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Improvement of the Wear Resistance of Ferrous Alloys by Electroless Plating of Nickel

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Abstract: The electroless nickel (Ni) and composite nickel – nanodiamond (Ni+DND) coatings are investigated in this study. The method EFTTOM-NICKEL for electroless nickel plating with nanosized strengthening particles (DND 4-6 nm) is applied for the coating deposition. The coatings are deposited on ferrous alloys samples. The wear resistance of the coatings is performed by friction wear tests under 50-400 MPa loading conditions – in accordance with a Polish Standard PN-83/H-04302. The microstructure observations are made by optic metallographic microscope GX41 OLIMPUS and the microhardness is determined by Vickers Method. Tests for wear resistance, thickness and microhardness measurements of the coatings without heat treatment and heat treatment are performed. The heat treatment regime is investigated with the aim to optimize the thermal process control of the coated samples without excessive tempering of the substrate material. The surface fatigue failure is determined by contact fatigue test with the purpose to establish suitable conditions for production of high performance materials.

Keywords: electroless coating, wear resistance, microhardness, contact fatigue, nanodiamond.

1. Introduction

The market demand for high reliability, effective, with environment protection working machines requires development of new materials and innovative methods for production of high performance parts and details. The involvement of nanotechnology in different fields of the industry open a wide perspective for the following application-related advantages to be expected: improved efficiency, increased component life, which is the expectation on the side of automotive, aircraft and airspace industry. The reduced costs, increased quality of the production makes the European enterprises a serious competitor in these sectors.

There are many studies proving the materials surface properties improvement by incorporation of super hard micro and nanosized particles by electroless metal deposition technique. Electroless method for nickel based composite coating deposition is especially important seeing the expectation to replace the toxic Cr-containing coatings [1]. The characteristics of the electroless plating method ensure the uniform dispersion of the particles within the composite coating. This makes the coating consistent and regenerative in use and offers a practical and economical opportunity to provide the properties of super hard particles to the substrate surface.



Authors in [2] study the effect of co-deposition of PTFE and/or MoS₂ particles on the morphology, wear, and corrosion properties of electroless nickel coating and achieve increase of the wear resistance of the coating by about 30% and a reduction of the average value of friction coefficient to 0.25 from 0.65. About corrosion studies the co-deposition of the PTFE and MoS₂ particles led to reduction in corrosion resistance by 10 and 5 times. Authors also prove the heat treatment influence on the coatings hardness and its maximum is gained at 400°C.

[3] investigate the possibility for reduction of the corrosion and enhancement of the wear resistance of ferrous based bearings used for water lubricated applications by application of electroless nickel coating with addition of silicon nitride (Si₃N₄) which possesses high hardness and chemical inertness. The amorphous to crystalline transition with precipitation of Ni₃P and Ni around 340 °C even when 2.9 wt.% Si₃N₄ is present in the composite is observed by Differential scanning calorimetry (DSC) and X-ray diffraction (XRD). Scanning electron microscopic (SEM) photographs of the cross-section and surface of the coated specimen reveal most desired uniformity at 2.9 wt.% from 5 g/l bath loading of the strengthening particles. Almost similar corrosion rate as that of carbon steel in demineralized water adjusted to pH 10 by addition of LiOH and satisfactory performances in terms of weight loss, pitting levels and coefficient of friction and life time of about 9 years under the actual application environment of coated AISI 52100 ball-bearings and races coated with 4 μm thick ENC are achieved.

In [4] Electroless nickel (EN), EN-silicon carbide, and EN-alumina composite coatings are put under testing by the two-body abrasive wear using a scratch test with a diamond indenter and are also heat treated at temperatures of 100–500° C. The coatings hardness increase with heat treatment temperature from 500 HV100 for the as-deposited condition to 1008 HV100 when fully hardened is determined. Scratch testing show that the as-deposited coating have scratch tracks with a high degree of plasticity, signs of micro ploughing and tensile cracking and is characterized as a ductile failure. On the other hand, the heat-treated coatings show chipping and cracking on the edge of the scratch tracks, failing in a brittle manner. The heat-treated EN-silicon carbide coatings, however, exhibit neither cracking nor chipping, believed to be due to its higher fracture toughness than the other heat-treated coatings, attributable to its lower phosphorus content. The volume of material removed from the silicon carbide scratch track is 1/3 of the volume removed from the steel substrate at a 20 N load, and show the best wear/ scratch resistance of any of the coatings tested.

