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Tribology of Nanostructured Nickel Chemical Coatings

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Abstract: *The abrasive wear and the wear resistance of composite nickel + SiC coatings are investigated. The coatings are deposited by the method for electroless nickel plating EFTTOM-NICKEL developed in Technical University in Sofia. Nanosized particles of SiC are used as a strengthened material. The size of the particles is between 35-40 nm.*

The thickness of the coatings is 50 µm. The investigation of the coatings deposited on the different roughness surfaces are performed. Some of the samples are thermal processed at 300ºC, 6 hours after deposition process.

The methods for wear resistance testing is developed and the experimental results for the dependence of the massive wear, wear speed, intensity of wear and wear resistance on the friction road and the time of a contact interaction are obtained.

Keywords: *Tribology, wear, coatings, nanotribology, nickel, thermography.*

1. Introduction

The calendaring is a process of material pressing with shafts (calendars) to obtain a sheet material – paper, card board, leader, foil, etc. The calendars work at different temperature and dynamic's conditions in production and as a result of complicated contact interactions under friction they are being wearied out [1-4]. In practice the extension of the calendars work life is achieved by the deposition of thin wear resistance hard chrome coatings.

The research group from the Institute of Information and Communication Technologies at the Bulgarian Academy of Sciences and the Technical University in Bulgaria are developing a project for the replacement of toxic chrome by nickel coatings strengthened with micro and nanosized particles.

The improving of the wear resistance of composite nickel coatings is observed using nanosized diamond particles (nanodiamond) and micro sized cBN particles as a strengthened material. The thermal processing of the coatings improves their wear resistance behaviour [7].

In the present work the investigation of the abrasive wear and the wear resistance of composite nickel coatings with nanosized SiC particles are performed in a laboratory.

2. Exposition

2.1. Description of the coatings

Nanostructured composite coatings are obtained by electroless nickel plating method EFTTOM-NICKEL, developed at the Technical University in Sofia [6]. SiC nanoparticles with average size of 35-40 nm are used as strengthened phase. The density of the SiC nanoparticles in the coating is between 5-7 Volume %. Five different type of coatings are tested: electroless nickel coating (Ni), composite nickel coating with nanoparticles (Ni-SiC). The coatings are deposited on different roughness surfaces Ra = 0.3 µm and Ra = 2.1 µm. Some of the samples with the composite coatings are put to thermal processing at 300°C, 6 hours to improve the microhardness and the adhesion of the coating to the padding. The microhardnes is measured by Knoop method under load of 0.5 N. The coatings are deposited on the steel samples and their thickness is 50 µm. The thickness is measured by the device "Pocket LEPTOSKOP 2021 Fe" on 10 surface points and the average value is taken. The data for the tested samples are presented in Table 1.

Table 1. Coatings data: nanoparticles content, thermal processing, microhardness and roughness

No	Content	Thermal processing	HK0.02	Ra, µm
1	Ni	300°C, 6 h	860	0.3
2	Ni + SiC	–	473	0.3
3	Ni + SiC	300°C, 6 h	980	0.3
4	Ni + SiC	–	485	2.1
5	Ni + SiC	300°C, 6 h	940	2.1

2.2. Wear: Methods of testing and experimental results

The experimental tests are performed in the Tribology center at the Technical University in Sofia.

The Mmthods for abrasive wear test of nickel coatings is developed using a device working on a cinematic scheme back to back disc. The functional scheme of the device is shown on Fig. 1.

The disc sample 1 (solid) with coating 2 is fixed on a horizontal disc 3, which is moved by an electric motor 4 with constant angular speed $\omega = 1\ s^{-1}$ around a vertical axis. The antibody 5 is a disc from a special abrasive material CS10. The desired normal load P in the contact surface K is set through mounted in the antibody axle 6, which is operated by a special device 8. In this way the body 1 and the antibody 5 are fixed on two cross axes. Upon a constant angular speed ω = const of the sample 1 and upon constant nominal contact pressure P_a = const, the friction in the contact surface K keeps constant rotation speed of the antibody 5.

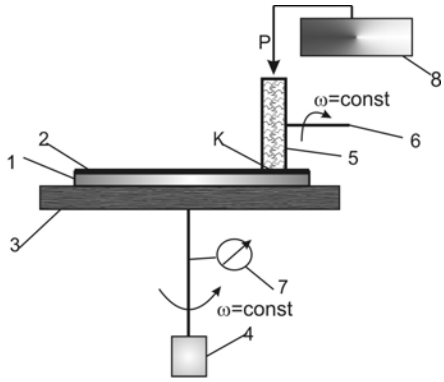


Fig. 1. Functional design of the device for wear testing under friction upon fixed abrasive

The test method consists of the following operations:

– Preparation of the samples with the same ring shape and size before coating deposition. Mechanical treatment, namely grinding and polishing to ensure equal surface roughness Ra=0.3 μm and Ra=2.1 μm. This is a binding requirement for the reliability of the tribological test accomplishment. This ensues from the specific character of the plating technology where the coatings copy the sample surface and the coating thickness is a few microns.

