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TRIBOLOGY OF CrAg7N COATINGS DEPOSITED ON VANADIS 6 LEDEBURITIC TOOL STEEL

TRIBOLOGIJA PREVLEK CrAg7N NA LEDEBURITNEM ORODNEM JEKLU VANADIS 6

Pavel Bílek, Peter Jurči, Mária Hudáková, Ľubomír Čaplovič, Michal Novák

Institute of Materials Science, Faculty of Materials Science and Technology in Trnava, Slovak University of Technology in Bratislava, Paulínská 16, 917 24 Trnava, Slovak Republic
pavel-bilek@email.cz

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Samples made from Vanadis 6 PM ledeburitic tool steel were surface machined, ground and mirror polished. Prior to the deposition, they were heat treated to a hardness of 60 HRC. The CrAg7N coating was deposited with the magnetron-sputtering technique, using pure-Cr and Ag targets in a composite low-pressure nitrogen/argon atmosphere and at a temperature of 500 °C in the Hauzer Flexicoat 850 device. Tribological testing using a pin-on-disc apparatus was carried out at ambient and elevated temperatures: (300, 400 and 500) °C, respectively. Al_2O_3, 100Cr6 and CuZn balls were used as the counterparts. The wear tracks after the pin-on-disc testing were analyzed with scanning electron microscopy and a microanalysis. The experiments have shown a strong dependence of tribological parameters on the temperature. The friction coefficient of CrN-Ag against the 100Cr6 ball at ambient temperature was μ = 0.56. Tribological sliding tests of this coating system against the alumina balls indicate a decrease in the friction coefficient due to the increasing temperature. At ambient temperature μ = 0.68 and its minimum occurred at the temperature of 400 °C, μ = 0.24. This is attributed to the diffusion of Ag particles to the sliding top surface at elevated temperature. The testing against the CuZn brass ball generally gave a lower friction coefficient at ambient temperature, μ = 0.39. In contrast, the friction coefficient slightly increased with the increasing temperature and was practically constant at elevated temperatures, ranging between μ = 0.43–0.48.

Keywords: Cr-V ledeburitic steels Vanadis 6, PVD, chromium nitride with silver, pin-on-disc, friction coefficient

Vzorci, izdelani iz ledeburitnega orodnega jekla Vanadis 6 PM, so bili površinsko obdelani, brušeni in zrcalno polirani. Pred nanosom so bili toplotno obdelani na trdoto 60 HRC. S tehniko magnetronskega napr̆sevanja in z uporabo tarč iz čistega Cr in Ag je bila nanesena CrAg7N-nanoprevleka v sestavljeni nizkotlačni atmosferi iz dušika in argona pri temperaturi 500 °C v napravi Hauzer Flexicoat 850. Tribološki preizkusi s preizkuševalnikom “pin-on-disc” so bili izvršeni pri sobni temperaturi in pri povišanih temperaturah (300, 400 in 500) °C. Krogle Al_2O_3, 100Cr6 in CuZn so bile uporabljene kot par. Sledi obrabe po preizkusu "pin-on-disc" so bile analizirane z vrstično elektronsko mikroskopijo in z mikroanalizo. Eksperimenti so pokazali močno odvisnost triboloških parametrov od temperature. Koeficient trenja CrN-Ag proti krogli 100Cr6 pri sobni temperaturi je bil μ = 0,56. Tribološki drsni preizkusi te prevleke proti kroglam Al_2O_3 kažejo zmanjšanje koeficienta trenja z naraščajočo temperaturo. Pri sobni temperaturi je bil μ = 0,68, minimum pa se je pojavil pri temperaturi 400 °C, μ = 0,24. To se pripisuje difuziji delcev Ag na drsno površino pri povišanih temperaturah. Preizkušanje v paru s CuZn in medeninasto kroglo je dalo na splošno nižje koeficiente trenja pri sobni temperaturi, μ = 0,39. Nasprotno pa je koeficient trenja malo narasel pri povišanju temperature in je bil pri povišanih temperaturah praktično konstanten v območju μ = 0,43–0,48.

