TRIBOLOGICAL PROPERTIES OF VACUUM ARC Cr-O-N COATINGS IN MACRO- AND MICROSCALE

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In this paper the tribological properties of Cr-O-N coatings deposited using vacuum arc plasma flux in macro- (sphere-on-disc test) and microscale (AFM – atomic force microscopy) are investigated. It was found that the specific wear rate determined in AFM measurements (micro scale) is approximately 2 orders higher than the macroscale. This is probably due to much higher Hertzian contact stress.

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INTRODUCTION

The wear of the tool, particularly premature failure is a serious technical and economical problem. The solution to this would be to modify the tool surface by deposition of a thin hard coating. Such treatment greatly improves the mechanical, and especially tribological properties of the tool.

Due to the good tribological and mechanical properties: high hardness and adhesion to the steel substrate, low coefficient of friction and excellent wear and corrosion resistance transition metal nitrides deposited by PVD methods are used in industry as protective coatings [1, 2].

Users requirements often exceed the possibilities of their use caused by the properties of a two-component coatings. Their modification by adding further elements can significantly increase the properties of the coatings, for example thermal stability, resistance to wear and corrosion. Cr-O-N system was also investigated, but the main topic of interest was the structure and morphology of coatings and their mechanical and tribological properties was not a major subject of investigations [3-5].

Cr-O-N coatings are interesting as protective coatings because of their resistance to oxidation and wear, but also due to photo-thermal conversion of solar energy as a solar selective coating of the absorber [6], or as a decorative coatings because of their different colors [7].

Friction and wear are the main characteristics of coatings resistant to abrasion. The investigations of the properties of thin coatings with a thickness of 1…3 μm require correction to the relatively soft steel substrate. Here may be helpful the atomic force microscopy (AFM) method [8], who meets high requirements for testing the vacuum deposited thin films on polished steel substrates.

The goal of the work is to investigate Cr-O-N coatings deposited using vacuum arc plasma flux with respect to their tribological properties as friction and wear both in macro- and microscale using AFM.

1. EXPERIMENTAL

The Cr-O-N deposition process was performed by unfiltered vacuum arc plasma flux method using Bulat system. The chemical composition of the HS6-5-2 (DIN standard) steel substrates is as follows (wt.%): C (0.87), W (6.4), Mo (5.0), V (1,9), Cr (4.2), Mn (0.3), Si (0.4) and Fe balanced. Before deposition, the substrates were quenched and tempered to the hardness of 63 HRC, ground and polished to a roughness Ra <0.02 μm, and then ultrasonically cleaned in an alkaline bath. Next, they were placed on the planetary rotating holder within the distance of 300 mm from the cathode in vacuum chamber. Rotation speed was about 30 rpm.

The chamber was evacuated to a pressure of 2×10^{-3}Pa. Prior to the deposition, the substrates were sputter cleaned at pressure of 0.005 Pa using chromium (Cr^+) ions under 1300 V negative substrate bias voltage for 3 min. The arc current was 90 A. The thin (~ 0.1 μm) chromium layer was also deposited onto the substrate to improve the adhesion. A deposition process was performed at parameters: substrate bias voltage of -150 V, arc current of 90 A, nitrogen pressure of 1.8 Pa. The substrate temperature was maintained at about 400 ^{0}C. In case of Cr-O-N coatings gas mixture (N_2+O_2) with different relative oxygen concentrations $O_{2(x)}=O_2/(N_2+O_2)$ where x equals 0, 5, 20 and 50 % were used. The deposition time was kept at 45 min in all cases to obtain about 7…8 μm thick coatings.

Taking into account the relative concentration of oxygen during deposition of the coatings the samples will be labeled as follows Cr-O(x)-N. This means that for example of Cr-O(20)-N coating was obtained at relative concentration of oxygen of 20 % in gas mixture.