A sliding wear tests performed at both room temperature and high temperature (100 and 300°C) on silicon carbide/nickel composite coatings deposited industrially on AISI 1020 steel discs by using a proprietary self-catalyzing chemical reduction process (Hardex) and annealed at 400°C in argon show nearly 23 and 11.6 times larger wear rate for the heat treated coatings tested at 100 and 300°C, respectively when compared with the wear resistance of the heat treated coatings tested at 25°C [5].

In the study [6] a prediction model for the wear depth of the electroless Ni-P deposits under lubricated conditions with varying normal load, sliding speed and sliding time using multiple regression analysis and fuzzy logic, which is a very efficient artificial intelligence technique for modeling and monitoring systems is compared with experiments carried out according to Taguchi's L27 orthogonal array of experiments and it is found a good agreement between the two approaches. The authors find that the wear mechanism is mild abrasive in nature.

[7] examine electroless nickel based alloy deposition as an excellent alternative to hard chromium and nickel-boron coating as a result of its superior hardness and outstanding wear resistance in abrasive environments. Therefore, this study reveals the inherent and enhanced mechanical properties that composition and heat treatment have on multi-alloy, and that duplex nickel based coating has on mild steel substrate. The coating is a supersaturated alloy in its as-plated state but became strengthened as a result of controlled moderate annealing by precipitation of nickel phosphide crystallites which effectually enhance its overall mechanical properties within a specified heat treatment temperature range. However, the entire mechanical properties of this coating decrease with excessive heat treatment above 450oC due to grain coarsening. The results here show that the optimum wear resistance of Ni-W-1.5% P and Ni-W-9.5%P coatings correspond to peak hardness at 350oC to 450oC respectively after one hour annealing in argon environment.

The authors in [7, 8] receive a uniform and compact surface morphology in Ni-P/nano-TiO₂ and Ni-P/nano-carbon nanotube composite coatings, ensuring better corrosion resistance than that in the 5083

aluminum alloy substrate and in the electroless Ni-P coating. The investigation of the influence of different concentration of the added particles shows that the corrosion resistance of the nanocomposite coatings is substantially increased to a maximum at 10 g/L nano-TiO₂ and nano-CNT introduction. The superior corrosion resistance exhibits nano-CNT codeposited coating compared to the nano-TiO₂ codeposited coating. [9].

In [10] electroless Ni-P coatings on magnesium composite reinforced with multiwall carbon nanotube under specific coating conditions are studied. In general, it is observed that the surface finish, micro hardness, specific wear rate and friction of the EN-coated layers improve significantly with the addition of nano additives. Nano additives to the EN bath such as Al₂O₃, SiO₂ and ZnO are used.

[11] proves the influence of the presence of the surfactant in the EN bath, its type and concentration on the process of electroless deposition of Ni-P-nano-ZrO₂ composite coatings. The most stable bath is obtained with the addition of dodecyl trimethyl ammonium bromide (DTAB). In the presence of DTAB, the Ni-P-nano-ZrO₂ layers change the surface topography and become smoother. The surfactant can increase the possibility for the particle being embedded changing the composition of obtained coatings and influencing the P/Ni ratio in the coatings produced without and with surfactant.

The purpose of the present study is to improve the ferrous materials surface properties by addition of nanosized diamond particles (DND) with the aim to increase fatigue lives of gears using a novel technology for electroless coating deposition. The electroless coating selected for this investigation is composite nickel-nanodiamond (Ni-DND) coating.

2. Materials and Investigation Methods

2.1. Manufacture of test samples

The samples are manufactured using 17CrNiMo6 steel and a new air hardening steel suitable for gears manufacturing OVATECTM 677L.

The 17CrNiMo6 steel samples are carburised, hardened and tempered and finish ground. The thermal treatment process cycle conditions are: soak for 30 minutes at 930°C; gas carburise for 120 minutes at 930°C and 1.2% C diffuse for 60 minutes at 930°C; fast cool to 830°C and hold for 60 minutes at 0.75% C; direct oil quench (standard speed 22 grade quench oil); temper for 120 minutes at 180°C.