– The weight of the sample is weighed before and after a determinate number of disc rotations by an analytical balance WPS 180/C/2 précised to 0.1 mg. The samples are treated with a special solution to neutralize the static electricity before the weighting.

– Sample 1 is fixed on a horizontal disc 3 and by the lever system in device 8 the desired normal load P is set. The friction road L is determined by the number of cycles N, accounted by a cyclometer 7.

The test basic parameters are:

- absolute massive wear Δm ;
- speed of massive wear $\dot{m}$, mg/min;
- intensity of wear i – this is the lost coating thickness for one friction cycle,

(1) $$i = \frac{m}{\rho . A_a . S},$$

where: ρ is the coating density; A_a is the nominal interaction contact surface: A_a = 26×10^{-6} m^2; S is the friction road, estimated by the number of cycles N: $S = 2\pi . R . N$ m, where R is the distance between the rotation axis of the bearing disc and the mass center of the contact place between the sample 1 and the contra body 5;

- absolute wear resistance

(2) $$I = \frac{1}{i} = \frac{\rho . A_a . S}{m},$$

i is the intensity of wear;

- specific wear intensity

(3) $$i_s = \frac{m}{S . A_a} \quad \text{mg/(m.mm}^2\text{)};$$

- specific wear-resistance

(4) $$I_s = \frac{1}{i_s},$$

the specific wear-resistance I_s indicates the sliding way S covered by the contact area of 1 mm^2, in order that a mass 1 mg of it will be destroyed. The dimension is (m.mm^2)/mg;

- nominal contact pressure $P_a = P/A_a = 47.15$ N/cm^2.

The experimental results of all type of coatings (see Table 1) are shown in Figs 2-5 for massive wear Δm, the speed of the massive wear $\dot{m}$, the intensity of wear i, as well as the wear resistance I, the specific wear intensity i_s and the specific wear-resistance I_s. The results for the intensity of wear and the wear resistance of all types of coatings are presented in Figs 6 and 7.

