

The Influence of Cu Nanoparticles on the Tribological Properties of Polydopamine/PTFE + Cu Films

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Abstract Polydopamine (PDA) is effective in increasing the adhesion between polytetrafluoroethylene (PTFE) and a stainless steel substrate, as a result reducing the wear in PTFE films by up to 500 times. By incorporating Cu nanoparticles in PTFE films, the wear resistance can be further enhanced. The present study examined the influence of Cu nanoparticles on the tribological properties of PDA/PTFE films. Tribological tests were carried out using a linear-reciprocating, ball-on-flat configuration. The surface morphology and film wear were characterized using atomic force microscopy, scanning electron microscopy, optical microscopy, and contact profilometry. The results show that at a particularly low concentration of 0.01 wt%, Cu nanoparticles doubled the wear life of PDA/PTFE films. The increased wear resistance resulting from the incorporation of Cu nanoparticles is explained by four factors: increased spreading of the film resulting from a lower melting point, the formation of micro-cracks on the film surface reducing debris size, the formation of a transfer film on the counterface, and enhanced adhesion between the PTFE + Cu composite and PDA, as well as enhanced cohesion in the composite film.

Keywords PTFE · Polydopamine · Cu nanoparticles · Thin film · Wear · Solid lubricant

1 Introduction

Polytetrafluoroethylene (PTFE) is a well-known, low-friction material. However, it exhibits poor wear resistance. To make PTFE more suitable for use in tribological applications, considerable focus has been placed in reducing its wear rate. Various fillers and polymer blends, such as glass fiber [1], alumina [2], graphite [3], and PEEK [4], are used to reduce the wear rate in bulk PTFE. These fillers increase the crystallinity, ultimate strength, elongation to failure, and toughness [5], and as a result, reduce wear in PTFE.

The emergence of micro-electro-mechanical systems (MEMS) technology and advances in thin film deposition methods have allowed thin films to be studied for many applications. In the field of tribology, thin films have drawn interest as solid lubricants. Various polymer, ceramic and metal films have been investigated to determine their friction and wear properties. Nevertheless, there is little research on PTFE thin films [6]. One study found that sputtered PTFE films have a wear rate ten times lower than bulk PTFE. However, the films exhibit a significantly higher coefficient of friction (COF), displaying values ranging between 0.3 and 0.4 [6, 7]. Another investigation determined that the incorporation of nanodiamonds in a PTFE film produced a quantifiable improvement in tribological properties [8]. However, the cost–benefit ratio of using nanodiamonds in this application may preclude their use. Consequently, there is still a need to find a satisfactory solution to the problem of wear in PTFE films and to further investigate their tribological properties.

Cu is a material with high ductility and thermal conductivity, which allows it to be used in many tribological applications. The use of Cu nanoparticles in lubricating oils, for example, can decrease friction, reduce wear, and provide self-repairing properties to sliding surfaces [9, 10].

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Furthermore, Cu nanoparticles have shown to improve the tribological properties of bulk PTFE. For instance, Jia et al. [11] added Cu nanoparticles to PTFE/serpentine composites and found that they significantly reduced friction and wear. Likewise, Yan et al. [12] added Cu nanoparticles to PTFE/nano-expanded-graphite and found that Cu increased the hardness, modulus of elasticity, yield strength, and tensile strength of the composite. An increase in these mechanical properties can improve the tribological properties of PTFE. Moreover, certain studies have investigated the effect of Cu on PTFE films. For example, thermal-sprayed PTFE-Cu-Al films can produce a strong transfer film on the counterface and exhibit moderate wear resistance [13].

The present study builds on our previous investigation [14] into the influence of a polydopamine (PDA) adhesive layer on the tribological performance of a thin PTFE top coat, referred to as PDA/PTFE. The use of PDA increased the wear life of PTFE films over 500 times without increasing the COF [14]. The current work studies the combined effect of using Cu nanoparticles as a filler in PTFE in addition to the use of a PDA adhesive layer. It was observed that the incorporation of Cu nanoparticles doubled the wear life of the PDA/PTFE film, even at a concentration as low as 0.01 wt%.

