Electrodeposited Ni-PTFE Dry Lubricant Coating

By M. Pushpavanam, N. Arivalagan, N. Srinivasan, P. Santhakumar and S. Suresh

The development of composite coating technology has progressed such that applications can now be found in general consumer products and, more applications are on the horizon. Using this technique, metallic coatings containing solid particles, such as carbides, oxides and borides, have been developed for improved wear resistance. A different approach to improve wear characteristics is based on the reduction of the coefficient of friction between moving surfaces by the introduction of a dry lubricant in the metal matrix. Polymers, especially PTFE, with their non-stick properties, are also used for dry lubrication. This paper reports the properties of nickel-PTFE composites, plus the description of a device fabricated to measure the coefficient of friction of the deposits.

Solid lubricants can generally be defined as materials that provide lubrication to two relatively moving surfaces, under dry conditions, to reduce friction and wear.[1] They find use where grease and oil lubricants cannot be used. Although solid lubricants are used for applications involving high temperatures,[2] extremely low temperatures, or high vacuum, they can as well be used advantageously under moderate conditions, such as rolling or sliding contacts and bearings where oil or grease is adequate.

There are several advantages in using solid lubricants. The need for periodic lubrication during service life is entirely eliminated; also, the absence of waste or spent lubricant eliminates pollution problems connected with vaporization. Their greater stability permits use in various environments, including food processing where material cleanliness is a major concern. Several kinds of solid lubricants, such as graphite,[3] molybdenum disulfide,[4] boron nitride,[5] mica, talc[6] and polytetrafluoroethylene have been used in industry, depending on the end use of the products.

PTFE retains its mechanical properties over a wider temperature range than other particles. It has an extremely high-impact strength and coefficient of friction (PTFE sliding on itself has a coefficient of friction as low as 0.05, which is the lowest known value for any solid.)[1] It is a result of low adhesion between the molecules, which can roll or slide over each other. The low surface energy of these materials makes it difficult to apply them as adherent coatings on metal surfaces, however. This can be avoided by codepositing them with a metal.

Composite materials with good anti-friction properties are produced by codeposition of solid lubricants in a metal matrix.[2] Electrodeposition has an edge over other available techniques in producing uniform coatings on intricately shaped objects without involving high-temperature treatments or costly equipment. This paper describes the design and fabrication of a device[3] for measuring the coefficient of friction and other properties of Ni-PTFE composites.

Experimental Procedure

Design and Fabrication

The measuring device consists of the following parts (Figs. 1–3): 1. shaft, 2. rotating disc, 3. inner and outer cylinders, 4. compression spring, 5. leaf spring, and 6. other lock-nut arrangements. In this device, the shaft (1), supported by two ball bearings (20), was driven by a motor (27), that controlled its speed. A rotating disc (6) was mounted at the end of the shaft and held in position by a suitable cap. The rotating disc, made of tool steel, should have a perfectly plane and smooth surface. Accordingly, it was ground and lapped to a surface roughness of 0.9 µm. The disc adjusted itself within the clearance inside the cap such that the surface of the disc and the test specimen (5) were parallel.

The test specimen was a steel disc of 3 cm dia. and 0.5 cm thickness, which was surface-ground to obtain a leveled and smooth surface. The disc with the Ni-PTFE composite on one side, facing the rotating disc, was mounted on the front face of the outer cylinder (4), by jaws (12), and wing nut (22). This outer cylinder, guided by a parallel key (11), could move over the inner cylinder (3) coaxially. The inner cylinder was attached to a stationary shaft (2), through two ball bearings, in such a way that the assembly of the two cylinders was floating.

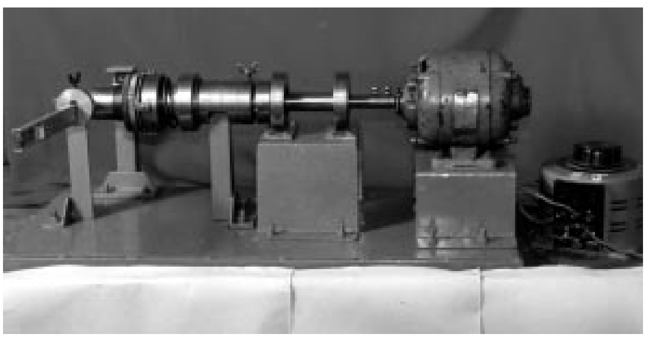

Fig. 1—Device for measuring coefficient of friction; front view.

