COMPLEX EVALUATION OF STRUCTURAL STATE DEGREE OF STRENGTHENING NANOCOATINGS

T.S. Skoblo[1], S.P. Romaniuk[1], A.I. Sidashenko[1], V.S. Taran[2], A.V. Taran[2], I.I. Dorozhko[3] N.N. Pilgui[4]

[1]Kharkov National Technical University of Agriculture, Kharkiv, Ukraine;
[2]National Science Center "Kharkov Institute of Physics and Technology", Institute of Plasma Physics, Kharkiv, Ukraine;
[3]H.S. Skovoroda Kharkiv National Pedagogical University, Kharkiv, Ukraine;
[4]National Technical University "Kharkov Polytechnic Institute", Kharkiv, Ukraine

E-mail: tservis@ticom.kharkov.ua

The degree of structural heterogeneity of TiN coatings on the surface of cold-rolled 65 G thin sheet steel obtained in standard PVD and RF modes was investigated using the optical-mathematical method. The factors such as the diffusion of chemical components, the density of the structure and the intensity of the resulting deformations that affect the stability of the tool during operation are estimated.

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INTRODUCTION

Operational stability of thin-walled cutting tool made of cold-rolled 65 G thin sheet steel (0.64 mm thick with a 0.1 mm cutting edge) used in confectory is insufficient. It processed of up to 1.8 tons of raw materials only. Various types of PVD coatings are applied to increase the tool service life. Characteristics and properties of the strengthened layer depend on the base material and application method [1, 2]. The conducted industrial tests showed that the tool service life reinforced by various methods differs [3]. Its durability is directly affected by the structure of the base metal, phase composition and mechanical properties of the coating. In addition, one of the reasons for the different operational resistance is structural heterogeneity and non-equilibrium phase formation.

There are GOSTs and computer programs, for example Thixomet Pro allowing determining the grain size, their number in the metal structure when analyzing the image of the surface being examined.

The aim of the research was to use a new optical-mathematical approach to determine the degree of heterogeneity of TiN coatings on thin-walled cutting tool.

METHOD AND CHARACTERISATION

Application of the TiN coating was carried out using the Bulat-6 type device. Two regimes of coatings deposition were comparatively studied [3]. First, TiN coating was applied by vacuum-arc method in standard PVD mode. To obtain the TiN coating, the vacuum chamber was filled with nitrogen at 99.99% purity to the pressure $P = 4 \cdot 10^{-1}$ Pa. The negative shift on the substrate was U_{shift}= -200 V, I_d = 100 A. The deposition time of the TiN coating was 24 min. The thickness of obtained TiN coating reached 4 μm.

TiN coating was also applied using RF discharge mode. The tool was cleaned with RF discharge for 10 min (U^{RF}_{shift} = -500 V). A sublayer of pure Ti was applied for 3 min at a pressure of $P = 2 \cdot 10^{-1}$Pa, I_d = 110 A, U^{RF}_{shift}= -100 V. To obtain TiN coating, the vacuum chamber was filled with nitrogen 99.99 % purity at a pressure of $P = 1 \cdot 10^{-1}$ Pa. The negative shift on the substrate was U_{shift} = -100 V. The total coating time was 15 min with a cyclic deposition regime (5 min deposition and 3 min pauses). The thickness of the applied TiN coating was 3.3 μm.

The surface topography was studied using JEOL JSM-6390LV scanning electron microscope (SEM) with an accelerating voltage of 20 kV, chemical composition was examined using EDX analysis.

Evaluation of the structural heterogeneity of TiN coatings was carried out using SEM images with the help of optical-mathematical method.

RESULTS AND DISCUSSION

When processing the SEM image, we considered a uniform rectangular grid of points (pixels) with 3 × 3, 4 × 4 and 5 × 5 cell sizes on which function values were set. In this case, these are the colors or shades of these points. An example of the arrangement of points around the mean c_1 is shown in Fig. 1. Heterogeneity was calculated as the ratio of this indicator to 9, 16 or 25 points, respectively.

$$\begin{matrix} c_3 . & c_4 . & c_5 . \\ c_2 . & c_1 . & c_6 . \\ c_9 . & c_8 . & c_7 . \end{matrix}$$

Fig. 1. The numbering scheme of the points around in the average cell 3 × 3 pixels

The histograms of the color distribution obtained with a specially developed program that includes 256 colors (shades from black to white) from 0 to 255 are calculated. They are divided into 16 intervals, describing 3 groups of phases: 1 – pure component of the Ti coating, 2–10 non-stable compounds of Ti, N with Fe and C (corresponds to the diffusion of the components from the knife base), 11-16 TiN nitrides and compounds of various non-stoichiometric composition.