Hardness and adhesion of the coatings were determined methods and devices described in [9]. The tribological properties of the coatings in macroscale: coefficient of friction and specific wear rate were computed based on studies conducted in the sphere-on-disc geometry. The parameters of the test were: load of 20 N, sliding speed of about 0.2 m/s, dry friction conditions, sliding distance of 1000 m, ambient temperature, air atmosphere, a humidity of about 50 %. The radius in wear tests was 12 mm. The alumina ball with a diameter of 10 mm and Ra<0.03 μm was used as a counterpart. Wear track profiles were determined using the tactile profilograph Hommel Werke T8000 with skid roughness pick-up and an optical surface profiler MicroXAM-800 (KLA-Tencor). The wear rate was calculated as a wear volume divided by sliding distance and normal load [10].

The morphologies, roughness, friction and wear of Cr-O-N coatings in microscale were characterized by atomic force microscope (NT-206, produced by MTM Belarus). The radius of the diamond tip about 200 nm was used.

2. RESULTS AND DISCUSSION

Roughness of the Cr-O-N coatings investigated using tactile Hommel tester and AFM are gathered in Table 1. The coatings deposited at relative oxygen concentration equal 0, 5 and 20 % shows similar roughness tested in both methods. It is obvious that roughness investigated using AFM is lower due to small scanned area 20×20 μm.

Table 1

Roughness of Cr-O-N coatings using tactile Hommel tester T8000 and AFM

Coating	Roughness [nm]	
	Hommel Werke T8000	AFM
Cr-O(0)-N	50±5	14.6
Cr-O(5)-N	46±4	9.5
Cr-O(20)-N	50±2	11.4
Cr-O(50)-N	64±2	21.5

Hardness of the coatings generally increase with rise of relative oxygen concentration during deposition, Fig. 1. The critical load Lc_2 when delamination of the coating occurs systematically decreases with $O_{2(x)}$ increase, starting from about 100 N and ending with 58 N. High hardness, about 30 GPa, for coatings deposited at $O_{2(x)}$ equal 20 and 50 % is probably the cause of the brittleness of these coatings and related to it low critical load Lc_2.

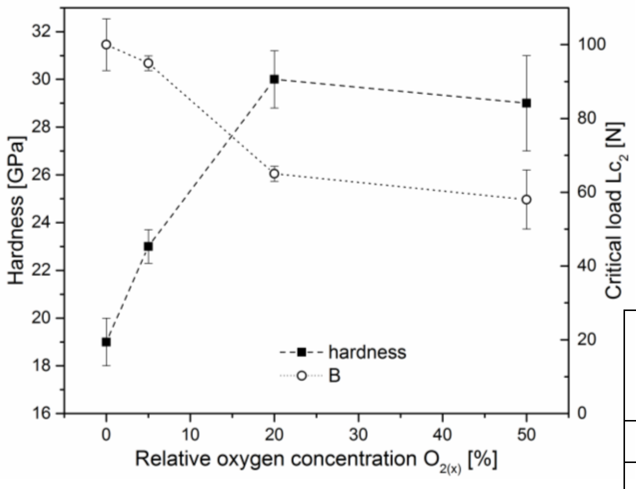
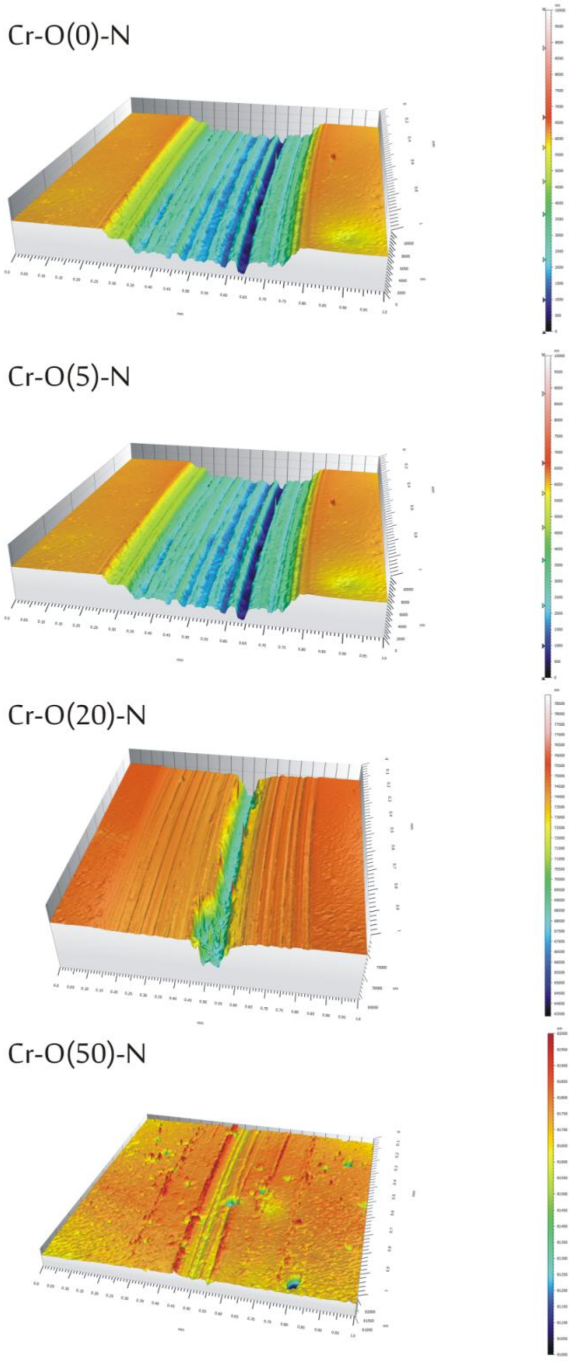