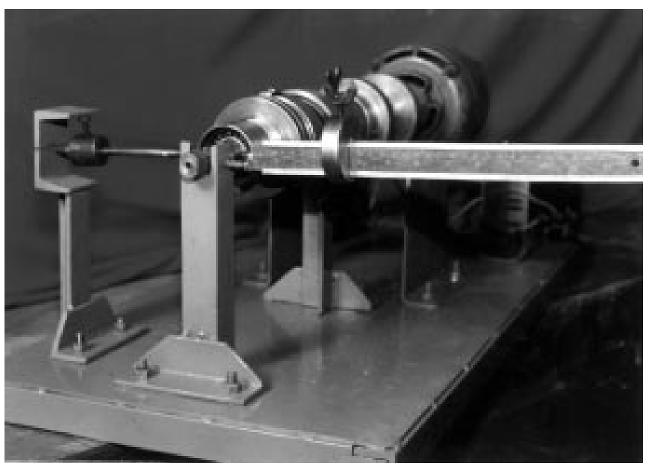

Fig. 2—Device for measuring coefficient of friction; side view.

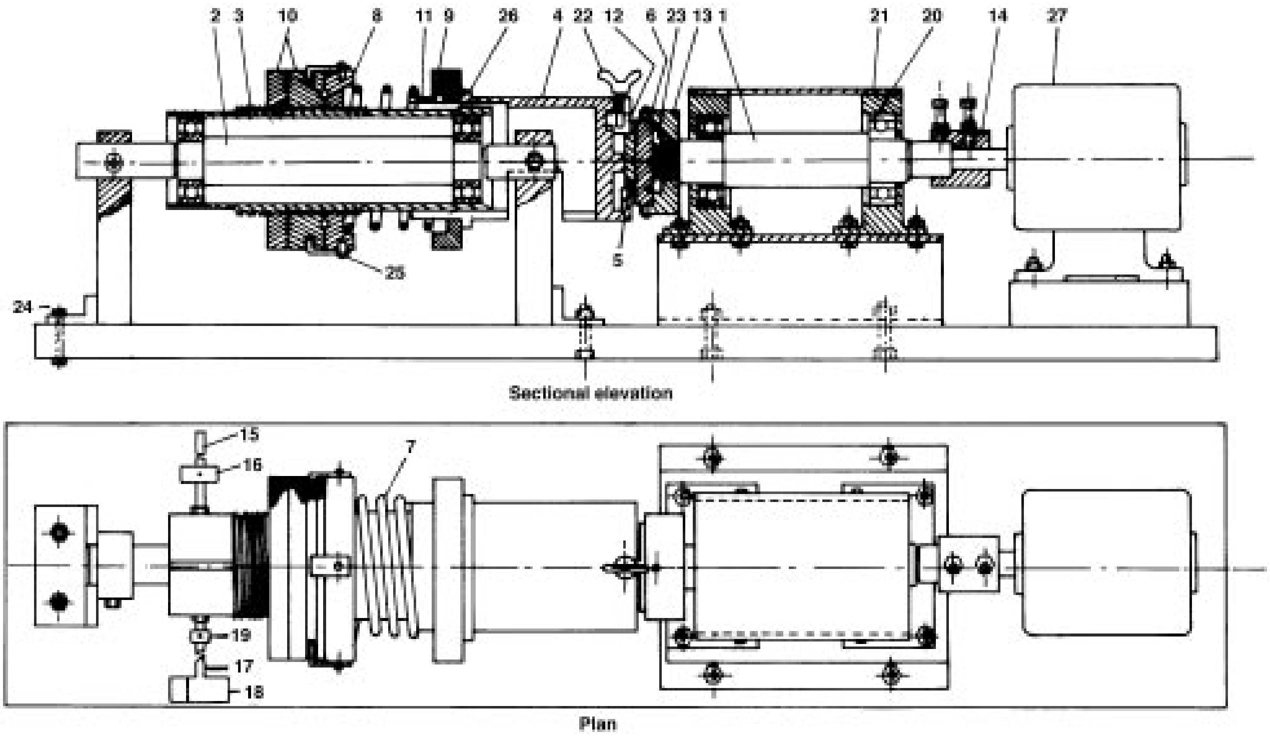

Fig. 3—Schematic diagram of design used for measuring device: 1 & 2, shafts; 3, inner cylinder; 4, outer cylinder; 5, test specimen; 6, rotating disc; 7, compression spring; 8, sliding cap; 9, cap; 10, nut and lock nut; 11, key; 12, jaw; 13, standard specimen; 14, sleeve coupling; 15, lever; 16, dead weight; 17, leaf spring; 18, stop; l9, balancing weight; 20, bearing; 21, bearing sleeve; 22, wing nut; 23, check nut; 24, nut and bolt; 25, screws; 26, collar; 27, motor.

The compression spring (7) was used to load the specimen. While one end of the spring rested on the cap (9), mounted, the other end rested on another cap (8) that moved over the inner cylinder. By rotating the nut over the inner cylinder, the spring was compressed, pushing the outer cylinder forward, toward the rotating disc, which loaded the bearing.

When the disc rotates at a particular speed, because of the friction developed at the interface between the disc and the coated specimen, the latter rotates, causing rotation of the assembly of cylinders through an angle proportional to the frictional torque on the composite. This causes a deflection in the leaf spring (17). The rotation of the cylinders was arrested by the step arrangement (18) in the leaf spring. The frictional torque was measured by applying a counter torque on the cylinder by using the lever (15) and dead weights (16), with the reference marking on the stop. The frictional torque (T) was calculated using the formula