With the help of EDX the distribution of chemical components in coatings for various deposition modes is carried out. It is established that the average quantitative ratio of distributed components on the entire surface of a thin-walled knife with TiN (PVD) was: 71.21 % Ti, 25.59 % N, 1.88 % C, and 1.32 % O [3]. In RF discharge mode, the EDX data revealed 63.43 %Ti, 31.76 %N, 2.25 % C, 1.84 % O, 0.03 Si, and 0.69 % Fe.

The formation of the TiN coating is accompanied by the formation Me_xN_y type phases, the Fe_xN_y, Fe_xTi_y, Ti_xN_y phases, as well as constituents including carbon, oxygen and other microadditives, which correspond to histograms numbered from 2 to 10.

It has been performed a comparative analysis of SEM images of TiN coatings using optical – mathematical processing. The number of phases was estimated on 14 SEM images (7 – applied by PVD and 7 – using RF discharge).

The obtained data showed that in different zones of the surface the phase concentration differs significantly. The share of the total nitride component (color numbers 11-16) varies from 1.8 to 11.9 %. In addition, a large number of various unstable compounds, including the components of the base (histogram 2–10), have been identified. Analysis of the coating applied by PVD showed that a large number of such phases are formed on its surface (their fraction in local zones varies from 0 to 44.2 %). The proportion of pure Ti (color 1) on the surface of the coating does not exceed 0.4…0.5 % and was detected only in two cases of the seven zones of the coating in the drop phase. The complete absence of a pure component is characteristic of the coating applied using RF discharge.

The comparative analysis of the obtained results showed that in RF discharge mode, there are significantly fewer non-stoichiometric compounds (no phases corresponding to the color numbers 2, 3, 4, and practically 12, 13, 14, 15). The concentration of the nitride component varies from 0.9 to 18.5 % (colors 11–16).

The largest proportion of phases in the test surface of the sample belongs to the Ti, N, Fe, C (colors 5–10) compounds, which correspond to the maximum concentration of base components (Fe, C), due to the presence of a large number of pores in base metal. Such defects are retained and they reflect concentration of the components of the parent metal with partial interaction with the coating components.

The degree of heterogeneity of the structural components, the hardened cutting tool by the optical-mathematical method was also analyzed for different ways of depositing the TiN coating. The quantitative value of the analyzed indicator for PVD is shown in Table 1 and Fig. 2.

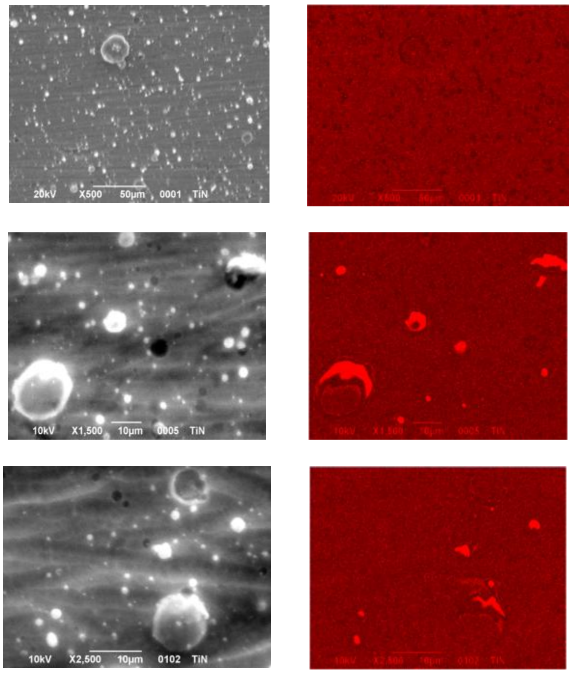
The degree of heterogeneity in electron images of a surface hardened by a TiN coating on the given fragments (3 × 3, 4 × 4, 5 × 5 and throughout the photo) was comparatively analyzed. The closer this index is to unity, the less heterogeneous structure is formed. The data obtained from Table 1 presents that analysis of the minimal regions allows locally detecting of homogeneous zones and structural defects. The larger the area of the image area being analyzed, the less the structural heterogeneity becomes. For a tool strengthened by standard PVD, a lower uniformity index of the coating composition is revealed, which does not exceed 0.309. Since the structure of coatings is finely dispersed, the analysis is recommended to be carried out according to the scheme of 3 × 3 pixels. With this evaluation of the structure, zone 7 is characterized by a lesser degree of heterogeneity (see Table 1) and it is 0.718. The value obtained is 1.7 times higher than the data for the unstressed tool. Such indications contribute to a 6-fold increase in the durability of knives coated with TiN coated by PVD in comparison with the original tool. The scatter of indications for the degree of heterogeneity for a reinforced thin-walled cutting tool in a 3 × 3 pixels scheme was from 5.9 to 26.3 %. With the increase in the analyzed area, the scatter in the results is also increased. In cells of 5 × 5 pixels, the detected deviations reach 4.8…30.8 %. For the whole image, this indicator was from 8.2 to 44.3 %.