Fig. 1. Hardness and critical load Lc_2 of Cr-O-N coatings deposited at different relative oxygen concentration $O_{2(x)}$

The wear in macroscale was investigated in sphere-on-disc geometry. The wear tracks presented in Fig. 2 exhibit decrease in wear depth with relative oxygen concentration increase.


Cr-O(0)-N

Cr-O(5)-N

Cr-O(20)-N

Cr-O(50)-N

Fig. 2. Micrographs of wear tracks of the Cr-O-N coatings investigated

Table 2

Tribological characteristics of the coatings investigated by sphere-on-disc test

Coating	Average coefficient of friction	Specific wear rate [m^3/Nm]	Wear depth [μm]
Cr-O(0)-N	0.65±0.03	$(4.4\pm0.1)\times10^{-15}$	6.3±0.6
Cr-O(5)-N	0.60±0.05	$(3.2\pm0.3)\times10^{-15}$	5.1±0.1
Cr-O(20)-N	0.48±0.01	$(9.7\pm5.2)\times10^{-16}$	2.5±1.3
Cr-O(50)-N	0.50±0.01	$(6.7\pm1.6)\times10^{-17}$	0.8±0.1

All computed data. i.e.: average coefficient of friction, specific wear rate and wear depth are collected in Table 2. It is interesting that all above data decrease with increasing the relative oxygen concentration. The main type of wear is abrasion (mechanisms: plowing, cutting, fragmentation), although adhesive wear or surface fatigue can't be excluded.

To form visible in optical microscopy track sufficient load should be applied. For this load a deflection of the substrate and destruction of the coating not due to wear, but because of the brittle fracture of the coating occur. From the other hand lower load does not damage the coating, but the wear can be so small that the use of profilometry would be impossible. The depth of the track of tens of nanometers compared to the height of droplets on the surface, which can have a height of hundreds of nanometers is not recognized and is lost in the "noise" deviation. It is helpful in this case the use of atomic force microscopy [11]. This allows the study with resolution of nanometers, the surface topography of the coating, and for detecting the non-uniformity of the surface properties, which is associated with the existence of different phases in the coating.