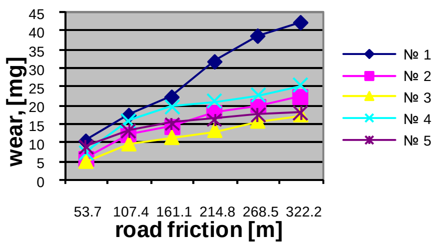


Fig. 2. Dependence of the massive wear m on the rotation road

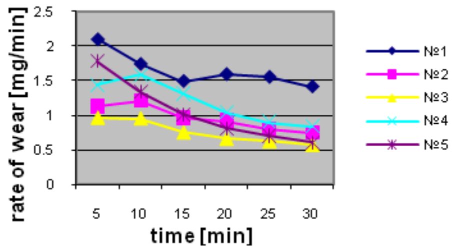


Fig. 3. Dependence of the intensity of wear $\dot{m}$ on time

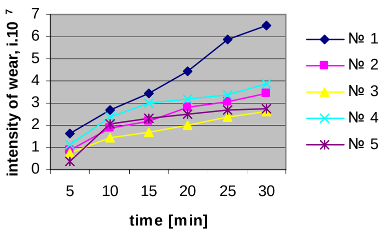


Fig. 4. Dependence of the intensity of wear i on time t

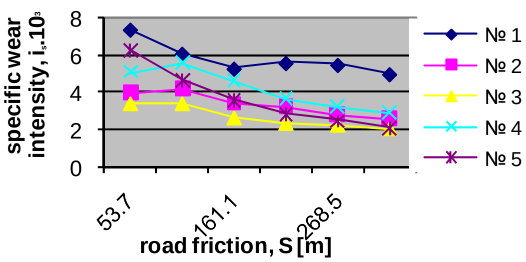


Fig. 5. Specific wear intensity i_s vs sliding way

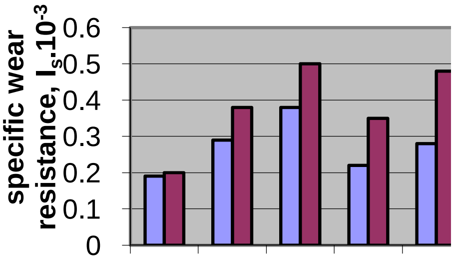


Fig. 6. Table gram of the specific wear-resistance I_s

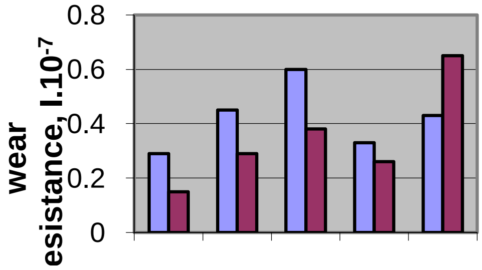


Fig. 7. Table gram of the wear resistance I

2.3. Tribothermal effects: approach and results

The effect of load, velocity, environmental conditions, etc., on the wear rate can be accounted for by considering the frictional energy dissipation within a contact. It is generally accepted that most of the frictional work during the wear process is converted into heat, which in turn, raises the interface temperature. The temperature, and particularly the temperature gradient, within the mating bodies plays an important role in the assessment of the tribothermal effects and wearing out. An experimental set up has been developed for determination of the temperature rise at the contact surface by infrared camera ThermoCam SC640, FLIR. A thermographic technique is employed to capture real-time images of the surface temperature distribution during the abrasive wear test of nickel coatings [6].

By this technique the magnitude and distortion of the near-surface temperature within a fretting contact has been assessed. The steady-state heat generation due to friction depends directly proportional on the coefficient of friction, the velocity and the normal load. The results of the experiments for samples 1, 2 and 3 show that there is a linear relationship between the temperature rise at the interface and the heat transferred to the disc of special abrasive material CS10. This material has got high emissivity that was measured before the test and used for emissivity map creation. Fig. 8 shows the result of the temperature rise plotted against the heat dissipation. The linearity between the temperature rise and the temperature of the abrasive disk can be expressed as $\Delta T = ST_1$,where S is the slope of the line.

The relation for wear coefficients K_1 and K_2 of two examined samples is

$$\frac{K_1}{K_2} = \left(\frac{\eta_1 \mu_1 H_1 S_1}{\eta_2 \mu_2 H_2 S_2}\right)\frac{\omega_1 \Delta T_2}{\omega_2 \Delta T_1}$$

where H is the hardness, A is the area of the contact, ω is the wear rate, and η is the heat partitioning factor [7]. The indices respond to the numbers of tested samples.

The last equation is the base of the methodology for evaluation of the wear coefficient merely by measuring the contact temperature. This method provides a simple and effective technique to quantitatively characterize the wear behaviour of a friction system.

Regarding the image subtraction as an image conversion function might seem strange, but that is really what this type of work is all about. It is possible by using

ThermaCam Researcher 2.9 to get another sequence, containing difference images as a result. The most important usage of this function is for making comparisons. We have compared images of the same (or similar) object(s), taken at different times, in order to detect the changes in temperature, position or shape.

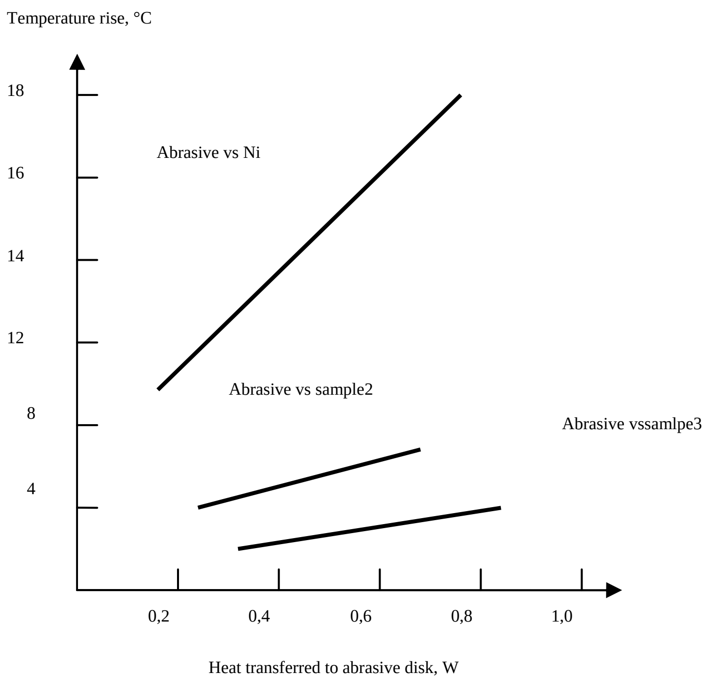


Fig. 8. Temperature rise plotted against heat dissipation

When the subtraction is over, and the output images are displayed, it may be noticed that the measurement units have changed. From °C to *dC*, for instance. This is because there is no longer any absolute temperatures in the real sense, just differences indicating how much the temperature of the object has changed (Fig. 9). On Fig. 10 some of the thermal images used in the proposed approach are shown.

