Cr/CrN/AlCrTiN	4.4	$30 \pm 2.1$	$337 \pm 11$	0.44/4.02/5.39
CrN/AlCrN/AlTiCrSiN	4.9	$28 \pm 2.5$	$322 \pm 34$	0.24/2.73/0.26

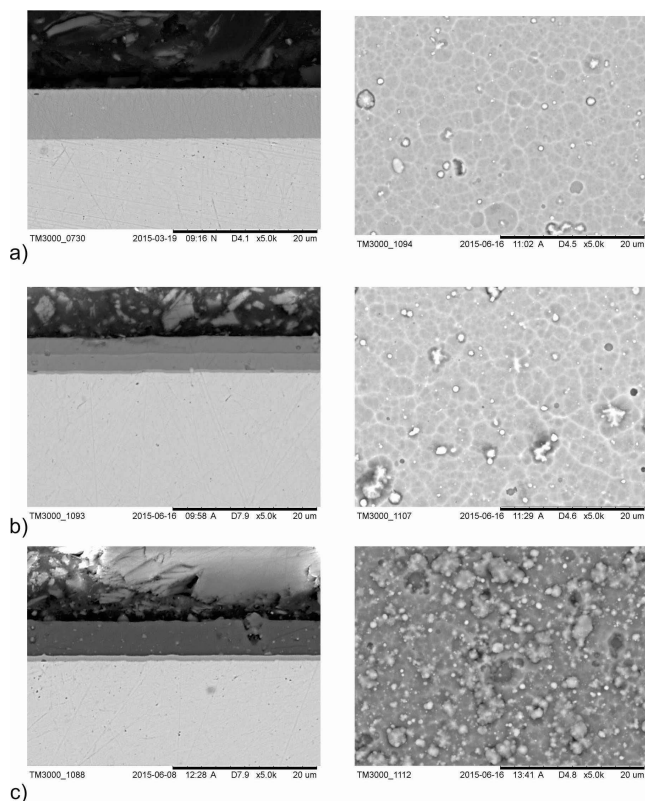


Fig. 1. Images of the surface and cross-sections of the tested coatings: a) Cr/CrN, b) Cr/CrN/AlCrTiN, and c) CrN/AlCrN/AlCrTiSiN

Analysis of the results of research of selected mechanical properties showed that, despite the differences in the structural construction of the coatings, they are characterized by a similar hardness and Young's modulus.

The Cr/CrN/AlTiCrN coating is characterized by both the highest hardness ( $H = 30$  GPa) and the highest surface roughness ( $Ra > 0.4$   $\mu\text{m}$ ) of all investigated coatings. Lower roughness values were sequentially obtained by the coatings with the participation of silicon ( $Ra_{\text{AlTiCrSiN}} = 0.15$   $\mu\text{m}$ ) and the chromium nitride coating ( $Ra_{\text{CrN}} = 0.15$   $\mu\text{m}$ ).

## 2.2. Tribological properties

In accordance with the accepted methodology, the wear resistance test of the investigated PVD coatings were performed at  $500^\circ\text{C}$ . For each of the three PVD coatings, the wear index ( $Wx$ ) was calculated. In addition, the friction coefficient and wear diameter of the counter sample were measured. The results of the tribological tests are shown in Table 3 and Figure 2.

Table 3. The results of ball on disc tribological tests

Temperature of the test [°C]	The volume of the wear track [mm <sup>3</sup> ]	Wear index [mm <sup>3</sup> /N * km]	Friction coefficient	The diameter of the ball wear [mm]
Cr/CrN				
500	9.21E-02	4.61E-02	0.11	2.3
Cr/CrN/AlCrTiN				
500	3.67E-01	1.84E-01	0.56	2.2
CrN/AlCrN/AlTiCrSiN				
500	1.02E-01	5.10E-02	0.24	3.1

The analysis of the wear index ( $Wx$ ) for the tested PVD coatings showed that the Cr/CrN and CrN/CrAlN/AlCrTiSiN multilayer coatings are characterised by the best wear resistance at elevated temperatures (500°C) for which the wear indexes were similar and ranged about 4.61E-02 to 5.1E-02 [mm<sup>3</sup>/N \* km]. The Cr/CrN/AlCrTiN coating has significantly worse wear resistance at elevated temperatures. The wear index was  $Wx_{Cr/CrN/AlCrTiN} = 1.84E-01$  [mm<sup>3</sup>/N\*km].