Ključne besede: ledeburitno jeklo Cr-V Vanadis 6, PVD, krom nitrid s srebrom, "pin-on-disk", koeficient trenja

1 INTRODUCTION

Chromium nitrides (CrN) have been extensively investigated in the applications of protective coatings due to their high hardness, good wear resistance as well as excellent corrosion and high-temperature-oxidation resistance.[1–5] They gained great scientific interest and industrial popularity due to these properties in copper machining, aluminium die casting and forming, and wood processing.[6] However, in many applications, the requirements on the coated-material surface cannot be met by such a single coating. A further development to adapt some of their properties to the levels required for specific applications leads to the production of composite coatings, combining different material properties so that certain new desired properties can be created.[7–9]

The effect of self-lubrication has gained a great scientific importance in the last few years. The main idea to develop self-lubricating and multi-purpose coatings is based upon the fact that commercially available lubricants (sulfides, oxides, graphite) exhibit considerable shortcomings and cannot be used effectively in tooling applications over a sufficiently wide temperature range.[10–12] Soft noble metals, on the other hand, show a stable chemical behavior and can exhibit self-lubricating properties due to their low shear strength. Noble-metal particles bring several benefits to the layer properties compared to metal oxides or graphite. They are stable up to relatively high temperatures, have a low hardness and do not behave as abrasive particles. A common disadvantage of noble metals is their high cost, but this can be optimized to an acceptable level. The self-lubricating effect is based on an incorporation of a small amount of noble metals, mostly silver, into the basic CrN film. Silver is completely insoluble in CrN and forms nanoparticles in the basic CrN compound. Silver-containing transition-metal-nitride films have been extensively studied in recent years.[13]

The current paper deals with the development of adaptive nanocomposite CrAgN coatings on the Vanadis 6 Cr-V ledeburitic tool steel. It describes and discusses the tribological properties, such as the friction coefficient and wear rate during the pin-on-disk testing, of the coating with the mass fraction w = 7 % content of silver.

2 EXPERIMENTAL WORK

The substrate material was PM ledeburitic steel Vanadis 6 with nominally 2.1 % C, 1.0 % Si, 0.4 % Mn, 6.8 % Cr, 1.5 % Mo, 5.4 % V and Fe as the balance that was soft annealed to a hardness of 21 HRC.[14,15]

The samples used for the investigation were plates with the dimensions of 50 mm × 10 mm × 10 mm, heat treated (austenitized at a temperature of 1050 °C, quenched in a flow of nitrogen gas and double tempered for 2 h at a temperature of 530 °C) to the final hardness of 60 HRC and then finely ground and polished with a diamond suspension up to a mirror finish.

The conditions for depositing CrN/Ag coatings were reported elsewhere.[16] The output power on the Cr cathode was 5.8 kW and on the Ag cathode it was 0.21 kW.

Tribological properties of the coating were measured using a CSM pin-on-disc tribometer at ambient and at elevated temperatures, up to 500 °C. Balls, 6 mm in diameter, made from sintered alumina, 100Cr6 steel and CuZn brass (55 % Cu, 45 % Zn) were used for the tests. The testing against the 100Cr6 counterpart was carried out only at ambient temperature due to a low thermal stability of the 100Cr6 steel. No external lubricant was added during the measurements. The normal loading used for the investigation was 1 N. For each measurement, the total sliding distance was 100 m. The volume loss of the coated samples was calculated from the width of the track using the following formula:[17]

$$s = 2\pi R\,[r^2 \sin^{-1}(d/2r) - (d/4)\mathrm{v}(4r^2 - d^2)]$$

where R is the wear-track radius, d is the wear-track width and r is the radius of the ball.

To relate the volume loss to normal load F and sliding distance l wear rates W[17] were calculated.

After the testing, the wear tracks were examined with a scanning electron microscope (SEM) JEOL JSM-7600F and an energy-dispersive X-ray analysis (EDX).

3 RESULTS AND DISCUSSIONS

Table 1 summarizes the results of the tribological investigations. In the case of the alumina counterpart, the friction coefficient at ambient temperature was μ = 0.68. Therefore, no positive effect of the Ag addition was found at the low temperature, which is in line with the previous investigation.[18]

Table 1: Results of tribological investigation of the CrAg7N coating
Tabela 1: Rezultati triboloških preiskav nanosa CrAg7N

μ	Counterpart		
Temperature	Al_2O_3	CuZn	100Cr6
20 °C	0.68	0.39	0.56
300 °C	0.40	0.43	
400 °C	0.24	0.48	
500 °C	0.29	0.46	

Basnyat et al.[19] and Yao et al.[20] established a beneficial effect of Ag on the friction coefficient at room temperature. However, the loading applied in their investigation was much higher, which makes the results incomparable with our measured data.