Two types of thin films were evaluated in this study: PDA/PTFE, and PDA/PTFE + Cu. PDA/PTFE consisted of an adhesive PDA layer and a PTFE top coat. PDA/PTFE + Cu consisted of an adhesive PDA layer and a PTFE + Cu nanoparticles composite top coat. The objective was to examine the tribological performance of these films and determine the influence of the Cu nanoparticle filler.

2 Experimental Details

2.1 Sample Preparation

The substrate selected for this investigation was type 316 polished stainless steel sheets with 0.76 mm thickness (9759k31, McMaster-CARR, USA). These sheets were first cut into 5 × 5 cm square samples. The squares were soaked in acetone in an ultrasonic bath for 20 min to remove oils and organic contaminants. They were then soaked in isopropyl alcohol in an ultrasonic bath for 5 min and subsequently rinsed in deionized (DI) water to remove residues. Finally, the samples were blown dry using nitrogen gas.

Thin films were deposited on the substrates by dip coating into aqueous colloidal dispersions due to its simplicity and low cost. Trizma base (T1503, Sigma Aldrich, St. Louis, MO, USA), dopamine hydrochloride

(H8502, Sigma Aldrich, St. Louis, MO, USA), a PTFE nanoparticle aqueous dispersion (TE-3859, DuPont, USA), and a Cu nanoparticle dispersion were used to prepare the dip-coating solutions. The PTFE nanoparticle size ranges from 50 to 500 nm according to DuPont's product information. The Cu nanoparticles were synthesized by reducing copper(II) acetylacetonate ($\text{Cu}(\text{acac})_2$, 0.2 mmol, 0.0525 g) with dodecylamine (DDA, 5 g) in a 25-mL round-bottom flask equipped with a magnetic stirring bar at 220 °C for 25 min under argon protection. The resultant Cu nanoparticles were purified by ethanol to remove excess reactants. The surface ligand, DDA, was replaced by butylamine in 50 % v/v ethanol/ H_2O solution overnight at 25 °C. After ligand exchange, the Cu nanoparticles were suspended in H_2O .

The thin films were dip coated at an insertion and withdrawal speed of 10 mm/min using a dip coater (KSV DC, KSV Instruments Ltd., Espoo, Finland). First, the PDA film was deposited by immersing the clean stainless steel squares in an alkaline solution of dopamine hydrochloride for 24 h [14]. Next, PTFE or PTFE + Cu top coats were deposited onto the PDA film. The PTFE aqueous dispersion has an as-received solids concentration of 60 wt%. In order to produce thinner coats of PTFE, the concentration of the as-received PTFE dispersion was diluted with DI water to produce a PTFE dispersion of 40 wt% concentration. For the PTFE + Cu top coat, the PTFE dispersion was mixed with Cu nanoparticles to produce a final composite dispersion of 40 wt% PTFE and 0.01 wt% Cu nanoparticles. Inductively coupled plasma mass spectrometry (ICP-MS) measurements revealed an average Cu content of $6.6 \pm 3.1 \mu\text{g}$ in the PDA/PTFE + Cu films, which were scalped off from the substrate using a glass slide, dissolved in 200 μL HNO_3 , and then diluted to 5 mL using 2 % HNO_3 as matrix for ICP-MS analysis.

Based on DuPont guidelines [15, 16] and preliminary results [14], the samples were heated on a hot plate at 120 °C for 3 min to remove water. Next, the samples were baked in a furnace at 300 °C for 4 min to remove wetting agents, and finally, the films were sintered in a furnace at a temperature of 372 °C for 4 min to fuse the film to the substrate. The total coating thicknesses, measured using a stylus surface profilometer (Dektak 150, Bruker, Santa Barbara, CA, USA), were an average of 1.32 μm for PDA/PTFE films and 1.17 μm for PDA/PTFE + Cu films. The total coating thickness was determined by running the stylus across the leading edge of the coating on the partially dip-coated stainless steel substrate, giving a value of the step height between the bare substrate and the coating surface. Of the total coating thickness, the PDA layer composed approximately 50 nm, determined by atomic force microscopy (AFM) height images of single PDA layer.