$$T = W x L$$

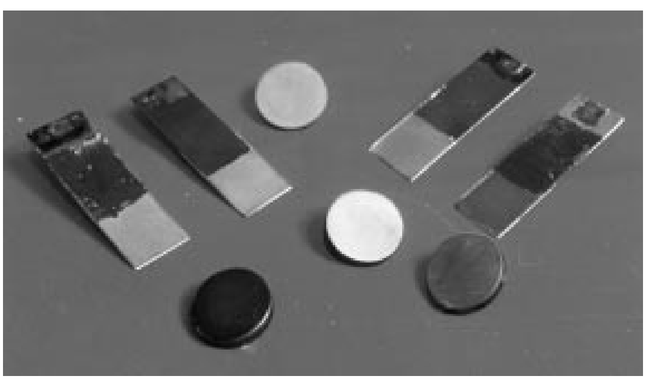

Fig. 4—Samples of mild steel panels and discs used for tests.

where T is frictional torque in kg-cm, W is dead weight in kg, and L is position of dead weight from the cylinder in cm. The coefficient of friction was calculated using the equation

$$U = \frac{3T}{FD}$$

where u is the coefficient of friction, T is the frictional torque in kg-cm, F is the applied load in kg, and D is the diameter of the test specimen in cm.

Production of Composites

A conventional Watts nickel solution of the following composition was used:

$NiSO_4 \cdot 6H_2O$	300 g/L
$NiCl_2 \cdot 6H_2O$	30 g/L
H_3BO_3	40 g/L

The solution was given high pH treatment and carbon treatment, then pre-electrolyzed to remove organic and inorganic impurities. The solution was prepared as a concentrate and diluted suitably after addition of a commercially available PTFE suspension (45 percent w/v and 0.2 to 0.5 µm size). Different levels of solution pH were made for the experiments.

A plate pumping device was used to keep the particles in suspension. Mild steel cathodes, 7.5 x 2.5 cm, and mild steel discs, 3 cm dia x 0.2 cm width, were used for estimation/property evaluation and determination of coefficient of friction, respectively (Fig. 4). The steel panels were mechanically polished and the discs were surface-ground and lapped to a bright finish before the experiments. These specimens were degreased, electrocleaned and acid-dipped before starting the experiments.