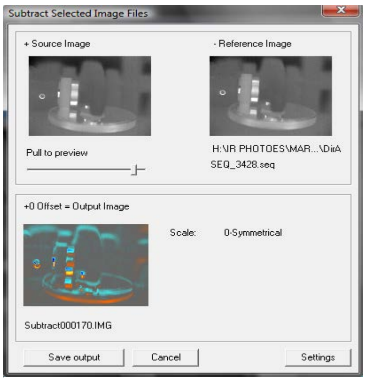
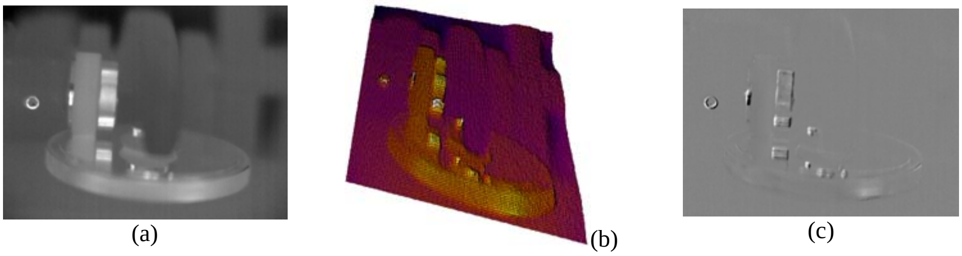


Fig. 9. Thermal image subtracting



(a) (b) (c)

Fig. 10. Thermal image for surface temperature measurement (a); 3D thermal image (b); negative subtracted image (c)

As a result it can be concluded that the ratio K_1/K_3 is about twice bigger than the ratio K_1/K_2.

3. Analysis of the results and conclusions

The variation in the massive wear value during the process of contact interaction under friction upon an abrasive surface is linear after a time of friction, corresponding to the contact system recast. The recast process is with different duration: the higher value is achieved for pure nickel coating: (No 1) – $S = 268.5$ m, the lower – for thermal processed composite coatings (No 3 and No 5) – $S = 107.4$ m (Fig. 2).

The maximum wear is observed for pure nickel coating (No 1). The thermal processed composite coatings have less wear than the coatings without thermal processing. The minimum wear is observed for the coatings with the smallest roughness (sample No 3 – $R_a = 0.3$ µm – Fig. 2).

The massive wear speed and the intensity of wear have a pointedly nonlinear character over time (Figs 3 and 4). This is in confirmation of the thesis for the nonstationary character of the contact interactions, irrespective of the constant value of the external dynamic factors – speed of sliding and nominal contact pressure. The contribution is of the contact surface and more of the roughness.

The analysis of the wear Table gram (Fig. 5) shows the significant influence of the roughness value at different stages of the contact interaction. It seems that the different resistance to abrasion is observed for the same coatings at different duration (cycles) of interaction. It can be argued that the higher wear resistance is achieved for thermal processed composite nickel coatings (No 3 and No 5). But at different cycles the wear resistance depends on the difference in the degree of the roughness. At $N = 900$ cycles the coating (No 3) with less roughness ($R_a = 0.3$ µm) has 1.6 times higher wear resistance than the rough coating – No 5 ($R_a = 2.1$ µm).

With twice as many cycles ($N = 1800$ cycles) contrariwise I_s observed – the rougher coating has 1.5 times higher wear resistance.

The investigation of the coatings properties during the overtime process, in stationary conditions, as well as the dependence on the roughness, microstructure and etc., are the subject of further authors' research.

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Трибометрия химических покрытий никеля наноразмера

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(Р е з ю м е)

В работе исследуется абразивный износ и устойчивость к износу покрытий никеля +SiC. Покрытие наносится методом EFTTOM-NICKEL, разработанным в Техническом университете в Софии. Частицы SiC наноразмера используются как укрепляющий материал. Размер этих частиц в границах 35–40 nm.

Толщина покрытий 50 μm. Проведены исследования покрытый для разных степеней шероховатости поверхности. Некоторые элементы обработаны термически при 300° С шестъ часов после процесса нанесения.

Предложен метод для проверки износа и получены экспериментальные результаты о скорости износа, интензитете износа и устойчивости износа по отношении фрикционного пути и времени контактного взаимодействия.