2.2 Tribological Studies

A Tribometer (UMT-2, Bruker, Santa Barbara, CA, USA) was used to perform linear-reciprocating tribological tests, measuring the static and dynamic COFs simultaneously. The tests were performed by repeatedly sliding a 7-mm-diameter chrome steel ball against the test sample in a linear-reciprocating fashion. They were repeated on three samples of each coating type under an applied normal load of 50 g, a stroke length of 15 mm, and a speed of 2.5 mm/s.

The tribological tests were categorized into three different studies: a durability study, a wear progression study, and a linearly increasing load scratch test. First, a durability study was carried out to determine the effect of the Cu nanoparticle filler on the film wear life. The durability tests were performed until a sharp increase in the COF was observed. A sharp increase in the COF indicated that the film on the sample had been worn to the extent that the stainless steel substrate was exposed to the counterface. As a result, the number of rubbing cycles until this point represented the wear life or durability of the film.

Second, a wear progression study was performed. This study consisted in repeating the tribological tests for 1, 10, 60, and 1000 cycles, analyzing the wear progression after each test. This method allowed examination of the wear track at various stages of wear. The purpose of this test was to observe changes in surface morphology, measure wear progression, and determine the wear mechanism affecting each sample type.

Finally, scratch tests with a linearly increasing normal load were performed using the UMT Tribometer to slide a 7-mm chrome steel ball across the surface of each sample at a constant speed of 0.1 mm/s. The normal load was increased from 100 to 460 mN within a distance of 20 mm. This test was repeated three times on each sample type.

2.3 Sample Characterization

A stylus surface profilometer was used to determine the wear track cross-sectional profiles. The profiles were measured using a 12.5- μm -radius stylus with 3 mg contact force, 800 μm scan length, and scan duration of 30 s. The wear track cross-sectional profile was established by running the stylus along the width of each wear track.

Scanning electron microscopy (SEM; model XL-30, Phillips/FEI, Hillsboro, OR, USA) was used to characterize the surface topography and wear of all samples. Several SEM micrographs at different surface locations were captured to examine the micro-scale topography of the coated surface and inside the wear tracks. To better show the surface topography, 45° oblique angle views were used to capture each micrograph. In addition, an atomic force

microscope (AFM, Dimension Icon, Bruker, Santa Barbara, CA, USA) was used to produce topographical images of the samples and measure the surface roughness.

3 Results and Discussion

3.1 Particle Size and Surface Topography

Transmission electron microscope (TEM) images of the Cu nanoparticles used in this study are shown in Fig. 1. The nanoparticles are spherical in shape, with diameters ranging from 5 to 20 nm and do not exhibit considerable agglomeration.

Figure 2a, b shows AFM images of PDA/PTFE and PDA/PTFE + Cu films, respectively. From the images, it is evident that the PDA/PTFE + Cu film is denser and less porous than PDA/PTFE. It is likely that the metallic nanoparticles aid in the spreading of the film by reducing the melting point of the composite. Studies have shown that nanoparticles typically have lower melting points than their bulk form [17, 18]. Cu nanoparticles, specifically, can begin to melt at the surface at temperatures as low as 200 °C [19]. The slightly lower thickness of the PDA/PTFE + Cu films compared to PDA/PTFE confirms that those films are more compact. The increased degree of melting and spread of the film aids in the adhesion between the PTFE film and the PDA-coated surface. AFM topographical images showed that PDA/PTFE + Cu films had R_a and R_q surface roughness values that were slightly higher than those of the PDA/PTFE films.