The test on wear resistance using AFM method was generally performed on the scan area 5×5 µm in two regimes: "fast" with load about 1.6 mN, 3 scans at the same place, 128 lines in scan with velocity 6.0×10^{-6} m/s (Fig. 3) and "slow" with load about 1.2 mN, 4 scans at the same surface and 256 lines in scan with velocity 3.5×10^{-6} m/s. Cr-O(20)-N the most wear resistant sample of was worn by 13 scans, Fig. 4. The radius of the diamond tip about 200 nm and load 1…2 m·N allows to create maximal Hertzian pressure in contact about 69…77 GPa for "fast" wear and about 61…69 GPa for "slow" wear. The maximal shear stresses on the surface of coatings were 21…24 GPa for "fast" wear and 19…22 GPa for "slow" wear.

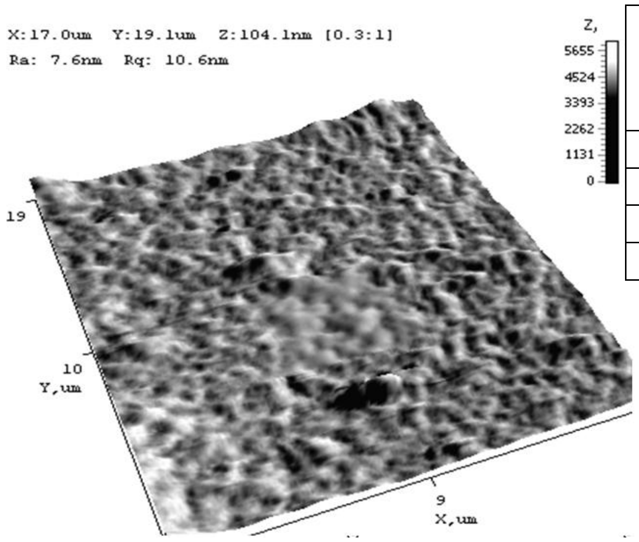


Fig. 3. Result of the "fast wear" 3 scans of Cr-O(20)-N coating

The image of the wear track (see Figs. 3 and 4) is visualized by the same diamond probe with load about 300 µN. The depth of wear was measured by cross-section profile of track. In case of "fast" and "slow" regimes the wear depth was defined as about 3 nm for 3 scans and 40 nm for 13 scans respectively.

Contrary to the sphere-on-disc method, in the AFM the friction and wear test are conducted at different loadings. Generally they decrease with relative oxygen concentration during deposition increases.

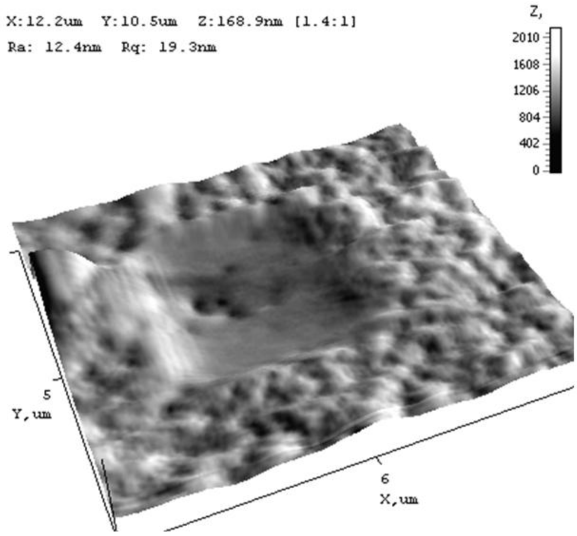


Fig. 4. Result of the «slow wear»13 scans of Cr-O(20)-N coating

To obtain comparable results in wear test after preliminary attempts to determine the conditions (see above) the following were chosen: scan area 5×5 µm, 4 scans, 256 lines/scan, load 1.2 mN, scan velocity 3.5×10^{-6} m/s. The specific wear rate was calculated according to Archard's formula. The results are gathered in Table. 3.

