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ABRASIVE WEAR OF CHEMICAL NICKEL COATINGS WITH BORON NITRIDE NANO-PARTICLES

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ABSTRACT
Using the disk-abrasive roller test rig, wear behaviour of chemical Ni coatings containing boron nitride nanoparticles has been studied. The coatings were deposited by means of EFTOM-Ni technology method patented at the Technical University - Sofia. Comparative study has been done of wear resistance of the heat treated (heating during 6 h at 300°C) and as deposited (no additional heat treatment) coatings, correspondingly with contents of particles of 10 μm averaged size and without nanoparticles addition.

KEYWORDS: Electroless deposition, Nickel coatings, BN nanoparticles, Abrasive wear

1. INTRODUCTION
The study deals with improvement of the wear resistance of working shafts (calenders) involved in the production of sheet-formed and foliate materials – paper, cardboard, leather, etc. Calender shafts work in dynamic conditions, i.e. in various temperature and contact interaction regimes, and are subjected to severe wear [1,2]. Usually, their resource is improved through wear resistant thin and hard Cr coatings. A team of researchers from the Institute for Information and Communication Technologies at the Bulgarian Academy of Sciences and the Mechanical Engineering Faculties in TU-Sofia and Belgrade develop a Project aiming replacement of the ecology non-friendly industrial Cr by Ni chemical coatings. High wear resistance is achieved through imbedding of various nano- and micro-size particles (SiC, diamond, BN, etc.) in the coating [3-5].

The Ni chemical coatings are obtained by the method of chemical electroless (no external source of electric current) deposition, also known as the “Electroless nickel plating”. This method is a reduction process between the positive metal ions $M^{n+}$ and negative electrons $e$:

$$M^{n+} + ze \rightarrow Me$$

where $z$ is the valence of the metal ion.

The necessary electrons are obtained as result of chemical reaction between the solution containing the salt of the considered metal and the surface of the sample to be coated. In this reaction the Ni ions of the solution, according to their valence, accept a certain number of electrons and the metal ions transform in neutral atoms (Me), which build gradually the crystal lattice of the coating [6,7].

The paper considers a comparative study of the wear properties of Ni chemical coatings containing boron nitride (BN) nanoparticles concerning both heat treated and non heat treated coatings.
2. COATINGS DEPOSITION AND EXPERIMENTAL DETAILS

Four type of chemical Ni coatings have been studied under equal conditions of dry sliding abrasion: 1) Ni coating without nanoparticles in as deposited conditions; 2) Ni coating without nanoparticles and with additional heat treatment (heated at 300 °C during 6 hours); 3) Ni coating containing BN nanoparticles of average size 10 nm in as deposited conditions; 4) Ni coating containing BN nanoparticles of average size 10 nm and with additional heat treatment (heated at 300 °C during 6 hours). Designation of tested coatings and their microhardness are shown in Table 1 gives the description of the samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Coating</th>
<th>Designation</th>
<th>Microhardness HK 0.02</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ni non-heated</td>
<td>Ni</td>
<td>790</td>
</tr>
<tr>
<td>2</td>
<td>Ni heated at 300 °C for 6 h</td>
<td>Ni&lt;sup&gt;HT&lt;/sup&gt;</td>
<td>860</td>
</tr>
<tr>
<td>3</td>
<td>Ni with BN nanoparticles non-heated</td>
<td>Ni-BN</td>
<td>456</td>
</tr>
<tr>
<td>4</td>
<td>Ni with BN nanoparticles heated at 300 °C for 6 h</td>
<td>Ni-BN&lt;sup&gt;HT&lt;/sup&gt;</td>
<td>1050</td>
</tr>
</tbody>
</table>

HT – heat treated

The coatings are obtained under equal conditions: composition, concentration and temperature of the chemical solution, time of coating deposition, and nanoparticle concentration of 5 vol. %. The coatings are deposited on steel specimens with composition given in Table 2.

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>S</th>
<th>Mn</th>
<th>P</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Fe</th>
<th>Percentage</th>
</tr>
</thead>
</table>

The specimens are round disks with 100 mm diameter and 3 mm thickness, with equal roughness of $R_s = 0.453 \, \mu m$ measured by profilometer TESA Rugosurf 10G (Figure 1).

![Figure 1: TESA Rugosurf 10G and sample roughness profile.](image)

The microhardness is determined using Knoop method with 0.2 N load (Table 1). Coatings with thickness of 12.5 $\mu m$ are created. The thickness is obtained as average value of 10 measurements by the device Pocket-LEPTOSKOP 2021 Fe (Figure 2) on both sides of the disk.

