Evaluation of Tribological and Mechanical Properties of Carbon Steel with Fluoroligomeric Film at Piezoelectric Actuator Contact

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Abstract: The tribological investigations of carbon steel surfaces modified with fluoroligomeric materials were performed. Compared with the uncoated steel, the fluoroligomeric coated surface showed higher plasticity of the modified surface in the scratch tests where the fluoroligomeric film was not destroyed by higher loading. This could be explained by the lower micro hardness of the steel after processing by fluoroligomer and the influence of the adsorbic Rebinder effect. This study includes original results of the loading influence on the efficiency of the piezoelectric actuator and the wear value of the frictional element, which is important explaining the greater longevity of the piezoelectric actuator. Fluoroligomer treatment of the surface considerably improved the performance of the piezoelectric actuator. Using the reference steel roller in the piezoelectric actuator under minimum loads was characterized by a decrease in rotor speed with increasing the pressing of the frictional element. When the rotor was coated by fluoroligomer, the speed remained stable when increasing the pressing force. Using the reference steel roller, the rotation after short-term overloads was not restored, and rollers with fluoroligomeric surface started to rotate soon as the short-term overload was removed. The higher efficiency of piezoelectric actuators with a fluoroligomeric layer on the roller is related to almost two times lower wear of a friction element operating with coated rollers as compared to reference rollers.

Keywords: fluoroligomeric film; carbon steel; friction force; indentation; scratch test; piezo actuator

1. Introduction

In most engineering problems, the selection of a polymeric material for tribological application is based primarily on the bulk mechanical properties. However, it has been determined that there is not necessarily a correlation between good bulk properties and adequate surface properties. In such precise positioning equipment as piezoelectric actuators (PEAs) the contact surface and its ability to operate efficiently for a possible time period are key factors.

The operation of PEAs involve two phases of conversion of electro-mechanical energy: (1) conversion of electricity to mechanical energy (based on excitation of high-frequency mechanical oscillations of the vibration element performed by piezoelectric transducer); (2) conversion of oscillations to rotation or linear motion using the friction force by pressing the vibrating oscillator with a second element [1].

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The characteristics of PEA are influenced by the frictional element properties: rotor, stator, and contact zone layers [1]. In all applications, the operation of friction pairs depends on geometrical and mechanical features (hardness, surface roughness and fragility, elasticity), working regimes (motion speed, loading, lubrication or its absence, etc.) Contact surface (roughness, hardness, contact area), tangential friction force, longitudinal vibration velocity, elastic modulus, and hardness of tribomaterials [2]. The contact layer is responsible...
for the formulation of stick zones, the amount of friction losses, wear resistance, and the overall efficiency, which could be influenced by the selection of correct materials of friction pairing [1].

Most investigations on possible friction materials were performed with special hard materials coatings, such as carbide (WC-Co8, TiC15-Co6) [3], ceramic (YSZ and Al2O3) [4], or silver coatings [5]. However, the efficiency of such kinds of friction pairs could be improved by comparably cheap and efficient surface processing by polymer materials. An investigation of PEA efficiency after the processing of contact surface with fluoroligomeric material (FOM) was conducted. Applying of such a layer stabilized the rotation and continuous operation of the piezoeutator and reduces the damage of the friction surface. In addition, the steel surface with a fluoroligomeric layer had a 20% higher elasticity compared to the non-coated surface [6].

Different kinds of fluorinated polymers are used for surface modification of engineering applications [7–9]. Thin polymeric films have been obtained by surface treatment with antiadhesive fluoroligomeric polymeric material Foleox. The formation and tribological properties of fluoroligomeric compounds on the surface of metal has been investigated as an efficient method for reducing friction losses and increasing the longevity of friction pairs [10–13]. This film of fluorine compound with oxygen, carbon, and hydrogen reduces the friction in sealing friction pairs by up to 1.2 times and wear up to 2 times [12].

Adhesion, roughness, and microhardness of such coatings are still poorly investigated. When a steel surface covered with a layer of FOM coating material slides against a steel surface, material transfer to the steel counter-body takes place. The formation of transfer films in the sliding contact of FOM coating against a steel slider is more complex. Different processes are taking place, including wear fragments adhering to both surfaces; plastic deformation and strain hardening of the transferred layers and patches, oxidation of layers, and removal of the layers and patches by fracture [14].

The wear resistance investigations of the surfaces using scratch tests show different decay mechanisms for the surfaces modified with fluoroligomeric compounds [14]. The worn surface after the scratch tests without fluoroligomeric coats (Figure 1a) clearly shows flaking and delaminating of the surface. In the presence of FOM, the wear particles, which are attached to the surface, are very thin. Figure 1b shows that with the presence of the fluoroligomeric layer, plastic and elastic deformation is takes place, but adhesion and mechanical damage is absent.