2.2. Coating procedure

The nanocomposite nickel coatings are obtained by electroless nickel plating method EFTTOM-NICKEL. This method is developed at Technical University of Sofia. The ultra dispersed diamond powder (DND) is used as a strengthening material. The particle size is between 4-6 nm. The nanosized diamond powder is produced by a detonation method developed at SRTI-BAS. The technology for nanostructured coating deposition is developed at BAS-SRTI. The electroless nickel coatings (Ni) are composed of one layer. The composite nickel coatings consist of two layers: electroless nickel followed by a second layer of Ni-DND. The coatings are heat treated at the following temperature modes:

1. 200°C for 10 hours
2. 220°C, 9 hours
3. 250°C, 7.5 hours
4. 290°C for 6 hours.

2.3. Test procedure

The microstructure observations are carried out by means of the optical metallographic microscope GX41 OLIMPUS at magnifications x100 before and after coating layering. The samples are treated with 3% HNO₃-C₂H₅OH solution before the examination. The microhardness of the coating is determined by Vickers Method. The used loading is 300gf.

The resistance to wear of samples is estimated by means of the friction test in 3-rollers-cone system (Polish Standard PN-83/H-04302). Testing procedure is carried out under the 4 various loading conditions: 50 MPa, 100 MPa, 200 MPa and 400 MPa. Tests are conducted with drop lubrication at a rate of 30 drops of Lux-10 oil per minute. Toughened 45 grade steel (Polish standard) with a hardness of 28-30HRC is used as a counter piece.

The contact fatigue tests are carried out using the 60mm centre distance disc rig. Two tests are performed on each coating and a summary of the operating conditions is shown in Table 1. The discs are run for 1×10^6 cycles at each of the load stages shown in Table 2 until the coating is removed. It is noted that a Hertzian contact stress of 1200MPa is selected for load stage 1 as the base steel would be expected to perform without concern under such conditions.

All studies are conducted with the presumption that the results will be used in the manufacture of gears.

Table 1. Operating conditions for contact fatigue tests on coated disc samples.

Lubricant:	100cSt mineral oil + 4% Anglamol A99 EP additive
Lubricant temperature:	70°C
Lubricant flow rate:	1.4l/min (into and out of mesh)
Speed:	1100rpm
Slide roll ratio:	0.33
Load cycles:	1×10^6 (per load stage)

Table 2. Loading conditions for contact fatigue tests on coated disc samples

Load stage	Applied load (N)	Hertzian contact stress (MPa)
1	143	1200
2	182	1300
3	227	1400
4	280	1500
5	339	1600
6	407	1700
7	483	1800
8	568	1900

3. Experimental results and analysis

3.1. Metallographic analysis

Micro-hardness measurements are carried out on the discs to determine if the coating process has adversely affected the properties of the substrate material. The micro-hardness profiles measured on each disc are plotted in Figure 1. The micro-hardness profile measured on the uncoated disc in the carburised and finish ground condition is also included for comparison. The Ni - DND coated discs that are thermally processed at temperatures of 200 and 290°C give a reduction in case hardness of approximately 5 to 65Hv relative to the carburised and finish ground reference condition. However, these reductions in case hardness are not unexpected given that the thermal processing is carried out at temperatures of between 20 and 110°C higher than the typical tempering temperature of the steel from which the discs are manufactured.

Despite the observed reductions in case hardness, the values measured on the Ni - DND coated discs that are thermally processed at temperatures of 200 and 290°C would not be deemed unacceptable for carburised gears. In each case, the hardness values measured at a depth of 0.05mm are greater than the minimum case hardness of 660Hv recommended for carburised gears.

The coatings applied show good adhesion to the surface of the substrate material and there is no evidence to suggest that the microstructure of the substrate material has been modified during the coating process. Ni- DND coatings can also be applied without modifying the gear geometry and surface finish present after finish grinding.