At a higher testing temperature, the friction coefficient of the CrN coating with w = 7 % of silver became much lower. The minimum value, μ = 0.24, was found at the temperature of 400 °C. This phenomenon is attributed to various factors. Firstly, there is an increasing mobility of silver at elevated temperature that has been reported.[21] These atoms can diffuse to the surface at elevated temperatures and effectively work as a solid lubricant due to the low shear strength of silver. The second possible contribution of the friction coefficient decreased with the increased testing temperature can be indentified on the basis of the assumption that an increased temperature makes the coating softer.[18]

Figure 1 shows the dependence of the friction coefficient on the sliding distance. At ambient temperature, the friction coefficient increased at the beginning of the test and after that it slightly decreased to a stable state of μ = 0.68, typical for a steady state of sliding. At elevated temperatures, the friction coefficient was first between μ = 0.55–0.65 and then it immediately decreased to the values of the steady state.

Figure 2 demonstrates the wear tracks obtained by testing the CrAg7N coating against alumina at ambient and elevated temperatures. At ambient temperature, the testing gave a smooth surface without any failure of integrity. The testing at elevated temperatures led to a creation of parallel grooves oriented along the sliding direction on the coated material and to a much wider wear track and a high volume loss of the coating and the wear rate, respectively, as documented in **Table 2**. The

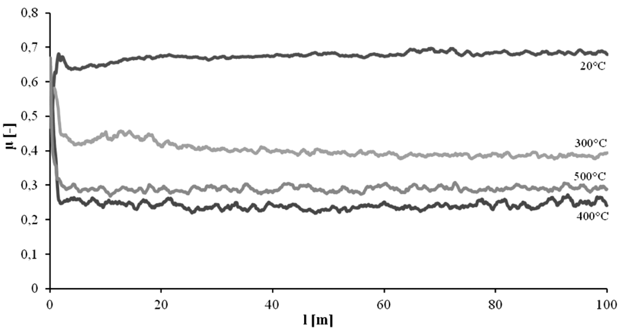

Figure 1: Dependence of friction coefficient on sliding distance for alumina counterpart
Slika 1: Odvisnost koeficienta trenja od poti drsenja za Al_2O_3 v paru

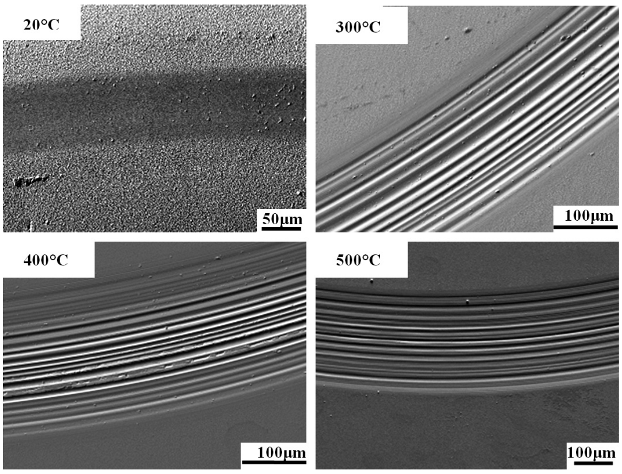

Figure 2: Wear tracks after pin-on-disc testing against alumina at ambient and elevated temperature

Slika 2: Sledi obrabe po preizkusu "pin-on-disc" v paru z Al_2O_3 pri sobni in povišani temperaturi

Table 2: Width of tracks, volume loss and wear rate after pin-on-disc testing against alumina

Tabela 2: Širina sledi, volumenska izguba in obraba pri preizkusu "pin-on-disc" v paru z Al_2O_3

Heat (°C)	d/μm	V/m^3	W/(m^3/(N m))
20	85	$2.72 \cdot 10^{-13}$	$2.72 \cdot 10^{-15}$
300	228	$5.17 \cdot 10^{-12}$	$5.17 \cdot 10^{-14}$
400	250	$6.85 \cdot 10^{-12}$	$6.85 \cdot 10^{-14}$
500	276	$9.24 \cdot 10^{-12}$	$9.24 \cdot 10^{-14}$

wider tracks can be attributed to the softening of the coating at elevated temperatures.

At the temperature of 400 °C a partial failure of the coating was observed. The EDX analysis showed the presence of iron (the base element of steel) on the surface of the track in some sites, while the contents of chromium and silver were found in the other sites (**Figure 3**). This result confirms a partial removal of the coating from the substrate.

