

Case study

Tribological characterization of electroless NiP coatings lubricated with biolubricants

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Abstract

Electroless nickel (EN) coatings are used in a wide range of applications concerning their excellent mechanical and tribological properties. The incorporation of solid lubricants, such as polytetrafluoroethylene (PTFE), could even improve the properties of the EN coatings. Above all, we can achieve a film with self-lubricating and excellent anti-sticking characteristics. These uses of electroless nickel coatings are widespread in dry contacts. However, it is well known that in the mechanical components there are a large number of applications that require especially low friction, usually not compatible with the use of unlubricated contacts. Moreover, nowadays, there is a general concern with the environmental impact in the use of lubricants. The tendency is to use fluids with small environmental impact, reducing the contamination made by lubricants of mineral origin. Hence, the aim of the present work was to investigate and improve the understanding of the behaviour of EN and EN + PTFE coatings under lubricated contacts using biolubricants.

Tests of coated cylinders were done in lubricated contact against hard AISI 52100 steel on a crossed cylinder sliding tester. The tests were carried out varying widely both the sliding speed and the normal applied load. To allow the comparison of different lubricants, Stribeck curves were used. The influence of the different lubricants under study on the wear amount was investigated by measuring the wear scar on the end of each test. Scanning electron microscopy was used to identify the wear mechanisms.

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1. Introduction

Electroless NiP coatings, known to have excellent properties such as corrosion and wear resistance, hardness, lubricity, uniformity of deposit regardless of geometries, solderability and bondability and nonmagnetic properties [1], are widely used in the mechanical, chemical and electronic industries. An improvement of some of those properties can be achieved by the incorporation of hard particles (SiC, B₄C, Al₂O₃ and diamond) [2,3] and dry lubricants (PTFE, MoS₂ and graphite) [1,3–5], resulting, in the last case, in a film with self-lubricating and excellent anti-sticking characteristics. This fact is reported by the work of several researchers that have investigated the wear behaviour of these coatings, using different wear testers and

mostly in nonlubricated conditions [1–5]. However, in several mechanical applications the use of lubricants is needed because the friction should be very small. In this stage, and to meet these requirements, most modern lubricants are complex formulated products consisting of 70–90% base oils mixed with functional additives to modify the natural properties (i.e. cold stability, oxidation stability, hydrolytic stability, viscosity and viscosity index, corrosion) and fulfil the requirements for the fluid. The base oil can be mineral, vegetable or synthetic. Another aspect that should be considered is that there is an increasing demand for environmentally compatible lubricants, particularly in areas where they can come into contact with water, food or people.

The present work aims to investigate and improve the understanding of the tribological behavior of EN and EN + PTFE coatings under lubricated contacts using biolubricants and the specific wear rate of these materials. The Stribeck curve at variable sliding speeds and loads was obtained.

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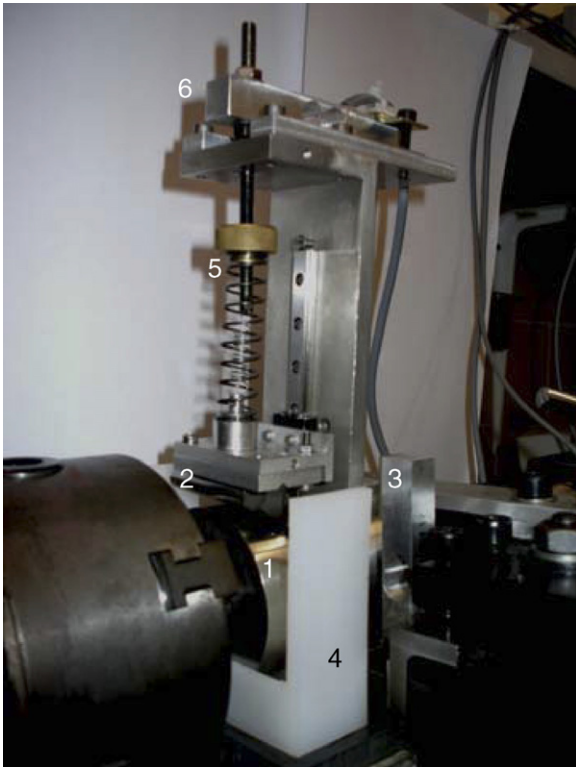


Fig. 1. Sliding tribometer with crossed cylinder contact.