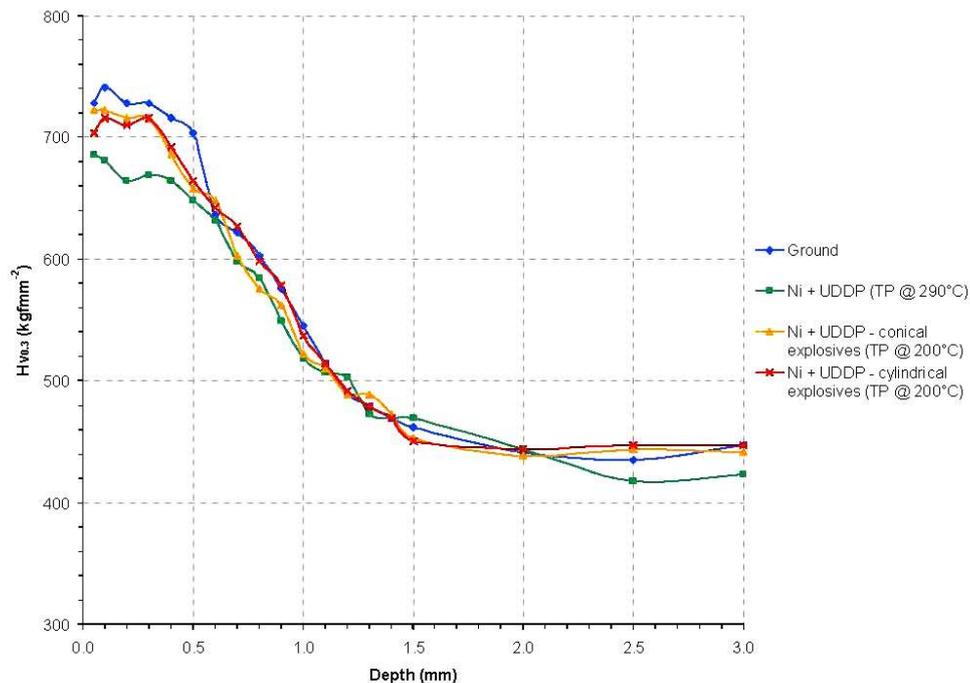


Figure 1. Figure 1: Microhardness profile measured on coated samples

3.2. Metallographic observations

A comparison of the near-surface microstructure present before and after coating is shown in Figure 3. The Ni-DND coating is found to be approximately 8µm in thickness and show good adhesion to the substrate. There was no evidence to suggest that the microstructure of the substrate material is modified during the coating process.

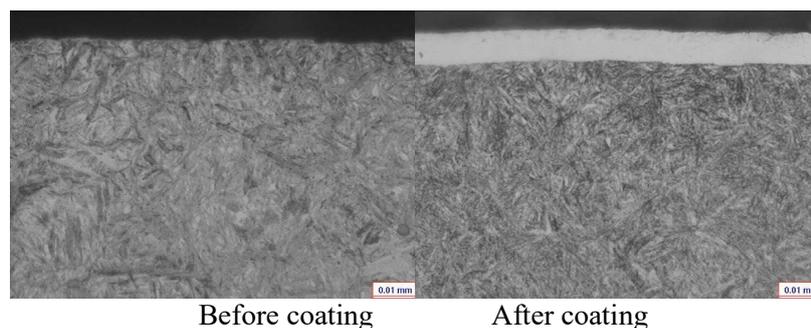


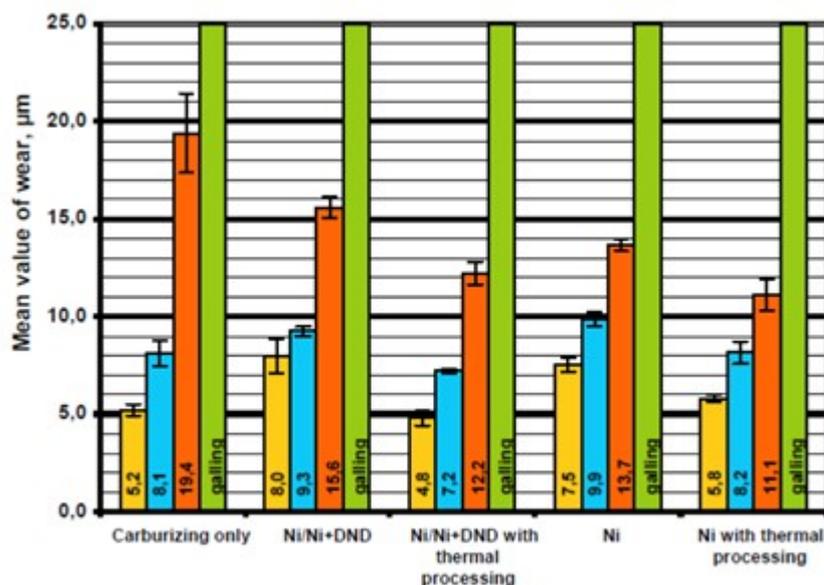
Figure 2. Near-surface microstructure before and after plating

3.3. Resistance to wear test

Friction tests are performed for evaluating the coating behaviour obtained by different technologies. Several unit pressure of loading are applied during the test. Low and medium range of the load are: 50 MPa, 100 MPa and 200 MPa. For high loads the pressure value of 400 MPa is used.