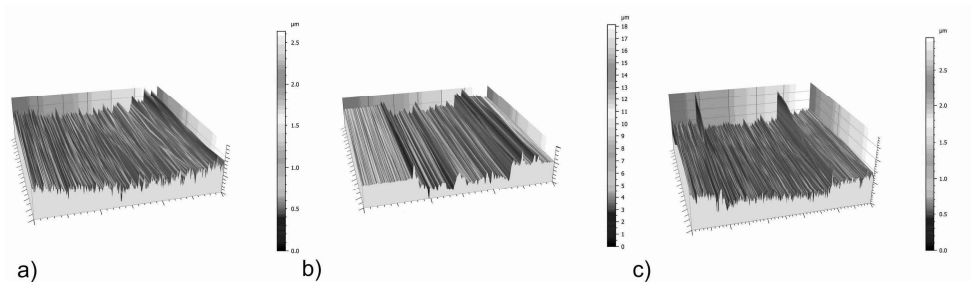


Fig. 2. The results of wear tracks after ball-on-disc tests performer at elevated temperature (500°C): a) Cr/CrN, b) Cr/CrN/AlCrTiN, and c) CrN/AlCrN/AlCrTiSiN

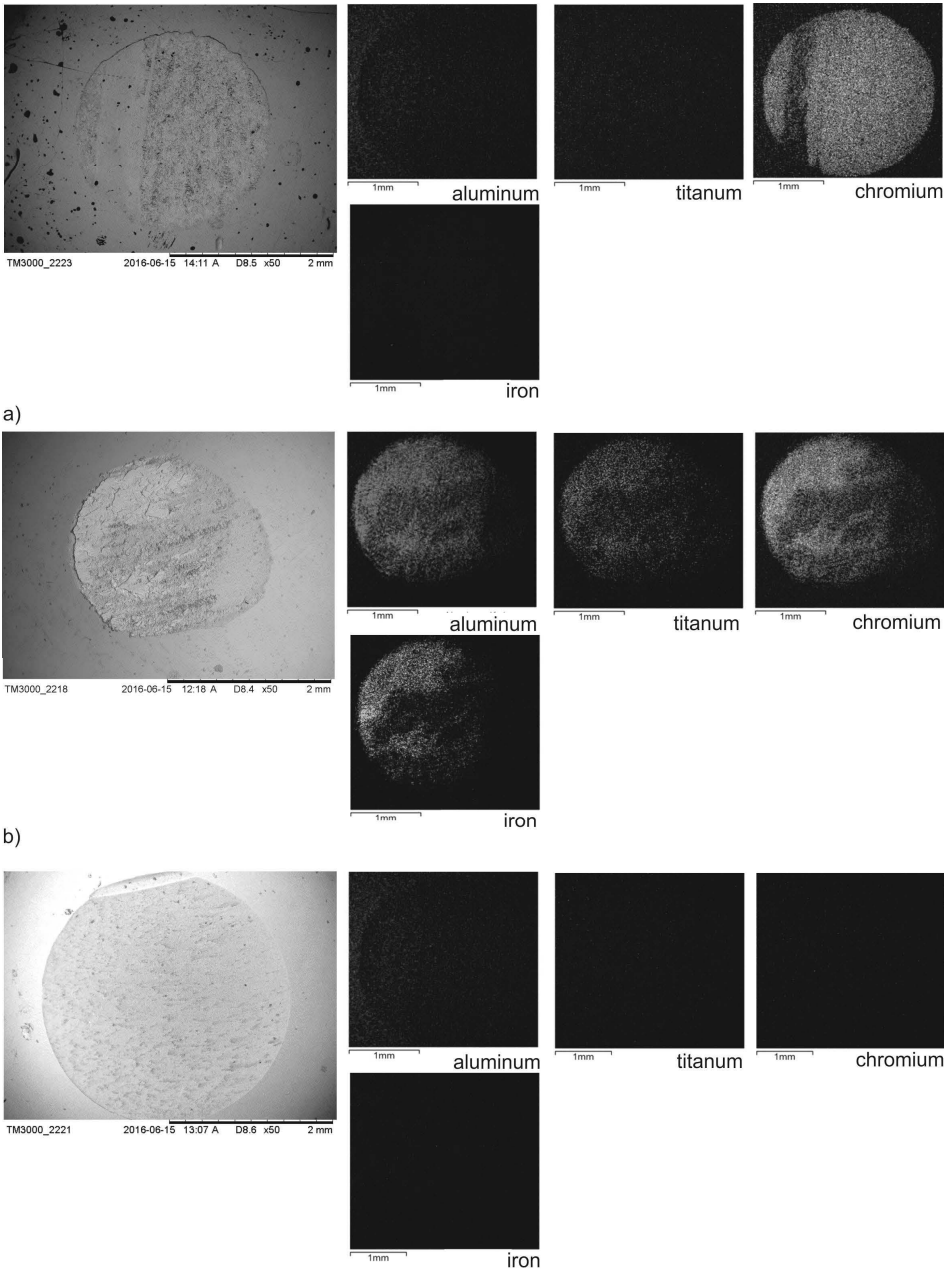


Fig. 3. The results of chemical analyses of the balls after tribological tests were performed on the investigated coatings: a) Cr/CrN , b) Cr/CrN/AlCrTiN, c) CrN/AlCrN/AlCrTiSiN

The results showed a significant effect of surface roughness expressed by the Ra parameter for the wear resistance of investigated coatings. The Cr/CrN/AlCrTiN coatings were characterized by the highest surface roughness and the highest hardness. At the same time, they were characterized by the worst tribological resistance. A significant reduction in the surface roughness of Cr/CrN and CrN/CrAlN/AlCrTiSiN coatings caused the reduction of their friction coefficient and wear intensity. As a result, the connection between the high hardness and roughness of the coating may have a destructive effect on the tribological properties [7].

