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## ANTIWEAR PROPERTIES OF MEDIUM-CARBON STEEL WITH Cr/CrN TYPE HYBRID COATING PRODUCED BY THE PVD METHOD

### WŁAŚCIWOŚCI TRIBOLOGICZNE STALI ŚREDNIOWĘGLOWEJ Z POWŁOKĄ HYBRYDOWĄ TYPU Cr/CrN, WYTWORZONĄ METODĄ PVD

**Key words:**

PVD treatment, hybrid coating, tribological properties.

**Abstract**

The paper presents the results of examinations of the structure, and mechanical and antiwear properties of hybrid coatings of the Cr/CrN type, consisting of chromium and chromium nitride, formed on the surface of the C45 medium-carbon steel by the Arc-PVD method. Investigations of the morphology and microstructure of hybrid coatings, as well as of their phase composition were carried out. The studies on mechanical properties included tests on hardness and Young's modulus using the nanoindentation method. Tests on adhesion were conducted using the scratch-test. Tribological properties (linear wear) of the coatings were performed by means of taper-three rolls test. The investigations showed that the linear wear of the C45 steel samples with the Cr/CrN hybrid coatings is 7–9 times smaller than that of hardened steel samples without any coatings. This indicates the excellent tribological properties of the C45 medium-carbon steel with the Cr/CrN type hybrid coatings deposited by the Arc-PVD method.

**Słowa kluczowe:**

obróbka PVD, powłoka hybrydowa, właściwości tribologiczne.

**Streszczenie**

W pracy omówiono wyniki badań budowy, mechanicznych i przeciwzużyciowych właściwości powłok hybrydowych typu Cr/CrN, składających się z chromu i azotku chromu, wytworzonych na powierzchni stali średniowęglowej C45 metodą Arc-PVD. Przeprowadzono badania morfologii i mikrostruktury powłok hybrydowych, jak również ich składu fazowego. Badania właściwości mechanicznych obejmowały pomiary twardości i modułu Younga. Badania adhezji przeprowadzono za pomocą testu rysy. Właściwości tribologiczne (zużycie liniowe) powłok oceniano metodą trzy wałeczki-stożek. Badania wykazały, że zużycie liniowe próbek ze stali C45 z powłokami hybrydowymi jest 7–9 razy mniejsze niż próbek z tej stali bez powłoki hybrydowej. Świadczy to o doskonałych właściwościach tribologicznych stali średniowęglowej C45 z powłokami hybrydowymi typu Cr/CrN, osadzonymi metodą Arc-PVD.

## INTRODUCTION

In recent years, the main activities in the field of surface engineering are aimed at the development of the technology of forming layers and coatings which are functional, that is designated to carry out certain functions in the process of service, e.g., the reduction of friction, the enhancement of corrosion resistance, etc. [L. 1–6]. Hybrid technologies that enable the adaptation of tools and machine parts to work in very difficult operating conditions are one of the solutions in this field [L. 7–11].

A pioneer of the hybrid technologies in surface engineering was Prof. Tom Bell [L. 9, 12], a world prominent specialist in the field of heat treatment and surface treatment technology, co-founder of the International Federation for Heat Treatment and Surface Engineering (IFHTSE). He was the first to recognize the possibilities of surface engineering, applicable to the extension of the service life and reliability of tooling and machine components. He brought about order into both terminology and knowledge in this field, and he drew attention to the effect of process synergy, which stems from mutual influence of the particular surface

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engineering methods utilized in the same technological process.

This article applies to the Cr/CrN type hybrid coatings, consisting of chromium and chromium nitride, formed on the steel surface by means of the arc evaporation method – Arc PVD (Arc Physical Vapour Deposition), [L. 13–15]. Hybrid technologies, consisting of a combination of different methods of surface treatment into one complex technological process, constitute currently one of the most advanced trends of investigations in the field of surface engineering [L. 7–11, 16–19].

Chromium nitride belongs to the PVD coatings group of which the technology of its formation by the Arc Evaporation method is well known [L. 20–23]. The technology of formation of hybrid coatings of the Cr/CrN type is also well-known, which, besides exhibiting very good tribological and anti-corrosion properties, are characterized by very good adhesion to the steel substrate [L. 9–11, 24–27]. A direct effect on good adhesion of these hybrid coatings to the steel substrate is exerted by the thin chromium coating deposited on its surface prior to the deposition of chromium nitride [L. 10, 16, 18].