Heterogeneity is represented by a continuous color from black to red (see Fig. 2). Red is the maximum degree of heterogeneity, black is the minimum. When analyzing images of a structure, it is necessary to comprehensively analyze and take into account all the obtained data, both digital indicators and structural images. A homogeneous structure is observed only in the large drop phase. It has been found that the smallest value of the analyzed index of the degree of heterogeneity was in small drops and along the edge of large ones.

Table 1

Calculation of histograms of heterogeneity of nitride phase distribution

Average heterogeneity in pixel cells								picture #
3×3 pixels		4×4 pixels		5×5 pixels		around the photo		
PVD	RF	PVD	RF	PVD	RF	PVD	RF	
0.495	0.92	0.453	0.903	0.428	0.888	0.292	0.545	1
0.525	0.892	0.489	0.869	0.467	0.848	0.324	0.435	2
0.535	0.911	0.504	0.895	0.488	0.879	0.309	0.536	3
0.51	0.87	0.476	0.837	0.458	0.809	0.142	0.534	4
0.518	0.809	0.485	0.764	0.467	0.729	0.14	0.479	5
0.68	0.608	0.639	0.542	0.611	0.498	0.272	0.285	6
0.718	0.799	0.69	0.752	0.671	0.711	0.281	0.297	7
0.569	**0.836**	**0.534**	**0.801**	**0.513**	**0.773**	**0.251**	**0.457**	Mean value



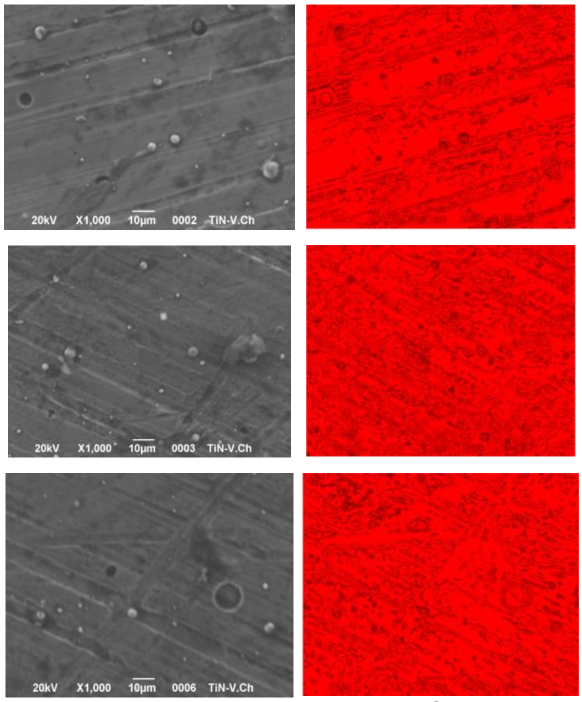
a b

Fig. 2. SEM images of TiN coating in standard PVD mode (a) and corresponding images of variation in the degree of heterogeneity (b)

Also, comparative studies of images of the structure of a TiN-hardened tool using RF discharge were carried out. Table 1 presents the obtained inhomogeneity data.

The scatter of the indications for the degree of heterogeneity for the TiN hardened thin-walled cutting tool using RF discharge according to a 3 × 3 pixels cell layout was 1.3 to 27.3 %. There is a significant increase in the scatter of the results obtained and according to the scheme 5 × 5 pixels. The observed deviations reach 1.8…35.6 %. For the whole image, this indicator was from 2.4 to 37.6 %.

