

A.T. Berdibekov¹, V.Ch. Laurinas², A.V. Dolya¹, V.V. Gruzin¹, S.A. Guchenko^{2*}, A.S. Baltabekov²

¹*Research Institute of Arms and Military Equipment of the Military Research Center of the National Defense University named after the First President of the Republic of Kazakhstan – Elbasy, Astana, Kazakhstan;*

²*Karaganda Buketov University, Karaganda, Kazakhstan*

*Corresponding authors e-mail: guchen@mail.ru

Possibility of using inexpensive steel protected by deposited vacuum-arc coatings as a basis for parts and tools coating technology

The aim of the work was to identify the possibility of using under extreme conditions parts made of inexpensive steel, protected by sequentially deposited films — Cr and then simultaneously TiN + CrN, micron thickness. Steel grade St3 (Fe ~ 97%) was chosen as the basis. In our country, as in many others, this steel is not only the most common structural material with easy processing and low cost, but also produced in large volumes. The deposition of two consecutive layers of films — Cr and TiN + CrN on steel substrates was carried out using vacuum-arc technology (Arc-PVD). The measurements were carried out after the process of periodic heating of the samples to a temperature of 650⁰C, followed by rapid cooling, implemented by immersing the samples in water. The main parameters of the coating were measured — wear resistance, heat resistance, microhardness. Each thermal effect lasted 20 hours, and the entire heating-cooling process was carried out 5 times. The results obtained in this work can be considered very promising for the protection of inexpensive steels.

Keywords: vacuum-arc technology, protective coating, wear resistance, heat resistance, microhardness.

Introduction

Starting from the end of the sixties of the last century, cutting tools with protective coatings applied to them began to appear on the international market. Since then, various methods have been used for applying coatings for various purposes to machine parts and mechanisms. Among a wide range of technologies for applying protective coatings, vacuum ion-plasma methods find great attention [1-6]. Their feature is the direct conversion of electrical energy into technological impact, based on structural-phase transformations in the precipitated condensate.

Currently, work is being intensively carried out to save expensive metals. With their help, cutting tools are made, as well as parts operating in an air atmosphere at high temperatures. One of the solutions to this problem can be the vacuum-arc deposition of protective coatings on parts made from more economical alloys. In this case, the thickness of the deposited films usually lies in the range of several microns. Such an insignificant film thickness is quite enough to, for example, significantly improve corrosion resistance, heat and heat resistance, wear resistance, and also reduce the coefficient of friction of parts. Thus, coatings meet many parameters of operational and technological requirements.

Materials and experimental details

For the deposition of two layers — Cr and TiN + CrN on steel substrates, the vacuum-arc technology (Arc-PVD) was used, which also has another name — cathode ion bombardment (CIB). Using this technology, a Cr coating was deposited in an argon atmosphere at a pressure of 0.1 Pa. Further, after removing argon from the chamber and replacing it with nitrogen, also at a pressure of 0.1 Pa, a CrN + TiN film was deposited simultaneously with two cathodes [7].

The films were deposited in an NNV-6.6-I1 vacuum setup, in which chromium and titanium cathodes were installed. The third was a plasma source with an incandescent cathode, which, at a current of 20 A, made it possible to clean the substrate surfaces by ion (Ar⁺) bombardment before coating [5]. During the deposition of coatings, the temperature of the substrates after ion cleaning was maintained at ~450⁰C, which significantly increased adhesion.

It should be noted that the design of the HNV-6.6-I1 setup allows one to deposit films on all surfaces of the substrates, since the substrates in the installation chamber rotate both around their axis and in a circle, with adjustable speeds of both rotations. This option made it possible to load 12 substrates for carrying out

the planned experiments. At least such a number of samples was necessary because the process of thermal action leads to an increase in the mass of the sample, and the measurement of wear resistance leads to a decrease. In this regard, one group of 5 samples was used to measure heat resistance, the second, also of 5 samples, was used to measure wear resistance. In addition, because when measuring wear resistance, friction contact was made at 20 points of the film surface, the sample that passed the wear resistance test was no longer used.