Fluoroligomeric film has certain properties: it is porous, has good adhesion to bulk body surfaces, changes the rheology of two-body contact, reducing the shearing forces,
facilitates sliding with a lower friction coefficient, and decreases the surface damage of plastic deformation [6].

When there is subsurface flow, external action takes place, and the Rebinder effect occurs [15]. The FOM penetrates into microcracks of the bulk body surface and maintains the balance between the formation and rewelding of these microcracks. In this way, the metallic bulk body beneath the surface becomes weaker, resulting in “hardness reduction”. If the strains in the subsurface are lower, microcracks do not form, and the Rebinder effect does not take place [15].

The aim of this study is to investigate carbon steel surfaces with fluoroligomeric coating in operation of piezoelectric actuators and evaluate the tribological and mechanical properties.

2. Experimental Procedure

The special testing device designed by the Department of Mechanical, Energy and Biotechnology Engineering of Vytautas Magnus University was used for investigation of the tribological characteristics of PEA (Figure 2). The following friction pair parameters can be investigated by this device: loading torque, wear, and rotating speed, as well as the dependence between some of these parameters.

![Figure 2](image)

**Figure 2.** Testing of piezoelectric friction pairs: (a) principal scheme of ultrasonic piezoactuator test equipment (1—Stator (holder of piezoelectric element); 2—Piezoelectric element; 3—Pneumatic cylinder with load sensor; 4—Air bearing; 5—Friction material element (counter-body); 6—Rotor (roller with special contact surface); 7—revolution counter; 8—Torque sensor with non-contact break; 9—Frequency generator; 10—Voltage amplifier; PC—Personal computer) [3]; (b) test cycle scheme (F—Change of frictional element pressing; T—Rotor braking (loading) torque).

Factors influencing the performance of PEA include frictional element pressure (clamping) on the rotor $F_N$ (N) and drive loading braking torque $T$ (Nmm). The duration of the test was 8 h, with a total friction run of 5.3 km. The variable clamping force of frictional elements was used for comprehensive evaluation of the operation characteristics of the PEA friction pair. For the first and fifth hours of operation, the actuator operated with a frictional element pressing force of 0.5 N, for 2nd and 6th h with a force of 0.75 N, for 3rd and 7th h with a force of 1.0 N, and for 4th and 8th h with a force of 1.25 N (Figure 2b).

According to the PEA loading, the test was performed in two stages: I—the drive was not loaded (1–4 h with 0.05 Nmm braking torque) and II—with the loading of the
drive (5–8 h at 2 Nmm). However, this loading torque was varied over the cycle, with the purpose to evaluate the effect of different PEA friction pairs on the actuator speed (or total operation run). In test hours 1–4, after each 35 min of operation, the load was gradually increased by discreetly increasing the braking torque to 1, 2, 3, and 4 Nmm for each subsequent five minutes (Figure 2b).

The controlled frequency of 27 kHz and voltage of 80 V of the signal generator DG 1022A (Rigol Technologies, Suzhou, China) and amplifier EPA-104 (Piezo systems Inc., Cambridge, MA, USA) were used. The computerized control of the device allowed us to maintain the loading parameters without deviating by more than 2–3%.

The frequency generator (9) and the amplifier (10) supplied the sinusoidal current of required amplitude and frequency to the piezoelectric element (2), exciting the standing wave effect. In the friction pair of the frictional element (5) and rotor (6), rotation torque appeared. The semi-automatic selection of the optimal excitation frequency was made according to the maximum rotation speed. Special software was used for regulation of the frequency generator output according to the required step, starting from the selected frequency. After estimation of the maximum speed, the frequency was regulated manually by the input of certain values [3].

Epoxy modified with 30% (the optimal content value to guarantee the hardness and avoid the brittleness that reduce the longevity of the frictional element) mass concentration SiO$_2$ (EPD + SiO$_2$) was used as the frictional material of the piezoactuator.

Polished carbon steel C45 with a hardness of HRC 50 was used as a bulk and reference material of the rotor ring (Ø 32 × Ø 20 × 2 mm) during the tests. The surfaces of the FOM modified specimens were annealed, polished, and cleaned by hexane. Then, they were processed with the antiadhesive fluoroligomeric polymeric material Foleox (trademark F5, structural formula R$_f$-CONHR$_2$, 1% solution) by immersing the specimens in liquid of Foleox and drying for 20 min at 100 °C; this procedure was repeated three times.

For further investigation of the mechanical characteristics, cylindrical carbon steel samples, 30 × 30 mm in size, of medium carbon steel AISI 1040 (DIN 1.1186, composition in wt%: Fe, (0.37–0.44) C, (0.6–0.9) Mn, (0.19–0.23) Si) were cut, quenched, and tempered for various hardness levels (40 < HRC < 63).