2. Experimental details

Friction and wear were experimentally studied using a sliding tribometer with crossed cylinder contact (Fig. 1). The equipment included a rotating specimen with cylindrical shape (1) and a smaller cylindrical stationary specimen made from 100Cr6 hard steel with 750HV30 (2). The stationary specimen, with a diameter of 10 mm and a surface roughness (R_a) of $0.078 \mu\text{m}$, was supported by a free rotating system, which was equilibrated by a load cell (3) used to measure the friction force. The rotating specimen, with a diameter of 60 mm, contacts in the base with the lubricant bath placed in a container (4). This disk, made from 100Cr6 hard steel with 780HV30, was used as a substrate to deposit the EN coatings with a thickness of approximately $8 \mu\text{m}$ and a surface roughness (R_a) of $0.129 \mu\text{m}$ for NiP and $0.540 \mu\text{m}$ for the NiP + PTFE. The normal load was applied by

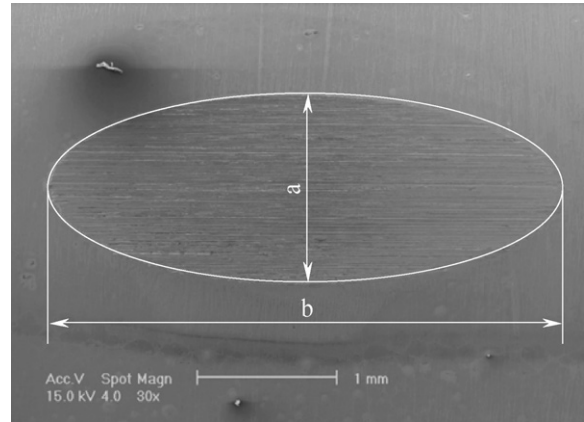


Fig. 2. Typical wear scar of the stationary specimen.

a spindle/spring system (5) and was measured by a second load cell (6).

With this tribometer, two set of tests were made, short and long duration tests. For the first kind of tests the rotation speed was set to 32, 159, 318 and 461 rpm, thus, the sliding speed was varied between 0.1 and 1.45 m/s. These tribological tests were carried out for a short period, enough to obtain a steady value of the friction; usually the friction data acquisition occurred between 150 and 1500 rotations after beginning the test. The study was done using lubricated conditions, and the applied normal load varied from 1 to 30 N, which corresponds to a Hertzian pressure ranging from 277 to 862 MPa [6].

In order to determine the specific wear rates and the mechanism of material removal, the NiP coating was tested considering longer tests. For this purpose, all lubricants, except water, were tested considering: a normal load of 5 N, a rotation speed of 159 rpm, which corresponds to a sliding speed of 0.5 m/s, and test duration of 60 min. Five lubricants were used with the properties indicated in Table 1. Two commercial biolubricants, 2 and 3, were tested. Besides that, mineral based oil was tested as reference, and two low viscosity fluids were also tested, namely an emulsion, 4, and water.

The friction force values were acquired periodically along the test. All tests were carried out at room temperature ($25 \pm 2 \text{ }^\circ\text{C}$).

At the end of the test, the stationary specimen shows an elliptical-shaped wear scar (Fig. 2). The volume of this wear scar can be calculated assuming an imposed wear shape and

Table 1
Lubricants properties

Lubricants	Characteristics	Viscosity (cP) at 25 °C	Viscosity (mm^2/s)	
			40 °C	100 °C
(1) Gearbox oil	Mineral-based multi-grade lubricant oil	302.14	132	13.9
(2) Chain Way Bio	Rape oil-based chain saw oil with extremely good adhesion and good temperature properties	105.78	64	14.6
(3) Safe Lube Gear Lube	SAFE LUBE™ Gear Lube combines the advantages of a blended vegetable oil base with an extreme pressure performance package compounded from botanical sources	248.54	a	a
(4) Emulsion	Emulsion of 20 vol.% of vegetable base oil in water	4.18	a	a
(5) Water		0.89	a	a

^a Not available.