The article discusses the results of the investigation of mechanical and antiwear properties of the Cr/CrN type hybrid coatings deposited by Arc PVD method on the surface of the C45 medium-carbon steel. The inexpensive C45 medium-carbon steel, which is widely

used in the industry for tools and machine parts, can be used with hybrid coatings can be used instead of expensive alloy steels.

## EXPERIMENTAL PROCEDURE

The Cr/CrN hybrid coatings, consisting of chromium and chromium nitride, were formed on samples made from medium-carbon steel of the C45 grade (containing 0.45% C, 0.65% Mn, and 0.25% Si). The hybrid coating deposition process was carried out by means of the arc-evaporation method (Arc PVD), utilizing a Standard 1 device at the Institute for Sustainable Technologies – NRI in Radom.

The C45 steel samples, placed inside the vacuum chamber, were initially preheated with the help of resistance heaters to a temperature of  $T \approx 300^\circ\text{C}$ , under a pressure of  $p = 5.0 \cdot 10^{-5}$  mbar. For the formation of Cr and CrN coatings, arc sources with pure chromium cathodes were used. The surfaces of steel samples were cleaned by etching, first by argon ions, and next by chromium ions.

The process of deposition of the Cr/CrN type hybrid coating was started by the deposition of a thin coating of chromium of a thickness below 1  $\mu\text{m}$ . Next, the chromium nitride coating was deposited. The processes were carried out employing technological parameters given in **Table 1**.

**Table 1. Parameters of the chromium and chromium nitride deposition processes by Arc-PVD method**

Tabela 1. Parametry procesów osadzania chromu i azotku chromu metodą Arc-PVD

Type of process	Substrate temperature $T$ [ $^\circ\text{C}$ ]	Substrate polarization voltage $U_{\text{BIAS}}$ [V]	Pressure $p$ [Pa]	Time of deposition [min]	Atmosphere
Heating	300	–	$< 1 \times 10^{-3}$	30	–
Etching by Ar ions	300	–300	$5.0 \cdot 10^{-1}$	25	Ar
Etching by Cr ions	400	–300	$5.0 \cdot 10^{-1}$	15	Ar
Deposition of Cr	400	–50	$5.0 \cdot 10^{-1}$	5	Ar
Deposition of CrN	380	–150	3.5	120	$\text{N}_2$

## METHODS OF INVESTIGATION

In order to analyse the structure and surface morphology of the Cr/CrN type hybrid coatings, microscopic observations were carried out with the aid of a Hitachi TM-3000 scanning electron microscope equipped with a BSE detector. Investigations of structure were carried out on polished metallographic cross-sections of steel samples with coatings. The X-ray phase analysis was

carried out with the aid of a Bruker D8 diffractometer with  $\text{CuK}\alpha$  X-rays.

Hardness and Young's modulus of investigated hybrid coatings were measured by means of the nanoindentation method, employing the Nano-Hardness Tester of CSM Instruments. Measurements were carried out with the Berkovich indenter using the following parameters:  $F = 10$  mN, and  $dF/dt = 20$  mN/min.

The testing of adhesion of coatings to the steel substrate was carried out using a scratch test made with a REVETEST tester [L. 28]. This tester is equipped with a measuring head with a Rockwell indenter, an optical microscope with high resolution video imaging system, a sensor of acoustic emission, a penetration depth sensor, as well as a sensor of friction and normal forces. The scratch tests were carried out using load forces from 0 to 200 N, increasing at a rate of 100 N/min. The rest of process parameters, like normal force range, scratch length, or the sensitivity of acoustic emission system, were selected during examinations.

Tribological properties of the samples, which is their wear resistance to friction, were evaluated by the three-cylinder-cone method, [L. 29, 30].