Table 3

Results of the wear, friction and roughness of CrON coatings obtained by AFM with the diamond tip

Coating	Coefficient of friction	Specific wear rate [m^3/N·m]	Wear depth [nm]
Cr-(0)-N	0.29±0.01	$1.6\cdot10^{-13}$	80
Cr-(5)-N	0.32±0.03	$6.1\cdot10^{-14}$	30
Cr-(20)-N	0.24±0.01	$2.5\cdot10^{-14}$	12
Cr-(50)-N	0.133±0.001	$2.0\cdot10^{-13}$	100

Both specific wear rate and wear depth decreases with relative oxygen concentration increase to about $O_{2(x)}$ = 20 %, and next radically increase for $O_{2(x)}$ = 50 %. It is interesting that specific wear rate is about 2 orders higher than in macroscale (calculated based on results from sphere-on-disc geometry). It can be connected with large difference in Hertzian contact stress, about 1.5 GPa in macroscale and about 60…70 GPa in microscale.

CONCLUSIONS

Vacuum arc plasma flux deposition method at constant pressure and with different relative oxygen concentration in chamber nitrogen atmosphere during the process allowed to obtain the set of the coatings characterized by different chemical composition. As a result of mechanical and tribological tests was found that:

1. Hardness of Cr-O-N coatings increases and critical load Lc_2 decreases with relative oxygen concentration increase.
2. The roughness of Cr-O-N coatings tested both by tactile Hommel tester and using AFN is approximately similar to $O_{2(x)}$ = 20 % and then increase.
3. The wear depth defined in sphere-on-disc test (macroscale) decreases monotonously with increase of $O_{2(x)}$. In case of measurements made by AFM (microscale) the wear depth decreases with increase of $O_{2(x)}$ to 20 % and for $O_{2(x)}$ = 50 % significantly increases.
4. The specific wear rate calculated in the basis of sphere-on-disc results (macroscale) monotonically decreases with increase on relative oxygen concentration to the lowest value $(6.7\pm1.6)\times10^{-17}$ m^3/Nm. In case of results from AFM the specific wear rate decreases to the lowest value $2.5\cdot10^{-14}$ m^3/Nm for $O_{2(20)}$ and then increases to value $2.0\cdot10^{-13}$ m^3/Nm for $O_{2(50)}$. The result of wear rate in microscale is about 2 to 4 orders higher than in macroscale. It is probably connected with significantly higher Hertzian contact stress.

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ТРИБОЛОГИЧЕСКИЕ СВОЙСТВА ВАКУУМНО-ДУГОВЫХ ПОКРЫТИЙ Cr-O-N НА МАКРО- И МИКРОУРОВНЯХ

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Исследовались трибологические свойства Cr-O-N-покрытий, осаждённых из потока вакуумно-дуговой плазмы на макро- (тест сфера-на-диске) и микроуровне (АСМ – атомно-силовая микроскопия). Было установлено, что удельная скорость износа в измерениях АСМ (микроуровень) примерно на 2 порядка выше, чем в макромасштабе. Это, вероятно, связано с гораздо более высокими контактными напряжениями.

ТРИБОЛОГІЧНІ ВЛАСТИВОСТІ ВАКУУМНО-ДУГОВИХ ПОКРИТТІВ Cr-O-N НА МАКРО- І МІКРОРІВНЯХ

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Досліджувалися трибологічні властивості Cr-O-N-покриттів, осаджених з потоку вакуумно-дугової плазми на макро- (тест сфера-на-диску) і мікрорівні (АСМ – атомно-силова мікроскопія). Було встановлено, що питома швидкість зносу у вимірюваннях АСМ (мікрорівень) приблизно на 2 порядки вище, ніж в макромасштабі. Це, ймовірно, пов'язано з набагато більш високими контактними напруженнями.