Fig. 3 shows the morphology of the TiN-hardened surface of the tool (using RF discharge) and the corresponding images of variation in the degree of inhomogeneity, according to Table 1. As can be seen from Fig. 3, the image of the change in the degree of inhomogeneity is much lighter than in the photo of the hardened surface by PVD (see Fig. 2). The revealed minimal heterogeneity is associated with pores in the main metal of the tool, which lead to local deformation and the development of damageability during operation. From the obtained results it follows that it is necessary to pay special attention to the choice of a quality material for the manufacture of a thin-walled tool and its further operation.



a b

Fig. 3. The morphology of the TiN coating obtained using RF discharge (a) and corresponding images of variation in the degree of heterogeneity (b)

As a result of the statistical analysis of the obtained data (see Table 1) it was established that the application of TiN coatings with the use of RF discharge reduces the structural heterogeneity of the coating by 76.4 %. When analyzing a 3 × 3 pixels cell, this indicator tends to one and reaches 0.92.

In the technique of mathematical image processing, such concepts are as follows:

– the absolute value of divergence, which describes the density of the image fragment. The greater the divergence value, the more structural changes are noted in the coating;

– the absolute value of the Laplacian, which describes the diffusion of chemical components;

– the generalized gradient, which is the average effective rate of color change; it corresponds to the intensity of the arising deformations. The obtained values of the functions are estimated in and presented in Table 2.

Table 2

Mean values of the functions studied

Color		Standard color deviation		Generalized gradient		Laplacian		3rd Laplacian		4th Laplacian		Divergence		# picture
PVD	RF	PVD	RF	PVD	RF	PVD	RF	PVD	RF	PVD	HF	PVD	HF	
118.3	102.1	17.5	8.3	16.6	6.3	52.9	23.4	91.6	42.7	175.6	83.0	29.3	12.4	1
118.3	94.6	16.3	9.8	15.2	7.5	50.2	28.2	87.9	49.4	169.6	96.3	27.4	14.8	2
120.7	90.2	16.3	8.3	15.3	6.4	50.4	24.2	89.5	43.3	173.0	84.5	27.4	12.6	3
114.7	91.3	31.9	9.0	27.7	7.0	87.2	26.4	156.0	47.4	300.3	92.7	49.3	13.7	4
105.2	93.1	31.2	9.4	28.0	7.4	90.7	28.0	168.2	48.7	326.0	95.3	49.7	14.5	5
66.2	151.4	19.0	15.1	15.9	13.0	52.3	42.4	93.2	74.4	178.8	141.5	29.2	23.7	6
50.1	134.7	18.9	13.8	15.7	11.2	53.3	38.8	96.5	70.0	186.8	134.3	29.2	21.3	7

CONCLUSIONS

The comparative analysis of the degree of heterogeneity of the hardened tool surface using SEM images is carried out using optical-mathematical method. This method made it possible to reveal significant heterogeneity in the structure of coatings deposited by the vacuum-arc method with bombardment of titanium ions.

It is established that coating a tool surface with RF discharge prevents diffusion of components from the base metal, 5 times reduces structural changes compared to the initial state. In addition, it prevents degradation of the working layer, ensuring its stability during operation, and 47 time increases durability. It is shown that further durability of the strengthened tool can be improved by using high-quality cold-rolled metal.

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КОМПЛЕКСНАЯ ОЦЕНКА СТЕПЕНИ СТРУКТУРНОГО СОСТОЯНИЯ УПРОЧНЯЮЩИХ НАНОПОКРЫТИЙ

Т.С. Скобло*, С.*П. Романюк, А.И. Сидашенко, В.С. Таран, А.В. Таран, И.И. Дорожко, Н.Н. Пильгуй

Выполнены исследования степени структурной неоднородности покрытий TiN, полученных на поверхности тонколистовой холоднокатаной стали марки 65Г в стандартном PVD- и ВЧ-режимах осаждения при помощи оптико-математического метода. Оценены такие факторы как диффузия химических компонентов, плотность структуры и интенсивность возникающих деформаций, влияющие на стабильность инструмента в процессе эксплуатации.

КОМПЛЕКСНА ОЦІНКА СТУПЕНЮ СТРУКТУРНОГО СТАНУ ЗМІЦНЮЮЧИХ НАНОПОКРИТТІВ

Т.С. Скобло*, С.*П. Романюк, А.І. Сидашенко, В.С. Таран, А.В. Таран, І.І. Дорожко, Н.М. Пільгуй

Виконано дослідження ступеню структурної неоднорідності покриттів TiN, отриманих на поверхні тонколистової холоднокатаної сталі марки 65Г у стандартном PVD- і ВЧ-режимах осадження за допомогою оптико-математичного методу. Оцінені такі фактори, як дифузія хімічних компонентів, щільність структури та інтенсивність виникаючих деформацій, які впливають на стабільність інструмента в процесі експлуатації.