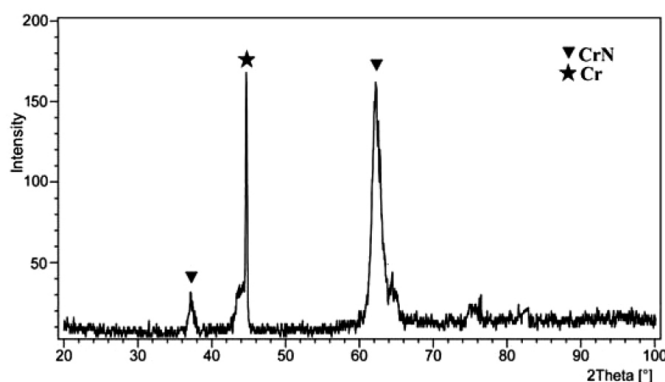
Measurements were taken with a rotating speed of the cone at  $n = 576$  r.p.m. and unit loading pressures of 50,

100, 300, and 400 MPa during a time of 100 min, applying lubrication by Lux 10 oil. The cone was made from C45 steel hardened and tempered to 30 HRC, according to the PN-83/H-04302 norm [L. 30].

## RESULTS AND DISCUSSION

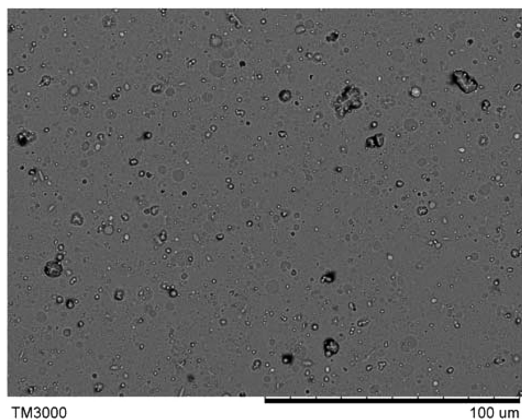
### Characterization of hybrid coatings

X-ray phase analysis of the medium-carbon steel sample with a Cr/CrN hybrid coating, consisting of chromium and chromium nitride, exhibited the presence of Cr and CrN nitride with a body centred cubic lattice and traces of the Cr<sub>2</sub>N nitride with a hexagonal lattice (Fig. 1). Similar results of X-ray phase analysis were obtained in other reports [L. 9–11, 20].



**Fig. 1. X-ray diffraction pattern (CuK $\alpha$ ) of the C45 steel surface with the Cr/CrN type hybrid coating**  
Rys. 1. Dyfraktogram rentgenowski z powierzchni próbki ze stali C45 z powłoką hybrydową typu Cr/CrN

An analysis of surface morphology of a sample with a hybrid coating, carried out with the help of a scanning electron microscope (SEM+BSE), revealed the presence



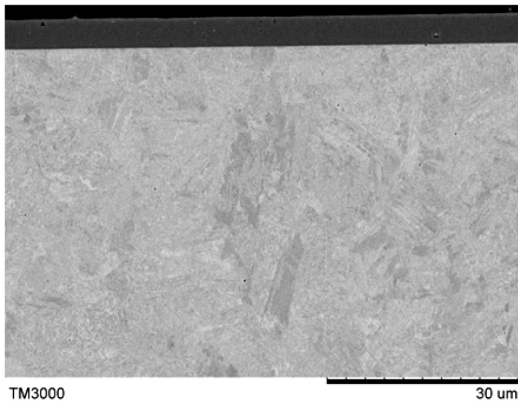
**Fig. 2. The scanning image (SEM+BSE) of the surface of the C45 steel sample with the Cr/CrN type hybrid coating**

Rys. 2. Obraz skaningowy (SEM+BSE) powierzchni próbki ze stali C45 z powłoką hybrydową typu Cr/CrN

of a homogenous, very fine microstructure with a small amount of a droplet phase, characteristic of the Arc PVD method (Fig. 2). Similar scanning electron microscope images of the surface of CrN coatings were also reported in other papers [L. 13–15, 23].

A scanning electron microscope image (SEM+BSE) of the C45 steel with a hybrid coating, obtained on polished metallographic cross-sections, is shown in Fig. 3. The dark grey Cr/CrN hybrid coating, of about 5  $\mu$ m thickness adheres uniformly to the steel substrate.