Table 1
Effect of Plating Variables on Volume Percent PTFE Incorporation

Trial No.	PTFE Conc. g/L	pH	Current Density A/dm²	Temp. °C	Electrode Position	Volume %
1.	25	4	3.5	55	CECD	8.6
2.	50	4	3.5	55	CECD	11.2
3.	50	4	3.5	55	SCD	20.0
4.	100	4	3.5	55	CECD	15.6
5.	100	4	3.5	55	SCD	25.0
6.	150	4	3.5	55	CECD	17.6
7.	150	4	3.5	55	SCD	30.0
8.	150	4	3.5	35	CECD	5.7
9.	150	4	3.5	45	CECD	10.6
10.	150	4	4.5	55	CECD	18.2
11.	150	4	2.5	55	CECD	14.5
12.	150	3	3.5	55	CECD	16.2
13.	150	5	3.5	55	CECD	18.2

Table 2
Effect of Plating Variables on Volume Percent PTFE Incorporation

Trial No.	PTFE Conc. g/L	pH	Current Density A/dm²	Temp. °C	Electrode Position	Deposition Rate μm/hr
1.	25	4	3.5	55	CECD	41.34
2.	50	4	3.5	55	CECD	41.00
3.	50	4	3.5	55	SCD	37.21
4.	100	4	3.5	55	CECD	40.52
5.	100	4	3.5	55	SCD	36.39
6.	150	4	3.5	55	CECD	39.69
7.	150	4	3.5	55	SCD	35.15
8.	150	4	3.5	35	CECD	36.6
9.	150	4	3.5	45	CECD	38.9
10.	150	4	4.5	55	CECD	41.3
11.	150	4	2.5	55	CECD	38.0
12.	150	3	3.5	55	CECD	35.6
13.	150	5	3.5	55	CECD	39.52

Both the conventional electrodeposition technique (CECD)[7-12] and the sedimentation codeposition technique (SCD)[13-15] were tried, using cold-rolled nickel anodes. Deposition was conducted by varying the operating conditions, such as pH, current density, temperature and PTFE concentration. The stroke length and rate were maintained at 4.6 cm and 100 to 200/min, respectively.

Deposits on steel panels were stripped and analyzed for nickel to calculate the percentage of PTFE incorporation. Hardness was determined by the Knoop indentation method at a load of 20 g. The deposits were heat treated at 250 and 400 °C in a reducing atmosphere for one hr to examine the heat compatibility of the deposits. Structural examination was made via scanning electron microscope.

Results and Discussion

PTFE differs from many second-phase particles that have been included in the nickel deposit because of its hydrophobic property. This necessitated the use of commercially available PTFE suspensions unlike the other cases, where the inert phase is added in powder form. This hydrophobicity was distinctly seen from the non-wetting nature of the deposits produced. Because of this property, these electrodes find use in many organic syntheses. The coatings became increasingly hydrophobic, soft, smooth and satin-like to the touch, with an increase in the codeposition percentage. The volume percentage of PTFE, in excess of 35 percent in the deposit, affected the adhesion of the coatings. At higher incorporation densities, the deposits appeared dull compared to the semi-bright appearance of the pure nickel deposits. Table 1 shows the effect of operating conditions on the volume percent of PTFE incorporation in the deposit. It is quite clear that incorporation is greater with the SCD technique than with the CECD technique.