The abrasive wear tests are done using the disc-abrasive roller rig (Figure 3). A disc specimen (1) with coating (2) is fixed on the horizontal base disk (3) driven with constant rotational speed of 60 rpm by electric motor (4). Abrasive roller (5), Taber abrading wheel Calibrase<sup>®</sup> CS-10, is mounted on horizontal axis (6) and provides through weights (8) the necessary normal load (P)
in the contact zone (K). Abrasive roller with thickness of 13 mm is driven with constant rotational speed. Wear is determined as the difference between the initial specimen mass and its mass after given number N of cycles counted by the counter (7). The mass is measured by the electronic balance WPS 180/C/2 with 0.1 accuracy.

![Figure 2: Pocket-LEPTOSKOP 2021 Fe](image1)

![Figure 3: Principle scheme of the disk-abrasive roller tribometer](image2)

All coatings are studied at equal load of 2.5 N. The average sliding speed was app. 0.232 m/s. The sliding distance (s) is calculated by the Equation (2):

$$s = 2r \cdot \pi \cdot N$$  \hspace{1cm} (2)

where $r = 37$ mm is the distance between the rotational axis of disc specimen (1) and the mass centre of the contact area (K), see Figure 3, and N is the number of rotating cycles.

Wear of the coatings are determined at various number of cycles, i.e. at N = 200, 400, 600 and 800, and corresponding wear curves are constructed. Mass wear rate ($W_m$) in mg/m is calculated by fitting the wear curves (it is the slope of wear curve), assuming that the steady-state wear was from the beginning of the tests (which is common thing for the abrasion wear). Linear wear rate is more convenient for engineering practice than mass wear rate. Linear wear rate ($W_h$) in mm/m, i.e. the intensity of the coating thickness loss ($h$) is calculated by the Equation (3):

$$W_h = \frac{h}{s} = \frac{m}{s \cdot \rho \cdot A_n} = \frac{W_m}{\rho \cdot A_n}$$  \hspace{1cm} (3)

where $m$ is the mass loss in mg, $\rho = 7.8$ mg/mm$^3$ is the coating density, and $A_n = 3.02 \times 10^5$ mm$^2$ is the coated disc specimen wear area (ring with outer radius of 43.5 mm and inner radius of 30.5 mm).

Influence of heat treatment and addition of BN nanoparticles on the reduction of wear rate are analysed by comparing the wear rates of testing coatings. These influences are expressed in percentage through the reduction index (Equation 4), which is the ratio of the difference of both specimen wear rate and the wear rate of the reference specimen.

$$R_{ij} = \frac{W_{hi} - W_{nj}}{W_{hi}} \times 100, \%$$  \hspace{1cm} (4)

3. RESULTS AND DISCUSSION

Experimental results for mass wear at various cycle numbers are given in Table 3.
Table 3. Abrasive wear of tested coatings

<table>
<thead>
<tr>
<th>Test parameter</th>
<th>Coating designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of cycles</td>
<td>Sliding distance, m</td>
</tr>
<tr>
<td>----------------</td>
<td>---------------------</td>
</tr>
<tr>
<td>200</td>
<td>46.5</td>
</tr>
<tr>
<td>400</td>
<td>93.0</td>
</tr>
<tr>
<td>600</td>
<td>139.5</td>
</tr>
<tr>
<td>800</td>
<td>186.0</td>
</tr>
</tbody>
</table>

Using the results from Table 3, wear curves are constructed, correspondingly in Figure 4 for coatings without nanoparticles and in Figure 5 for coatings with BN nanoparticles.

**Figure 4:** Mass loss vs. sliding distance for Ni coatings without nanoparticles (with and without additional heat treatment)

**Figure 5:** Mass loss vs. sliding distance for Ni coatings with BN nanoparticles (with and without additional heat treatment)
The heat treatment leads to reduction of wear for both versions of Ni coating (with and without BN nanoparticles). The highest mass wear rate ($W_m$) of $4.04 \times 10^{-2}$ mg/m is observed for as deposited Ni coating without BN particles, and the lowest mass wear rate of $1.12 \times 10^{-2}$ mg/m for heat treated Ni coating with BN particles. The wear process is more stable for as deposited coatings, which can be seen from the $R^2$ (R-squared) value. Influence of imbedded BN nanoparticles on wear process stability can be neglected. Generally all R-squared shows acceptable goodness of fit ($R^2 = 1$ is a perfect fit).