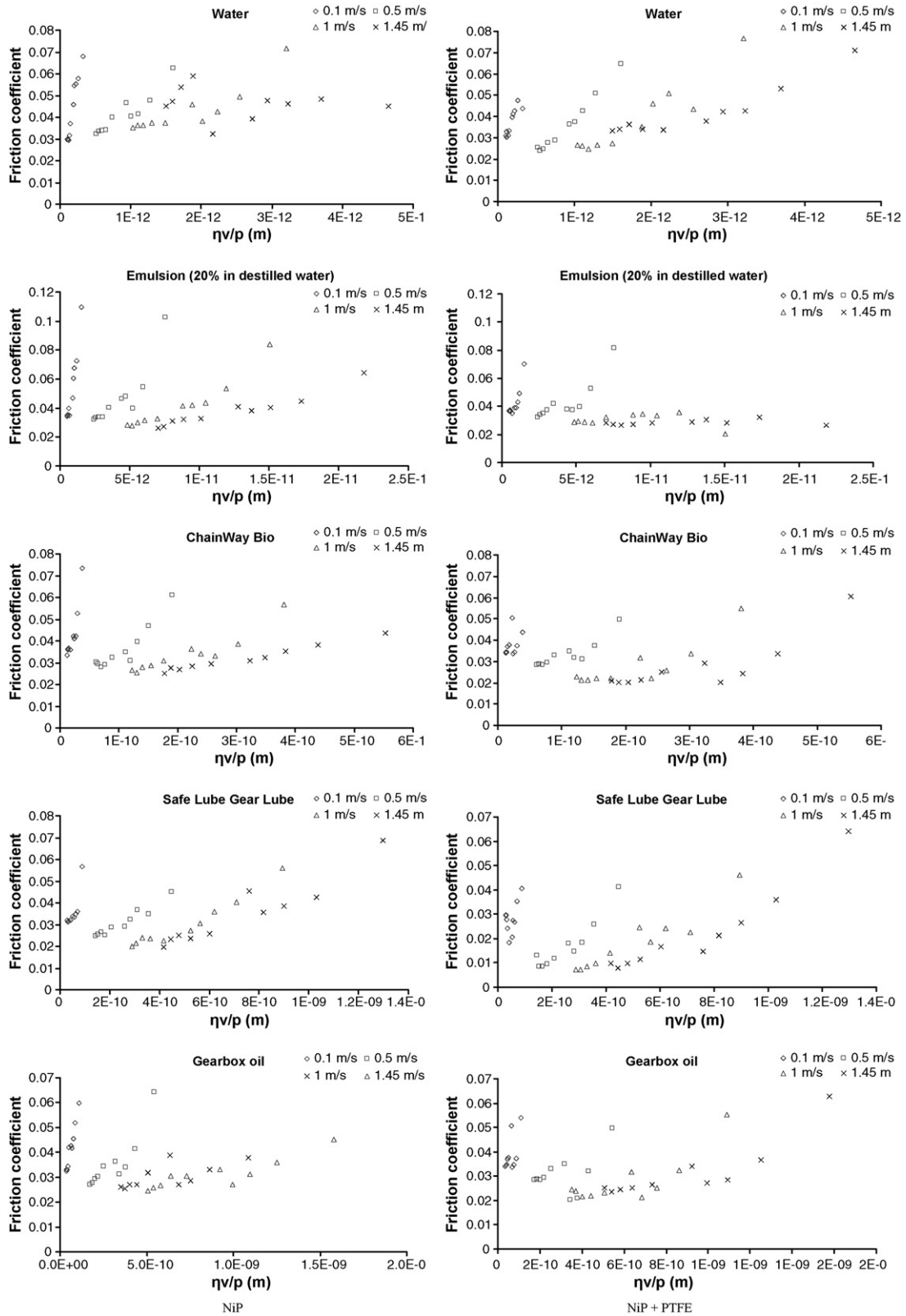


Fig. 3. Stribeck curves for all the lubricants with sliding speeds selected in this study.

using the approximate expression (1) derived by Ramalho [7]. This simple equation is very accurate, with errors smaller than 0.2% [7].

$$V = \frac{\pi}{2} h^2 \sqrt{d_1 d_2} \quad (1)$$

where d_1 is the diameter of the stationary specimen, d_2 the diameter of the rotating specimen and h is the depth of the scar.