It is well known that the Cr/CrN hybrid coatings obtained by Arc-Deposition of the CrN coating on the surface of the chromium coating consist of two separate sub-zones: an external sub-zone containing CrN nitride, and an inner sub-zone containing chromium, placed between the CrN coating and the steel substrate [L. 9–11, 24–26]. However, in this work, the boundary separating the very thin inner sub-zone of the hybrid coating of a thickness below 1  $\mu$ m containing chromium from the outer sub-zone, containing the CrN nitride was not revealed. The boundary between the two sub-zones, Cr and CrN, in the Cr/CrN hybrid coating was revealed



**Fig. 3. The scanning image of the C45 steel with the Cr/CrN type hybrid coating**

Rys. 3. Obraz skaningowy stali C45 z powłoką hybrydową typu Cr/CrN

using a microscope with much higher resolution and higher magnification by Smolik [L. 9].

### Hardness and Young's modulus

Hardness measurements of the Cr/CrN hybrid coatings, characterized by small thickness (about 5  $\mu\text{m}$ ) and their Young's modulus were made with the help of the Nano-Hardness Tester, which enables a selection of test loads from 0.05 to 500 mN, as well as a precise selection of penetration depth of the indenter into the material being tested, eliminating the influence of the substrate on the obtained results. Measurements of hardness and of Young's modulus of the Cr/CrN hybrid coatings were made maintaining the maximum penetration of the indenter within  $< 10\%$  of the coating thickness. Results of measurements are given in **Table 2**.

**Table 2. Results of measurements of hardness and Young's modulus**

Tabela 2. Wyniki pomiarów twardości i modułu Younga

Coating type	Thickness $g$ [ $\mu\text{m}$ ]	Hardness $H$ [GPa]	Hardness HV [Vickers]	Young's modulus $E$ [GPa]
Cr/CrN	4.68	$28 \pm 2.8$	$2627 \pm 260$	$337 \pm 46$

An analysis of results of the hardness measurements showed that the Cr/CrN hybrid coating is characterized by high hardness, 2627 HV, while its Young's modulus is similar to values determined in other reports [L. 9–11, 22, 23, 27].

### Adhesion

Investigations of the adhesion of the coatings was carried out by means of the scratch test with a gradual increase of the normal force loading, which applied a load on a penetrator realizing the scratch on the surface of sample. As an effect of the penetrator's pressure, the increasing elastic-plastic deformations of coating occur until the emergence of damages, which are the effect of the loss of coating adhesion or decohesion.

The measure of adhesion is a critical force, which is the lowest normal force causing the loss of adhesion between the coating and the steel substrate [L. 28]. The

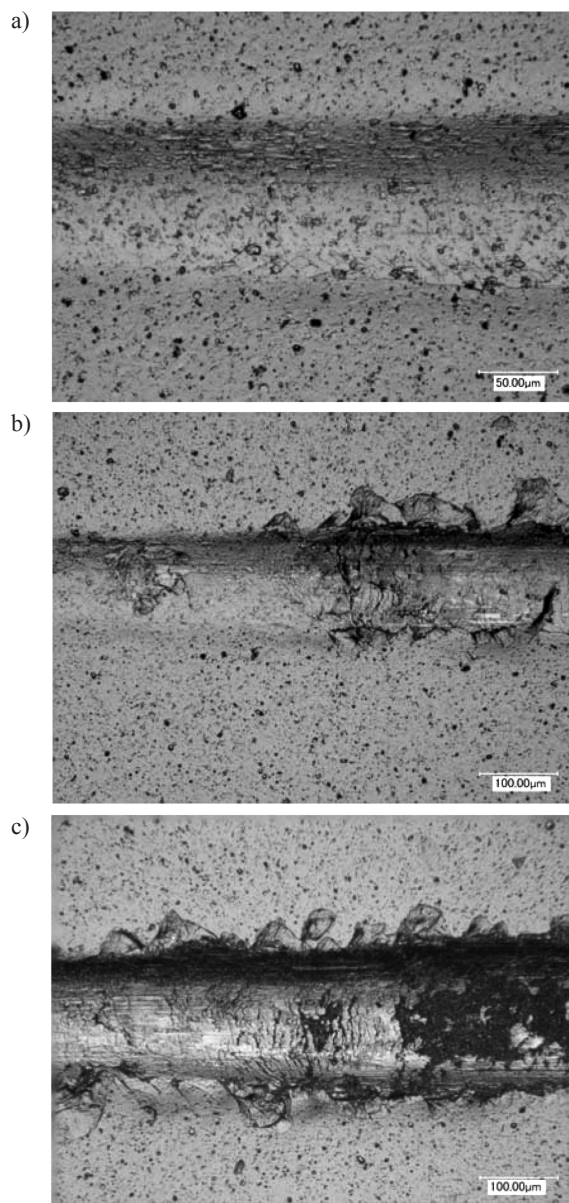
adhesion of coatings was evaluated by the Revetest CSM tester, using load forces from 0 to 200 N, increasing at the rate of 100 N/min. The values of the force loading the penetrator at which destruction of adhesion occurs were determined based on a microscopic analysis of damages in the region of scratch, changes in the value of the coefficient of friction ( $\mu$ ) between the penetrator and the coating, as well as changes in the acoustic signal (AE) emitted in the process of scratch. The effect of penetrator loading on the following three different adhesion parameters was analysed: Fc1, at which cracks are generated in the tested coating; Fc2, where local adhesion loss of the coating occurs in the area of the scratch and its edge; and Fc3, where decohesion of the entire coating from the substrate occurred. Three scratches were made on each sample. Adhesion parameters determined during scratch test of the C45 steel samples with the hybrid coatings are shown in **Table 3**.