During tests carried out under low and medium range load, it is observed that all of the coating layers tested don't undergo galling or the wear rate during the whole testing cycle don't exceed the critical value of wear (maximum of the measured wear range is 25 μ m).

The baseline of the resistance to wear friction test is the wear resistance of carburized specimen treated without any additional surface processing. The results of wear test (Figure 2) show relatively high resistance to wear under the load of 50 and 100 MPa. Under the load of 100 MPa more intensive wear is observed, while under load of 400 MPa specimens quickly (20 minutes of the test) undergo galling. While the tests are worked out under low and medium loading conditions of unit pressures, resistance to wear of all coated variants are comparable to wear resistance of the carburized only specimens. Among all of the coated specimens, the best wear resistance result is obtained by Ni – DND with next thermal processing coating variant of the surface treatment. Under the high loading conditions all of the chemical method specimens undergo galling. Galling occur after 80 minutes of test of thermally processed Ni -DND coating, while for all other coating variants the galling take place much quicker (50-60minutes of friction test).

**Figure 3.** Wear test of the samples

3.3. Contact fatigue test

The contact fatigue tests are carried out on the coatings showing the best performance during previous test procedure. The results are summarized and the minimum Hertzian contact stresses at which the coatings are seen to be removed during testing are shown in Table 3.

The results described indicate that the contact fatigue performance of the Ni - DND coatings that are thermally processed at 200° C is limited by delaminating of the Ni plate. Therefore, the heat treatment regime is optimized with the aim the Ni-plate adhesion to the substrate to be improved by increasing the thermal processing temperature without causing excessive tempering of the substrate material.

As four different regimes of thermal process of the coatings are investigated - at 200°C, 10 h; 220°C, 9h; 250°C, 7,5h and 290°C, 6h and the coated samples are tested for contact fatigue examination. The work outlined is demonstrated that thermal process at 290°C can give the greatest improvement in contact fatigue performance without adversely affecting the properties of the substrate material.

Table 3. Overall results of contact fatigue tests on coated disc samples

ID	Coating	Thermal processing	Material of Twin Discs	Hertzian contact stress required to remove coating, σ_H [MPa]		
				Test 1	Test 2	Test 3
1	Ni - DND (conical explosives)	200°C, 6 hours	17CrNiMo6	≤ 1200	≤ 1200	-
2	Ni - DND (cylindrical explosives)	200°C, 6 hours	17CrNiMo6	≤ 1200	≤ 1200	-
3	Ni - DND	220°C, 9 hours	17CrNiMo6	1200...1300	1500...1600	-
4	Ni - DND	250°C, 7.5 hours	17CrNiMo6	1500...1600	1400...1500	-
5	Ni - DND	290°C, 6 hours	17CrNiMo6	1700...1800	1200...1300	1200...1300

There are noticeable differences in the results obtained. We suppose they are due to the bed thermal control, which is proved during the next experiments on real details. The contact fatigue tests are performed on three pairs of through hardened and finish ground OvatecTM 677L spur gears coated with Ni - DND and thermal processed for 6 hours at 290°C under controlled conditions. They all show the same result: Ni - DND coating give 14% increase in maximum Hertzian contact stress required to generate failure (Table 4).

Table 4. Contact fatigue test results

Material	Surface condition	Maximum Hertzian contact stress required to generate failure (MPa)	Maximum profile deviation at end of test (μm)
Ovatec TM 677L	Ground	1404	13 μm
	Ni - DND	1601	15 μm

4. Conclusions

Metallographic observations, microhardness measurements, wear and contact fatigue tests of the electroless Ni and Ni-DND coatings plated on 17CrNiMo6 and OvatecTM 677L samples in as-plated condition and after heat treatment show that the temperature at which the Ni - DND coatings are thermally processed can be increased to 290°C without causing excessive tempering of the substrate material. The contact fatigue performance of the Ni - DND coatings can be significantly improved by increasing the temperature at which thermal processing is carried out. Improved control of the thermal processing temperature is required to reduce the variability in the contact fatigue performance of the Ni - DND coatings. 14% increase in maximum Hertzian contact stress required to generate coatings failure is obtained for Ni-DND coating after heat treatment.

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