Scratch tests were carried out with a Vickers pyramid indenter, using a Micro-combi tester (CSM Instruments, Peseux, Switzerland). A diamond indenter of standard geometry, typically a 136° square-based pyramidal diamond (Vickers), was indented under a known load into the surface of the sample. During tests, load–scratch indentation depth-time data were recorded.

For analysis of surface morphologies of the modified layers and bulk surface, and wear value evaluation of friction element after the tests, an optical metallographic microscope Eclipse MA100 (Nikon, Tokyo, Japan) was used.

3. Results and Discussion

3.1. Operation of Piezoelectric Actuators with the Fluoroligomeric Contact Surface

Operation parameters and tribological characteristics of the PEA rotor with a polished carbon steel surface and FOM-modified rotor surface were compared, including rotation speed, braking PEA loading torque, and output torque of the rotor.

During the tribological test procedure (Figure 3a), the performance of PEA with a polished carbon steel rotor and with a steel rotor coated with a fluoroligomeric layer was compared.
When using the polished carbon steel, the rotor speed was low, and in the first stage of tests (without loading) when the normal frictional element pressing force $F_N$ was increased, at the tribo-contact, the rotation speed decreased from 140 to 110 rpm. At different output torques, the rotor stopped only at $F_N = 500$ mN (Figure 3b) during the first 4 h of the test. At a higher pressing force $F_N$ (750, 1000, or 1250 mN), the rotor reacted much less, and at $F_N = 1250$ mN, even at 4 Nmm output torque, the rotor still rotated with 60 rpm rotation speed (Figure 3c).

In the second stage, when the 2 Nmm braking load was applied, the speed decreased regularly from 100 to 80 rpm when the output torque increased. Figure 4 shows the operation speed and output torque when the overloadings by the clamping (pressing) force $F_N$ of 500, 750 and 1000 mN were applied. The PEA with the reference steel rotor stopped for 25 min at the overloading with pressing force $F_N = 500$ mN (Figure 4a), and at $F_N = 1000$ mN the rotor stopped altogether. The stopping of the rotor was related to the concentration of wear debris in the contact zone.
Further experiments show the influence of processing with fluoroligomeric material on the operation of tribo-contact of the PEA. The FOM-coated rotor rotated at a high and stable rotation speed of 130–145 rpm at the first stage of the tests (Figure 3). At the loading with the output torque, the rotor slowed down and stopped (Figure 3b). This regular speed increase is not characteristic of counter-body friction elements of EPD + SiO₂, and it could be related to the viscous structure of the fluoroligomeric layer on the rotor surface. However, this kind of friction pair successfully overcame the short-term overloadings (Figure 4a–c), restoring the speed and outcome torque of the PEA after the overloading was removed.

Higher efficiency of PEA with the rotor covered by the FOM layer is based on higher longevity of such friction pairs. Figure 5 presents the wear of counter-body frictional element after operation with the reference steel rotor and steel rotor coated with FOM. The wear of the friction element operated with the FOM-coated rotor is almost two times lower.
Figure 5. The wear of frictional element after PEA operation with reference steel rotor and steel rotor coated with fluoroligomeric layer.

Analysis and comparison of friction surfaces helps to ascertain the active participation of fluoroligomers in the friction processes of PEA friction pairs when using the steel rotor (Figure 6) and rotor with FOM coating (Figure 7).

Figure 6. Surface pictures of PEA contact bodies after applying the reference steel rotor: (a) rotor surface before the testing (×200); (b) rotor surface after the testing (×500); (c) frictional element EPD + SiO₂ after the tests in non-polarized light (×100); (d) frictional element EPD + SiO₂ after the tests in polarized light (×100).
The traces of grinding-polishing (vertical grooves in Figure 6a) and traces of friction element contact after the test (vertical tracks with regular micro-cuts; Figure 6b) are visible on the rotor surface. The area of contact of the friction element with the rotor (Figure 6c) is visible as lightly abraded spots of SiO\(_2\) particles. In polarized light (Figure 6d), the brown spots are the rust spots formed by corrosion of the micrometric abrasion products. When the piezoelectric rotor stops or decelerates to the minimum speed, the fretting wear occurs on the rotor surface.