Figure 5 and Table 2 show the cathode current efficiency and the corresponding rate of deposition. It was observed that the latter was less in the SCD technique than in the CECD and that it decreased with increasing concentration of the dispersoid in the solution. This can be understood as caused by increased blocking of the deposition sites by the PTFE particles. The rate of deposition increased with increase in temperature and current density, but showed a slight decrease

Table 3
Effect of Plating Variables on Coefficient of Friction

Trial No.	PTFE in Deposit Volume %	Electrode Position	Coeff. of Friction *u*
1.	0.0	CECD	0.86
2.	8.6	CECD	0.58
3.	11.2	CECD	0.53
4.	15.6	CECD	0.50
5.	17.6	CECD	0.48
6.	20.0	SCD	0.39
7.	25.0	SCD	0.20
8.	30.0	SCD	0.17
9.	5.7	CECD	0.56
10.	10.6	CECD	0.50
11.	16.2	CECD	0.60
12.	18.2	CECD	0.40
13.	18.2	CECD	0.40
14.	14.5	CECD	0.5

Table 4
Effect of Load on Coefficient of Friction Speed 350 rpm

Load kg	Coefficient of Friction u
0.75	0.60
1.125	0.582
1.50	0.55
2.25	0.53
2.62	0.50
3.0	0.45

Table 5
Effect of Rotational Speed on Coefficient of Friction Load 2.25 kg

Speed rpm	Coefficient of Friction u
350	0.53
400	0.54
500	0.55
625	0.54

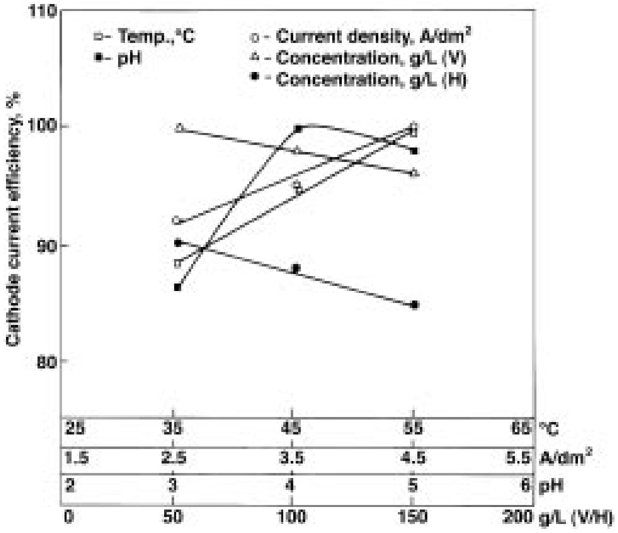

Fig. 5—Effect of plating variables on cathode efficiency: PTFE, 150 g/L; pH 4.0; CECD technique unless otherwise stated; V = CECD technique; H = SCD technique.

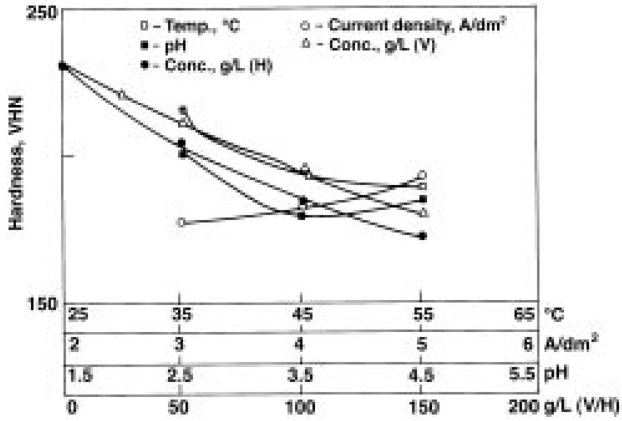

Fig. 6—Effect of plating variables on hardness of deposits. Conditions as in Fig. 5. SEM micrographs of Ni-PTFE composites.

with increase in pH, probably a result of high viscosity of the solution and consequent impeded flow of the particles. It has been reported that the hardness of Ni-graphite (28.5 percent) is 150 to 180 DPN; Ni-MoS_2 (19 percent) is 170 to 180 DPN; and Ni-WS_2 (15 percent)is 150 to 160 DPN.[4] Figure 6 shows that the hardness of the Ni-PTFE composites is also of the same order. The PTFE particles distributed uniformly throughout the matrix at high incorporation densities made the measurements difficult.