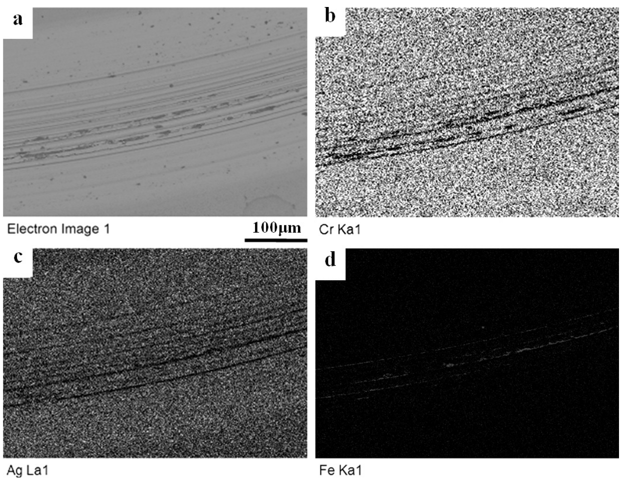

Figure 3: Partial failure of coating CrAg7N after pin-on-disc testing against alumina at the temperature of 400 °C: a) overview, b) EDX of chromium, c) EDX of silver, d) EDX of iron

Slika 3: Parcialne poškodbe nanosa CrAg7N po preizkusu "pin-on-disc" v paru z Al_2O_3 pri temperaturi 400 °C: a) videz, b) EDX kroma, c) EDX srebra, d) EDX železa

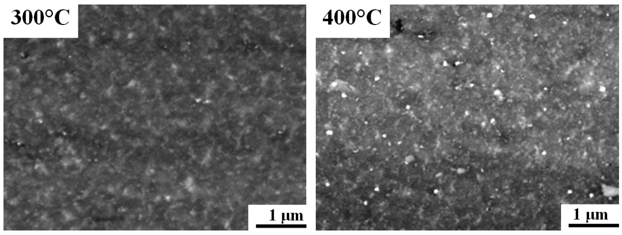

Figure 4: Nanoparticles of silver on the surface of track after pin-on-disc testing

Slika 4: Nanodelci srebra na površini sledi po preizkusu "pin-on-disc"

Our previous investigation of the CrN coating with w = 3 % of silver gave similar results.[22] The lowest value of the friction coefficient was also found at the temperature of 400 °C. However, the CrAg3N coating showed a much higher failure, and even at the temperature of 500 °C it was completely removed from the surface of the substrate after the test.

Figure 4 shows the surface of a track after the pin-on-disc testing at the temperatures of 300 °C and 400 °C. In both cases, Ag particles are well visible and these particles are responsible for a better friction and could act as a solid lubricant. In the wear track formed during the testing at 500 °C Ag particles were also identified, but their population density was reduced in comparison with the samples tested at lower temperatures.

Figure 5 shows the wear tracks formed by the sliding of the coating against CuZn brass. Compared to the tracks caused by the sliding of sintered alumina these tracks are wider, but no damage of the coating is observed.

On the other hand, a considerable amount of the material transferred from the counterpart to the surface of the coating was detected **(Figure 6**).

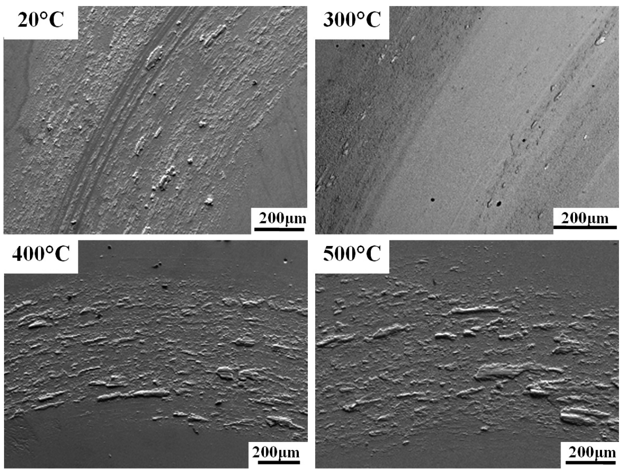

Figure 5: Wear tracks after pin-on-disc testing against CuZn at ambient and elevated temperature

Slika 5: Sledi obrabe po preizkusu "pin-on-disc" v paru s CuZn pri sobni in povišani temperaturi