The substrates were made of St3 steel. They were a disk, 25 mm in diameter and 3 mm thick. Both planes of the disk were ground on a surface grinder, and since coatings were planned to be applied to all surfaces of the substrate, they were polished on a polishing machine. Next, the substrates were washed in an ultrasonic bath and treated with steam using a steam-jet device, wiped with coarse calico soaked in ethanol, placed in an oven and kept in it for two hours at a temperature of 150°C.

Results and discussion

In the first series, a TiN + CrN coating was deposited on the substrates without an intermediate Cr layer. The St3 steel substrate showed a low Vickers microhardness (194 HV) measured on an HVS-1000A instrument. The adhesion of the deposited TiN + CrN layer also turned out to be low. Probably, this led to the fact that the film cracked when trying to measure its microhardness. The crack is quite well observed along the circumference of the indenter, which is shown in Figure 1.

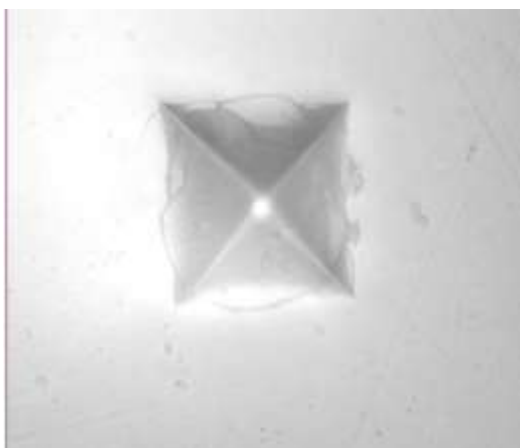


Figure 1. Image taken during microhardness measurement (400 magnification).

To exclude the occurrence of such defects in the future, a layer of chromium with a thickness of $\sim 3 \mu\text{m}$ was deposited, which has a higher microhardness compared to the substrate.

After making sure that the deposited chromium layer does not crack during microhardness measurements, layers of Cr ($\sim 3 \mu\text{m}$ thick) and TiN + CrN ($\sim 5 \mu\text{m}$ thick) were successively deposited on 12 substrates.

Figure 2 shows the spectrum taken from the applied coating, which was not subjected to thermal action, using a MIRA-3 LMU scanning electron microscope.

Next, the microhardness and wear resistance to thermal impact were measured. The results are shown in Table. To measure the wear resistance parameters, we used a tribometer developed by employees of the Research Center for Ion-Plasma Technologies and Modern Instrumentation [5] and a RADWAG AS60/220R2 electronic balance with an accuracy of 0.06 mg.

To determine the effect of thermal action on microhardness and wear resistance, as well as the heat resistance of the coating itself, the samples were placed in a muffle furnace at a temperature of 650°C [8]. The temperature was maintained in automatic mode with an accuracy of 5°C for 20 hours, then the oven was turned off and the samples were placed in distilled water at a temperature of 15°C in a twenty-liter thermostat. After rapid cooling, the samples were dried with a stream of warm air and were ready for measurements. With such a sequence, the procedures were performed in 5 cycles.

All obtained measurement results are shown in Table.

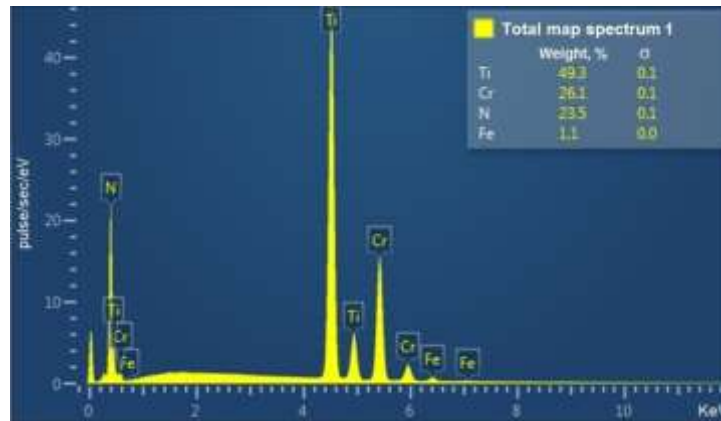


Figure 2. Spectrum taken from the central part of the sample using a scanning electron microscope before thermal treatment.