The analysis of the surface of counter samples indicated that, in all three cases, there was the friction wear of the glass ball. Among the studied of coatings, the largest wear of the glass counter sample was observed in the case of the coating CrN/AlCrN/AlTiSiN (wear crater  $\varnothing 3.1$  mm). In other friction pairs, the glass ball wear is at a comparatively low level and is about  $\varnothing = 2.2$  mm. In the cases of friction pairs, analysis of the chemical compositions formed on glass balls indicated that the coating material was transferred to the counter sample – glass sphere – coating of Cr/CrN (Fig. 3a) and the glass sphere – coating of Cr/CrN/AlCrTiN (Fig. 3b). The presence within the formed wear of the elements constituting the chemical composition of the coating (Ti, Al, Cr), confirms this phenomenon. Additionally, the presence of iron within the wear of the glass ball in the case of Cr/CrN/AlCrTiN coating indicates the partial removal of the coating from the wear area. The presence of the transferred coating material on the surface of the glass ball caused a decrease in the intensity of wear, thus smaller diameters of the formed wear crater. This phenomenon is not preferred in the glass forming process. It will result in the adhesion of the coating material to the moulded glass elements causing their ruin. In the case of the friction pairs, analysis of the chemical composition within the wear ball glass ball indicated the lack of the presence of the elements constituting the chemical composition of coatings, which confirms the lack of the transmission of the coating material on the wear ball – CrN/CrAlN/AlCrTiSiN coating (Fig. 3c).

### 3. Conclusions

The obtained research results enable the formulation of the following final conclusions:

1. The surface roughness has a significant influence on the tribological characteristics of antiwear multilayer coatings. The Ra parameter has the biggest impact on the coefficient of friction value, which was changed in the range of 0.15 to 0.44 microns. Reduction of the coefficient of friction values and wear obtained using the coatings were caused by the reduction of surface roughness.



2. The results concerning the tribological ball-on-disc tests of friction pairs led to the following conclusion: The sodium-calcium glass ball and selected PVD coatings (CrN, AlTiCrN, AlTiCrSiN) at an elevated temperature ( $T = 500^{\circ}\text{C}$ ) showed essential differences among them. Each of the tested friction pairs has wear that caused the loss of weight of the glass ball and PVD coating material (Tab. 3). In two cases, the coating material (CrN and AlTiCrN) was transferred onto the glass ball surface that indicates an adhesive wear. The study of the chemical composition of the friction pair, i.e. glass ball – AlTiCrSiN coating, did not show the transfer of coating material onto the surface of the glass ball. However, the largest wear track of glass balls in this combination (wear track  $\varnothing 3.1$  mm) may suggest the dominant abrasive wear mechanism. The lowest rate of consumption of all the friction pairs were characterized by the following coatings: CrN ( $Wi = 0.0461$  [ $\text{mm}^3/\text{N} \cdot \text{km}$ ]) and AlTiCrN ( $Wi = 0.051$  [ $\text{mm}^3/\text{N} \cdot \text{km}$ ]).

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## **Analiza odporności tribologicznej powłok dedykowanych do poprawy trwałości narzędzi wykorzystywanych w procesie formowania szkła**

### **Słowa kluczowe**

Powłoki wieloskładnikowe, odporność tribologiczna, matryce do formowania szkła.

### **Streszczenie**

W artykule przedstawiono analizę możliwości wykorzystania nowoczesnych rozwiązań inżynierii powierzchni w celu poprawy trwałości eksploatacyjnej narzędzi wykorzystywanych w procesie formowania elementów szklanych, narażonych na jednoczesne działanie kilku czynników niszczących w procesie eksploatacyjnym, tj. cyklicznych szoków cieplnych, korozyjnego działania masy szklanej oraz intensywnego tarcia spowodowanego obecnością osadów z masy szklanej pozostających na narzędziu pomiędzy procesami formowania. W artykule przedstawiono wyniki badań właściwości materiałowych oraz właściwości tribologicznych wybranych powłok wielowarstwowych. Metody badawcze zaprezentowane w artykule koncentrowały się na analizie właściwości mechanicznych, topografii powierzchni, jak również właściwości tribologicznych wybranych warstw hybrydowych. Analiza właściwości mechanicznych obejmowała badania twardości modułu Younga oraz badania adhezji metodą zarysowania.