Each scar is measured by taking the dimensions of the larger, b , and the smaller, a , axis of the elliptical wear surface (Fig. 2). This calculation method is well explained elsewhere [8]. On the rotating specimen, the wear produces a circumferential track. To measure this wear volume, were acquired 12 profiles of roughness with a laser Rodenstock RM600 3D equipment. For each one of the profiles, the corresponding area to the consumed valley was determined. Finally, with this set of values, the average value was determined and then multiplied by the nominal perimeter of the rotating disk, in order to obtain the corresponding wear volume.

The specific wear rate values were calculated, for the stationary and rotating specimens, by the equation $k = V/(SN)$, where V is the wear volume in mm^3 , S the total sliding distance in m and N is the normal load in N.

Under lubricated conditions, a Stribeck curve is often used to indicate lubrication mechanisms [9–11]. In the Stribeck curve, the friction coefficient is plotted as a function of the Hersey parameter (H), which is defined as the product of the dynamic viscosity η of the oil in Pa s and the sliding speed v at the contact in m/s, divided by the pressure p in the contact zone in Pa, i.e. $H = \eta v / p$.

The wear scars were examined with a Philips XL30 TMP scanning electron microscope (SEM).

3. Results and discussion

Fig. 3a and b shows the Stribeck curves, respectively, for NiP and NiP + PTFE electroless coatings, tested for the loads and sliding speeds selected in this study. When tests were done with small values of sliding velocity and for small Hersey parameter values, which correspond to higher loads, the friction coefficient for the NiP + PTFE coatings is generally smaller than that obtained in the NiP coatings. This fact suggests that a mixed lubrication mode occurred, i.e. the thickness of the lubricant film is insufficient to effectively separate the sliding surfaces, and so, metallic contact will occur at the asperities of the cylinders. In these conditions, the self-lubrication and excellent anti-sticking properties of PTFE particles disperse in the NiP coating act and minimize the friction when the sliding surfaces contact. By other hand, the presence of PTFE particles in the NiP coating create like a pattern and increase the geometric perturbation on the top of the surface (Fig. 4) leading to a high surface roughness. This surface pattern can lead to a local turbulent regime in the lubricant film [12,13], decreasing the friction even for low Hersey parameter values. On the other side, when tests were done with higher values of sliding speed, and for small values of contact pressure, the lubrication occurs in hydrodynamic regime. The increase in the sliding velocity leads to smaller values of the

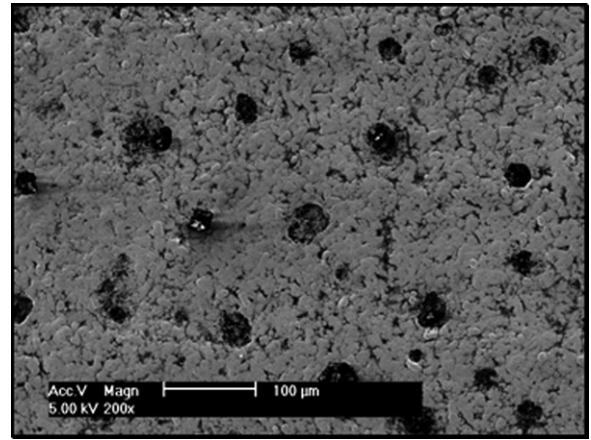


Fig. 4. The scanning electron micrographs of EN + PTFE composite coatings.

friction coefficient, i.e. the Stribeck curve goes down. This fact is more evident for poor lubricants, with small values of viscosity, because require highest speed to reach a hydrodynamic regime.