T a b l e

The table shows the dynamics of the weight gain of samples under the influence of temperature, the parameters of microhardness and the rate of abrasion of coatings

N	Microhardness, HV	Wear rate, $\mu\text{g/s}$	Dynamics of weight gain of samples exposed to a temperature of 650°C for 20 hours, $\mu\text{g/cm}^2$
Before thermal exposure	830	0.020	0
After 1 cycle	780	0.023	0.287
After 2 cycles	760	0.025	0.205
After 3 cycles	750	0.025	0.163
After 4 cycles	740	0.025	0
After 5 cycles	740	0.025	0

Figure 3 shows the spectrum taken from the coating after passing through all 5 cycles of thermal exposure using a MIRA-3 LMU scanning electron microscope.

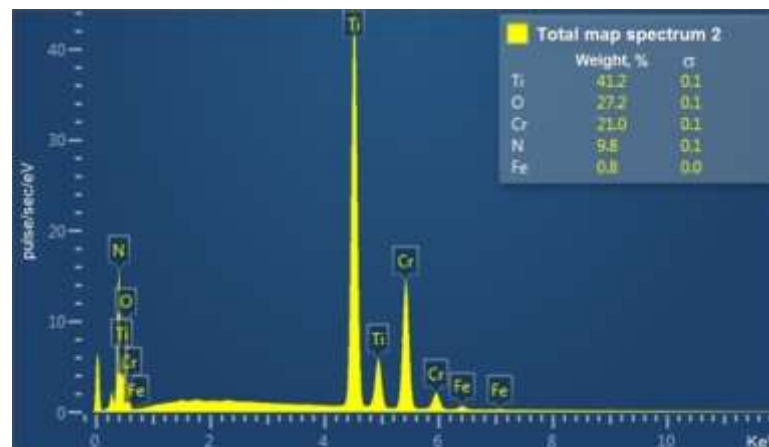


Figure 3. Spectrum taken from a coating on a substrate using a scanning electron microscope.

All parameters obtained as a result of thermal exposure are shown in Table.

Before the thermal treatment of the samples, their coating color was light yellow, the wear rate was $0.020 \mu\text{g/s}$, and the microhardness was 830 HV.

After passing through the first cycle of thermal exposure, the color of the coatings changed and became saturated yellow, the mass of the samples increased by $0.287 \mu\text{g/cm}^2$, the wear rate increased to $0.023 \mu\text{g/s}$, and the microhardness decreased to 780 HV. The second cycle changed the color to dark yellow, the mass of the samples increased by another $0.205 \mu\text{g/cm}^2$, the wear rate increased to $0.025 \mu\text{g/s}$, and the microhardness decreased to 760 HV.

The impact of the third cycle led to a change in the color of the coating, it turned purple, the weight of the samples increased by $0.163 \mu\text{g}/\text{cm}^2$, the wear rate did not change, $0.025 \mu\text{g}/\text{s}$, and the microhardness decreased to 750 HV.

Completion of the fourth and last, fifth, cycles did not lead to a change in the color of the coatings, as well as the wear rate. As for the mass gain, the further dynamics of the mass gain became equal to zero. The microhardness, which decreased to 740 HV after the fourth cycle, also did not change after the fifth.

Thus, it follows from the obtained data that over the entire studied time interval (100 hours) of keeping the samples at 650°C , the dependence of the mass increase slows down over time, and after 80 hours becomes close to zero. This fact can be explained by the fact that practically one of the components, titanium nitride, was completely converted into titanium oxides, and chromium nitride at this temperature was little susceptible to oxidation.