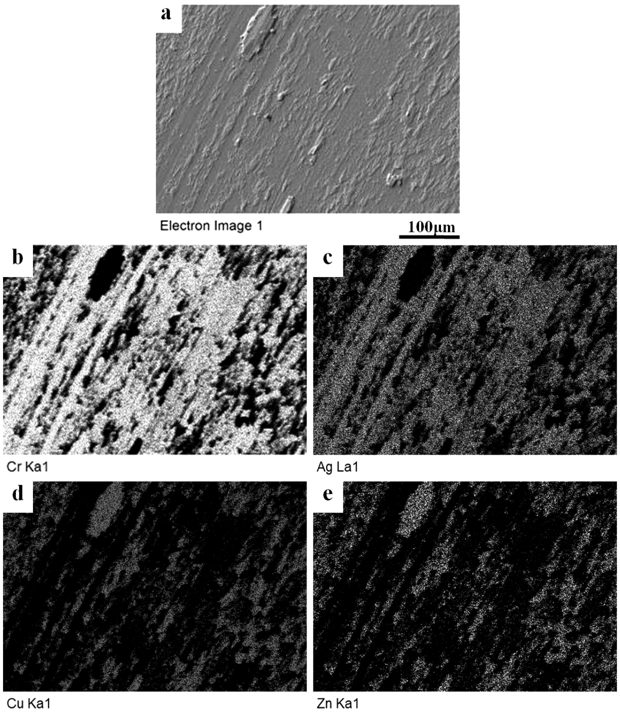

Figure 6: Material transferred from counterpart CuZn to the surface of coating CrAg7N after pin-on-disc, at the temperature of 20 °C: a) overview, b) EDX of chromium, c) EDX of silver, d) EDX of copper, e) EDX of zinc

Slika 6: Prenesen material s para CuZn na površino nanosa CrAg7N po preizkusu "pin-on-disc" pri temperaturi 20 °C; a) videz, b) EDX kroma, c) EDX srebra, d) EDX bakra, e) EDX cinka

This is due to the very low shear strength of CuZn, especially at a higher temperature. It should be noted that such a material transfer was detected irrespective of the testing temperature. These results are in good agreement with the previous work.[22]

The measurement results for the friction coefficient of the CuZn counterpart are shown in **Table 1**. The minimum friction coefficient of $\mu = 0.39$ was found at ambient temperature. With the increasing temperature, the friction coefficient becomes higher, ranging between $\mu = 0.43–0.48$. No effect of silver on the tribological performance was found here – the material transfer can be considered as a plausible explanation (**Figure 6**).

The friction coefficient against the 100Cr6 counterpart was found to be $\mu = 0.56$ (**Table 1**). The material of steel transferred to the surface of the coating is documented in **Figure 7**. The surface of the coating stayed smooth, without any damage.

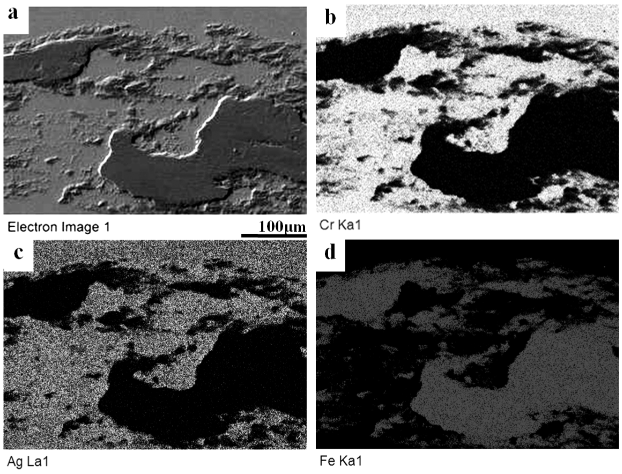

Figure 7: Material transferred from counterpart 100Cr6 to the surface of coating CrAg7N after pin-on-disc, at the temperature 20 °C: a) overview, b) EDX of chromium, c) EDX of silver, d) EDX of iron

Slika 7: Prenesen material iz para 100Cr6 na površino nanosa CrAg7N po preizkusu "pin-on-disc" pri temperaturi 20 °C: a) videz, b) EDX kroma, c) EDX srebra, d) EDX železa

4 CONCLUSIONS

The friction and wear characteristics of the CrAg7N coatings prepared with the magnetron-sputter-deposition method were examined at different temperatures and with different counterpart materials. The results can be summarized as follows:

- The friction coefficient rapidly decreased with the increasing testing temperature when an alumina ball was used as the counterpart, with its minimum at the temperature of 400 °C. The coating showed partial damage at elevated temperatures, but it was not removed from the substrate.
- The friction coefficient was not positively influenced when tested against the CuZn counterpart.
- No removal or other coating damage was established after the sliding against the CuZn ball and 100Cr6 ball, but a considerable material transfer from the ball to the sample was detected.
- The presence of silver in the CrN coating results in improved tribological properties at moderate temperatures. Under these conditions, the CrN coating with $w = 7$ % of Ag could find its application in industry.

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