Linear wear rate, i.e. the intensity of the coating thickness loss is calculated from mass wear rate by the Equation (3), and the results are shown in Table 4. In order of easier comparison of different coatings, a value of relative wear rate is also introduced (relative to the sample 1, i.e. coating Ni).

Table 4. Linear wear rate and the influence of heat treatment and addition of BN nanoparticles on the reduction of wear rate

<table>
<thead>
<tr>
<th>Sample</th>
<th>Coating</th>
<th>Wear rate, $\mu$m/m</th>
<th>Relative wear rate</th>
<th>Influence of heat treatment, %</th>
<th>Influence of BN nanoparticles, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ni</td>
<td>$1.71 \times 10^{-3}$</td>
<td>1</td>
<td>without particles</td>
<td>as deposited</td>
</tr>
<tr>
<td>2</td>
<td>Ni$^{HT}$</td>
<td>$7.97 \times 10^{-4}$</td>
<td>0.47</td>
<td>with particles</td>
<td>heat treated</td>
</tr>
<tr>
<td>3</td>
<td>Ni-BN</td>
<td>$1.05 \times 10^{-3}$</td>
<td>0.61</td>
<td>$R_{1,2} = 53.5$</td>
<td>$R_{1,3} = 38.7$</td>
</tr>
<tr>
<td>4</td>
<td>Ni-BN$^{HT}$</td>
<td>$4.74 \times 10^{-4}$</td>
<td>0.28</td>
<td>Average: $R = 54\ %$</td>
<td>Average: $R = 40\ %$</td>
</tr>
</tbody>
</table>

Comparative quantitative assessment of the wear rate reduction is done through the reduction index (R), Table 4. Heat treatment shows higher influence on wear reduction than imbedding of BN nanoparticles. With heat treatment an average reduction of wear was 54 %, and with imbedding of BN nanoparticles wear reduction of about 40 % is obtained. Nevertheless, both influences on wear reduction are significant. Obviously the highest wear reduction of 72 % was noticed for heat treated Ni coating with imbedded BN nanoparticles.

**Figure 6:** Comparative values of relative wear resistance and relative hardness of tested coatings

In addition to the wear data, the hardness of each of tested materials was determined (Table 1), as an ancillary mechanical property, to make appropriate correlations. It is well known that hardness of commercially pure metals influences its abrasive wear resistance and that higher hardness implies a higher wear resistance. Khruschev [8] finds out that increase of the wear resistance depends on the way in which the metal is being hardened (alloying, heat treatment or
work-hardening) and that in some cases wear resistance decreases with increase of hardness. The results of other researchers also show that abrasion wear resistance of quenched and tempered steels has a much weaker dependency on bulk hardness [9]. The use of hardness as a parameter for predicting the wear behaviour of materials must be done with caution since, very often they are not in correlation [10].

The relationship between obtained wear values with hardness of tested materials is shown in Figure 6. For better comparison wear is expressed through the relative values of hardness and wear resistance (reciprocating value of the wear rate). Coating Ni was taken as a reference material and relative hardness and wear resistance for this coating are equal to 1. Coating Ni^{HT}, for instance, is harder by approximately 10 % (relative hardness of 1.09), and posses more than twice higher resistance to wear (relative wear resistance of 2.15) than reference coating Ni.

The first feature from Figure 6 is that the hardest coating shows highest wear resistance as well (coating Ni-BN^HT), but from the other coatings it is obvious that relationship between the abrasive wear and hardness values of any kind did not exist.

4. CONCLUSIONS

Four different chemical Ni coatings were investigated on abrasive wear resistance. Influence of heat treatment (heating at 300 °C for 6 hours) and addition of BN nanoparticles (5 vol. % of 10 nm average size) on dry abrasive wear was analysed.

Heat treatment provided less stable wear process, but significantly decreases the abrasive wear rate (by approximately 54%).

Addition (imbedding) of BN nanoparticles also decreased the abrasive wear rate, but the decrease was lesser (by approximately 40%). The smallest wear rate was noticed for heat treated Ni coating with imbedded BN nanoparticles (wear rate decrease was approximately 72%).

Correlation of any kind between hardness and wear resistance of tested coatings could not be established.

5. ACKNOWLEDGEMENTS

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6. REFERENCES