As for the change in the color of the films, it can most likely be assumed that titanium nitride, which gives a light yellow color, is mainly converted into oxides. Chromium nitride gives a gray color, while chromium oxides can only be green or black. At the first stage of thermal treatment, mainly TiO appears, leading to the appearance of yellow and dark yellow color of the samples. The emerging Ti_2O_3 gives a violet color, so a dark yellow color appeared.

Conclusions

Thus, using the results obtained in the work, we can draw a number of conclusions:

1) The obtained dynamics of change in the mass of the samples, which became equal to zero already after the fourth cycle and did not change any more, allows us to speak about the high thermal stability of the applied coatings at temperatures not exceeding 650°C .

2) The wear resistance of the protective coating is more than 2 orders of magnitude greater than the wear resistance of St3 steel, and is little affected by changes after thermal exposure.

3) Stabilization of microhardness and wear resistance, which also appeared after the fourth cycle, indicates the possibility of using these coatings at large temperature differences — in the range from heating 650°C to rapid cooling to 15°C .

Consequently, the results obtained correspond in a number of cases to the possibility of using parts made of St3 steel with sequentially deposited micron layers of Cr and $\text{TiN} + \text{CrN}$, and operation is also possible at high temperatures.

Acknowledgments

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А.Т. Бердібеков, В.Ч. Лауринас, А.В. Доля, В.В. Грузин, С.А. Гученко, А.С. Балтабеков

Вакуумды-доғалы технологиямен қапталған жабындармен қорғалған арзан болатты бөлшектер мен құралдардың негізі ретінде пайдалану мүмкіндігі

Негіз ретінде St3 (Fe~97%) маркалы болат таңдалды. Аталған марка біздің және өзге де елдерде болат өндеуге оңай, құны арзан, ең кең таралған конструкциялық материал болып табылады және үлкен көлемде шығарылады. Cr және TiN + CrN қатарлас екі қабықша қабаттарымен болат төсемелерін қаптау вакуумдық-доғалық технология арқылы жүзеге асырылды (Arc-PVD). Өлшем үлгілері 6500С температураға дейін мезгіл-мезгіл қыздыру процесінен кейін үлгілерді суға батыру арқылы жылдам салқындату арқылы жүргізілді. Қаптаманың тозуға төзімділік, ыстыққа төзімділік, микроқаттылық сияқты негізгі параметрлері өлшенді. Өрбір термиялық экспозиция 20 сағатқа созылды, ал барлық қыздыру-салқындату процесі 5 рет жүргізілді. Бұл жұмыста алынған нәтижелерді арзан болаттарды қорғау үшін өте перспективалы деп санауға болады.

Кілт сөздер: вакуумды-доғалық технология, қорғаныш қаптама, тозуға төзімділік, ыстыққа төзімділік, микроқаттылық.

А.Т. Бердибеков, В.Ч. Лауринас, А.В. Доля, В.В. Грузин, С.А. Гученко, А.С. Балтабеков

Возможность использования в качестве основы деталей и инструментов недорогой стали, защищенной нанесенными вакуумно-дуговой технологией покрытиями

В качестве основы была выбрана сталь марки St3 (Fe~97%). В нашей стране, как и во многих других, данная сталь не только является наиболее распространенным конструкционным материалом, обладающим легкой обработкой и низкой стоимостью, но и производится в больших объемах. Нанесение двух последовательных слоев пленок — Cr и TiN + CrN — на стальные подложки производилось с использованием вакуумно-дуговой технологии (Arc-PVD). Измерения проводились после процесса периодического нагрева образцов до температуры 650⁰С с последующим быстрым охлаждением, реализованным путем погружения образцов в воду. Измерялись основные параметры покрытия: износостойкость, жаростойкость, микротвердость. Каждое термическое воздействие длилось 20 ч, а весь процесс «нагрев–охлаждение» проводился 5 раз. Результаты, которые были получены в настоящей работе, можно считать весьма перспективными для защиты недорогих сталей.

Ключевые слова: вакуумно-дуговая технология, защитное покрытие, износостойкость, жаростойкость, микротвердость.