**Table 3. Adhesion parameters determined during scratch test of the C45 steel samples with the Cr/CrN type hybrid coatings**

Tabela 3. Parametry adhezji określone w teście zarysowania dla próbek ze stali C45 z powłokami hybrydowymi typu Cr/CrN

Coating type	Adhesion parameter	Critical load value [N]			
		Scratch 1	Scratch 2	Scratch 3	Average
Cr/CrN	Fc1	20	13	17	<b>17</b>
	Fc2	34	17	23	<b>25</b>
	Fc3	103	116	92	<b>104</b>

The microscopic images of scratch, obtained during scratch test of the steel sample with the hybrid coating, are shown in **Fig. 4**.



**Fig. 4.** The microscopic images of scratch, obtained during scratch test of the C45 steel sample with the Cr/CrN hybrid coating: a) Fc1 - the first cracks of the hybrid coating, b) Fc2 - the local adhesion loss of the hybrid coating, c) Fc3 - decohesion of the hybrid coating from the substrate

Rys. 4. Obrazy mikroskopowe rysy otrzymanej podczas testu rysy próbki ze stali C45 z powłoką hybrydową typu Cr/CrN: a) Fc1 - pierwsze pęknięcia powłoki hybrydowej, b) Fc2 - lokalna utrata adhezji powłoki hybrydowej, c) Fc3 - dekohezja powłoki hybrydowej od podłoża stali

The first cracks of the Cr/CrN hybrid coating were observed under a load  $F_{c1} = 17$  N (**Table 3**, **Fig. 4a**). The local adhesion loss of the coating, occurring in the area of the scratch and its edge, appeared under

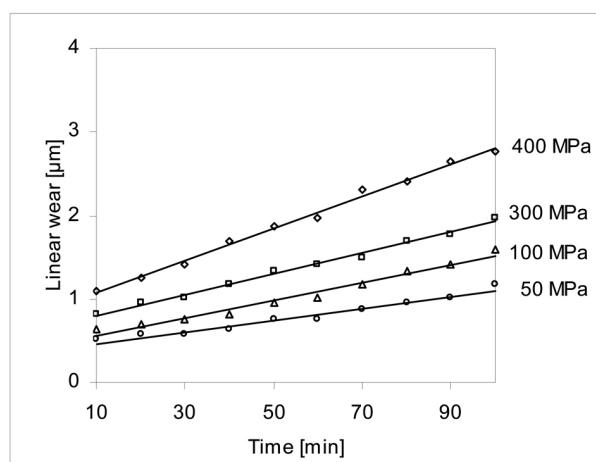
a load  $F_{c2} = 25$  N (**Fig. 4b**). The total decohesion of the coating from the substrate came about only under a load  $F_{c3} = 104$  N (**Fig. 4c**).

Results of tests show good adhesion to the steel substrate of hybrid coatings. Similar results were reported in other papers [L. 9–11, 22, 27].