Table 3 shows the values of the coefficient of friction obtained at different operating conditions. With increasing percentage incorporation, the coefficient of friction decreased considerably and was minimum (0.17) at 30 percent v/v obtained by the SCD technique. With CECD, the minimum value obtained was 0.4. When frictional contact occurs, the top layer of the particles is smeared over the surface, while the other particles, constrained by the matrix, are slowly released as the matrix erodes. The matrix consequently acts as the reservoir of the polymeric lubricant. Comparison of this value with that obtained for pure nickel made it clear that Ni-PTFE composites are far superior as self-lubricating coatings. Tables 4 and 5 show the effect of varying the load on the specimen and the shaft speed on the coefficient of friction of the composites. It was observed that the coefficient of friction decreased with increasing load and remained almost constant with increase in speed.

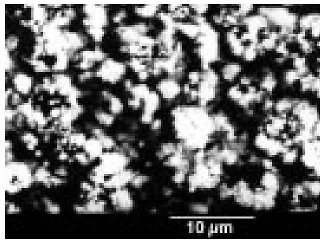

Fig. 7—Ni-PTFE 25% (SCD).

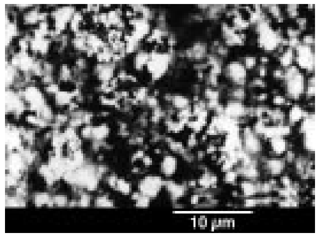

Fig. 8—Ni-PTFE 30% (SCD).

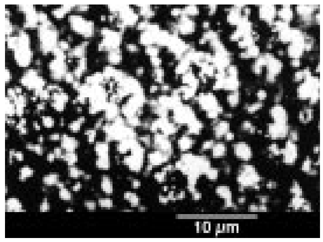

Fig. 9—Ni-PTFE 15.6% (CECD).

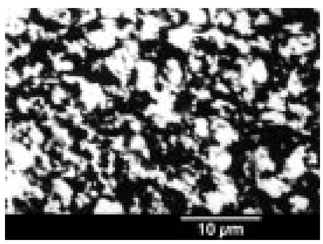

Fig. 10—Ni-PTFE 15.6%, heat treated at 250 °C.

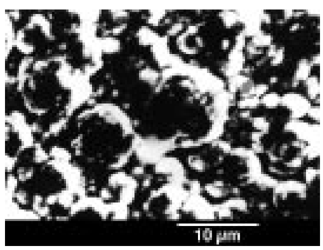

Fig. 11—Ni-PTFE 15.6%, heat treated at 400 °C.

SEM micrographs of the Ni-PTFE composites given in Figs. 7–9 indicate a gradual increase in the percentage incorporation of PTFE particles with increase in concentration in the electrolyte. The particles are uniformly distributed because of the plate-pumping system adopted. The SCD technique enabled more PTFE incorporation than the CECD technique. The structure of the Ni-PTFE composites showed refinement on heat treatment at 250 °C for one hr (Fig. 10), as evident from the smooth, compact and satin nature of the coating, caused by easy flow of the PTFE particles, which solidified on cooling (u = 0.20). Above 400 °C, the particles disintegrated easily, as indicated in Fig. 11 and from the black appearance of the coating.

Summary

The coefficient of friction, as determined with the new device, offers a relative idea of the frictional characteristics of Ni-PTFE composites under different conditions of processing. Ni-PTFE electrocomposites produced by the SCD technique have a very low coefficient of friction. The deposit quality can be improved by heat treatment at 250 °C.

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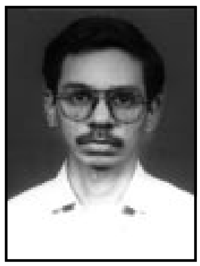
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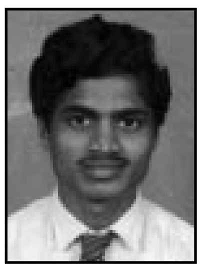
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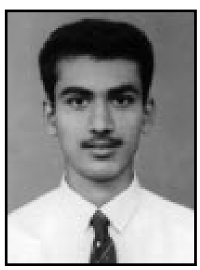
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