Fig. 5 represents the Stribeck curve for all the lubricants and for a sliding speed of 0.5 m/s. Similar graphs were obtained for the other tested values of the sliding speeds. The increase in viscosity of the lubricant moves the curves to the right because of the raise induced by the viscosity on the Hersey parameter. In a general way, the friction coefficient has a slightly decreasing with the increase of the viscosity.

The long duration tests reveal that the wear mechanism of the specimens was a mild abrasive wear with typical parallel wear scars along the direction of the moving body (Table 2). The mineral oil was the best lubricant tested with the smallest specific wear rate obtained for the fixed cylinder. For that lubricant, the wear of the rotating disk could not be measured because it was too small. However, a pronounced scratch that crosses the whole length of the specimen can be observed on the side of the wear track. Probably this scratch was due to the action of an occasional wear particle that became entrapped between the sliding surfaces.

The biolubricants perform similarly between them. However, while the lubricant 2 reveals the lower specific wear rate of the rotating specimen, the lubricant 3 has inferior wear on the fixed cylinder.

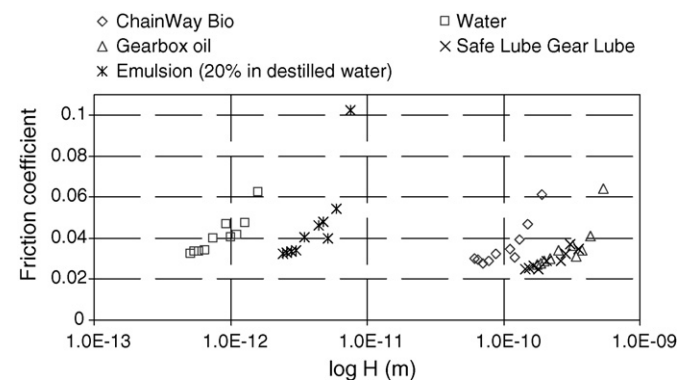
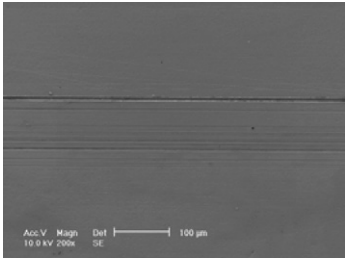
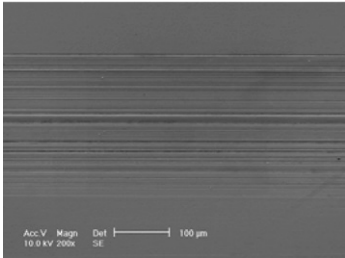
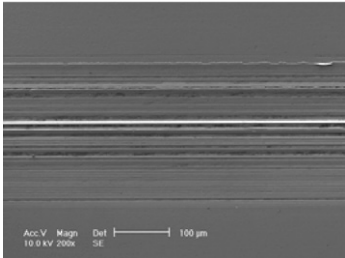
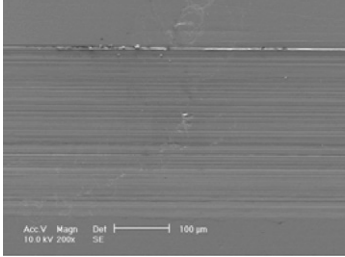


Fig. 5. Stribeck curve for the NiP coating tested with a sliding speed of 0.5 m/s.

Table 2
Average values of the wear rates obtained in the NiP disk for long wear tests

Lubricants	Specific wear rate (mm ³ /N m)		SEM image of wear surfaces
	Rotating disk	Fixed cylinder	
1	– ^a	7.59E–09	
2	5.79E–06	1.25E–07	
3	7.37E–06	9.54E–08	
4	9.75E–06	2.14E–08	

^a This value could not be measured because the wear was very small.

4. Concluding remarks

The friction and wear of NiP and NiP + PTFE electroless coatings were investigated under lubricated conditions, particularly with biolubricants.

The addition of PTFE particles to the NiP coating is helpful in boundary lubrication conditions. The effect of the PTFE derives from its intrinsic low friction coefficient against steel, and probably from the beneficial effect of the surface pattern created by the effect of the PTFE particles on the coating surface.

In boundary lubrication conditions, the wear of the NiP coating occurs by mild abrasion.

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