### Antiwear properties

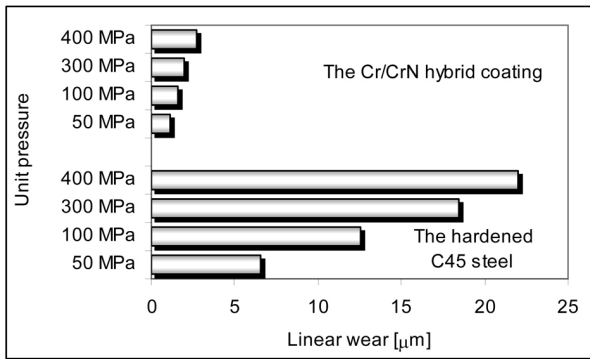
Linear wear of hybrid coatings were evaluated on the basis of sliding wear tests with concentrated contact [L. 29, 30]. Wear resistance testing by the three cylinder-cone method was carried out on samples of the C45 steel with the Cr/CrN hybrid coatings and for hardened steel substrates without any coatings. Linear wear of the C45 steel samples with the hybrid coatings as a function of friction time and unit pressures is shown in **Fig. 5**. A comparison of the linear wear of the C45 samples with hybrid coatings and hardened steel samples without any coatings, for various unit pressures and friction time of 100 min, is shown in **Fig. 6**.

The investigations showed that the linear wear of the C45 steel samples with the Cr/CrN hybrid coatings is 7–9 times smaller than that of hardened steel samples without any coatings. For example, linear wear of samples with the Cr/CrN hybrid coatings was  $1.6 \mu\text{m}$  under 100 MPa unit pressure for a friction time of 100 min, while that of samples made from the same C45 steel, but only hardened, under the same conditions, was  $11.8 \mu\text{m}$ . Linear wear of samples with the hybrid coatings was  $1.9 \mu\text{m}$  under 300 MPa unit pressure for a friction time of 100 min, while that of samples made from the C45 hardened steel was  $18.4 \mu\text{m}$ . This indicates the excellent antiwear properties of the hybrid coatings.



**Fig. 5.** Linear wear of the C45 steel samples with the Cr/CrN type hybrid coatings vs. friction time and unit pressures

Rys. 5. Zużycie liniowe próbek ze stali C45 z powłokami hybrydowymi typu Cr/CN, w zależności od czasu tarcia i nacisków jednostkowych



**Fig. 6. Linear wear of the C45 steel samples with the Cr/CrN type hybrid coatings and hardened C45 steel samples, without any coatings, for friction time of 100 min and various unit pressures**

Rys. 6. Zużycie liniowe próbek ze stali C45 z powłokami hybrydowymi typu Cr/CrN i próbek ze stali 45 utwardzonej, bez powłok, dla czasu tarcia 100 min i różnych nacisków jednostkowych

## SUMMARY AND CONCLUSIONS

The subjects of the study were hybrid coatings of the Cr/CrN type on the C45 medium-carbon steel, obtained by deposition of a thin chromium coating (of a thickness below 1 μm) on its surface prior to the deposition of chromium nitride by the Arc PVD method. The thickness of the Cr/CrN hybrid coatings was about 5 μm. X-ray phase analysis of the surface of the C45 steel sample

with the Cr/CrN coating showed that it is composed mainly of chromium nitride (CrN) and chromium (Cr). Similar results of X-ray phase analysis were obtained in other reports [L. 9–11, 20].

The hardness of the Cr/CrN coating and its Young's modulus, evaluated with the aid of the Nano-Hardness Tester, amounted to  $H = 2627$  HV and  $E = 337$  GPa (Table 2). Similar results were reported in other papers [L. 9–11, 22, 23, 27].

Adhesion of the Cr/CrN coatings to the steel substrate, measured by the scratch test, was very good. Total decohesion of the coating was observed only under a load of  $F_{c3} = 104$  N (Table 3).

The investigations of linear wear using the three cylinder-cone method showed that the linear wear of the C45 steel samples with the Cr/CrN hybrid coatings is 7–9 times smaller than that of hardened steel samples without any coatings. This indicates the excellent antiwear properties of the hybrid coatings in conditions of sliding friction and concentrated contact. Very good results of these tests of steel samples with the Cr/CrN hybrid coatings were also reported in other publications in which the ball-on-disc method was used to assess antiwear properties [L. 6, 9, 25, 27].

Therefore, the inexpensive C45 medium-carbon steel with the Cr/CrN type hybrid coatings deposited by Arc PVD method, can be used in the industry, instead of expensive alloy steels, for machine parts exposed during service to friction wear, for example, pump impellers, spindles, or valves.

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