Surface pictures of the rotor modified with FOM show that the structure of the surface of the rotor before (Figure 7a) and after (Figure 7b) the tests is analogous to the reference surface; however, in the case of FOM coating, the interference colors are present even after the tests, which is typical for oligomeric materials. This also proves the sustainability and renewability of the FOM layer during the operation of PEA contact. The surface images of the frictional element confirm the transferability of the FOM layer, showing clearly visible oligomeric material accumulations at the counter-body’s “end” zone (Figure 7c). This shows that fluoroligomeric layers have a specific texture, close to “jelly-type” coatings, and in such places, the bodies possibly come into contact through the FOM layer. This causes greater slippage, especially at the lowest pressing force of 500 mN (with this force, the PEA works at very uneven torque and rotation speed). It is likely that the progressive increase in the PEA operation speed is caused by displacement of the FOM film from the friction pair gap (the first phase of the test, at 500 and 750 mN pressing force). The picture of the friction element surface in polarized light (Figure 7d) shows that the content of the FOM film on the counter-body is different, taking into consideration the accumulation of metallic wear debris (which is darker in polarized light because of the cubic grid) in different zones of the contact area.
3.2. Mechanical and Structural Properties of Fluoroligomeric Films

The nature of FOM efficiency in tribological applications is related to the mechanical and structural peculiarities of fluoroligomeric layers. An important point in considerations of efficiency of the FOM is the question of whether a protective layer is formed or, alternatively, whether the bulk material is modified. The depth profiles prove that foreign elements can be detected up to a depth of several hundred nanometers in the volume. For the investigation of the mechanical properties of the FOM-coated surface, scratch-indentation and frictional measurement at conventional hardness testing was performed.

Typical loading and unloading curves from a scratch-indentation test cycle are presented for the reference steel surface (Figure 8) and the surface with fluoroligomeric film (Figure 9). Tests were performed for two different loading forces (2.5 and 5.0 N). The depth of the circle of contact was obtained from the difference in scratch-indentation depth and residual depth.

![Typical loading and unloading curves from a scratch-indentation test cycle](image)

**Figure 8.** Typical loading and unloading curves from a scratch-indentation test cycle for reference steel surface: (a) loading force 2500 mN; (b) loading force 5000 mN.
Results of the scratch-indentation tests show that the final residue depth of the steel surface (about 2.5 mm) was higher at lower loading, and this shows a higher plasticity of the FOM coated surface (2.3 mm residual depth). However, at higher loading, the thin fluoroligomeric film (about 1 µm) was destroyed and the surface lost its plasticity.

The variations of the friction force, the polymer transfer of the slider material onto the indenter, and the friction process of contacting the surface by the indenter take place during scratch-indentation. At the static contact point, no polymer transfer occurs, and it seems that the surface films do not break. This follows, because during the scratch process, there is slipping of the metal along the face of the indenter. After sliding, transfer fragments of polymer are found on the indenter and on the circular contact surface; the black shaded regime is formed because of the material removal. This is the bulk body shearing regime, and the other is the interface shearing regime, which may cause an increase of the real area of contact together with a progressive increase of friction force. This phenomenon can be well explained by the fact that the shear strength in the bulk body is higher than that of the interface shearing regime.

The friction force generated between sliding bodies has two principal components, commonly described as the adhesion and deformation. Adhesion is related to the shear
strength of the adhesive junctions formed on the real area of contact. Under dry conditions, the adhesion contribution to total friction is so large that the deformation component of friction is almost always negligible.

The micro-mechanical tribological and the tribo-chemical mechanism can explain the resistance to the friction of polymer materials. Surface material of polymer will abrade and adhere to the steel (or tungsten-carbide indenter) counter-body surface, forming the extensive FOM film [16]. When the steel surface covered with the layers containing FOM coating material slides against the pure steel surface the FOM material, transfer to the steel surface occurs. This formation of new transfer films on the steel surface is more complex. This results in the adhesion of wear debris to both surfaces, plastic deformation and strain hardening of transferred layers and patches, fractioning, and layer oxidation [17]. After sliding, the friction pair becomes the FOM layer on FOM film on the steel, with a much lower friction coefficient. This could be the basis for the long-term effect of fluoroligomers. In addition, in the wear reduction mechanism of fluoropolymer films, important roles are played by the contact conditions of sliding surfaces, creating a third body in the form of transfer film and degraded polymer particles [18]; the mechanochemistry of polymer defluorination, lowering the energy barrier by the polymer fillers; surface functionality; and the amount of fracture products in transfer film [19].

The efficiency of fluoroligomeric materials is based on its surface strengthening ability; it not only decreases the surface microcracks, but can re-generate its fluoroligomeric layers at the process of abrasion and friction. The stability of FOM bonds with bulk material under friction contact is explained by the mechanism of exo-electronic and reductive reactions. Those reactions change the initial structure of fluoroligomeric film and its products, creating the regenerating “tribological film”, which survives even after abrasion of much thicker metal layers as the thickness of FOM film [12,20].

4. Conclusions

Compared with the uncoated steel, the fluoroligomeric coated surface showed higher plasticity of the modified surface during scratch tests when the fluoroligomeric film was not destroyed by higher loading.

Fluoroligomer treatment of the surface considerably improved the performance of the piezoelectric actuator, including sustaining of a stable speed and restoring the rotation after the overloads.

The higher efficiency of piezoelectric actuators with a fluoroligomeric layer on the roller is related to almost two times lower wear of the friction element operating with coated rollers compared to the reference steel rollers.

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