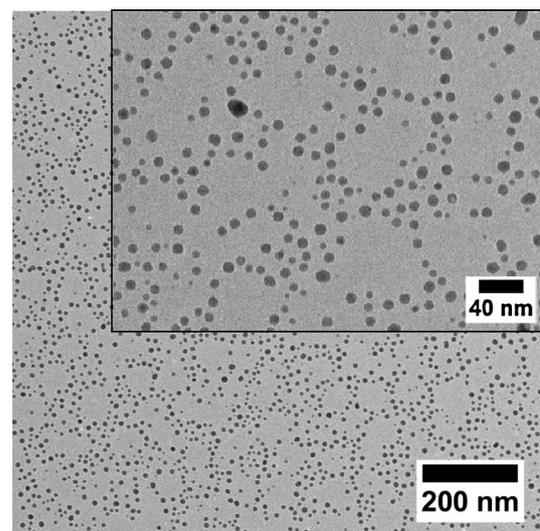


Fig. 1 TEM of copper nanoparticles

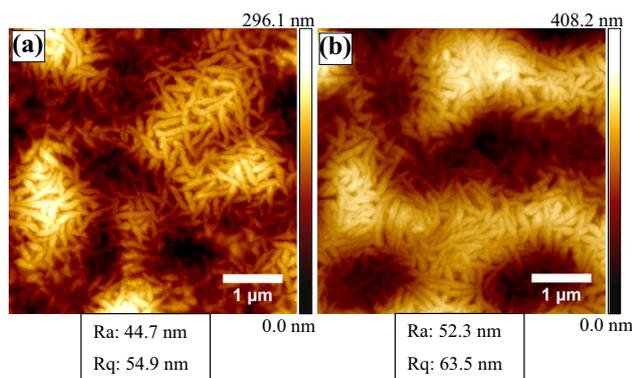


Fig. 2 AFM topographical images and surface roughness values for **a** PDA/PTFE and **b** PDA/PTFE + Cu films

3.2 Friction and Durability

The durability test results for the top performers of PDA/PTFE and PDA/PTFE + Cu films are shown in Fig. 3. Both sample types typically experienced a slight increase in COF as the tribological test progressed. This increase in friction is caused by changes in the chemical properties of PTFE due to the high shear forces. As was determined through chemical analysis in our prior study on PDA/PTFE [14], it is evident that the PTFE molecules undergo scission and crosslink with each other, reducing the wear rate of the film with successive cycles, but also progressively increasing the COF over time. In Fig. 3, the spike in friction representing failure of the film is clearly visible. At this point, the tribological tests were stopped to allow observation of the worn surfaces. The drop in friction observed at approximately 11,000 and 18,000 cycles for the PDA/PTFE + Cu film is typically observed after a defect or tear in the film is corrected or replenished with material transferred from the counterface back to the sample surface.

Figure 4 shows a summary of tribological test results for three PDA/PTFE and three PDA/PTFE + Cu samples. Both films showed similar COF values, which averaged

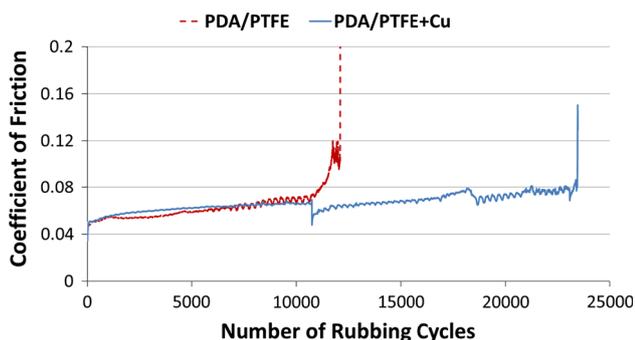


Fig. 3 Durability test results for PDA/PTFE and PDA/PTFE + Cu films

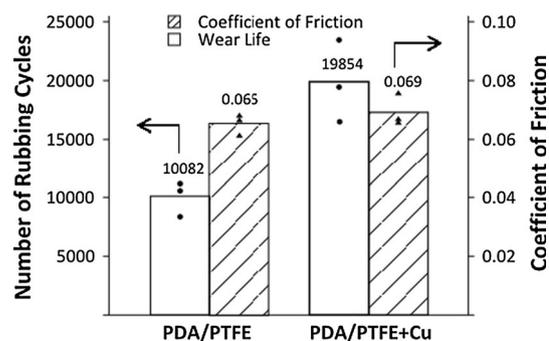


Fig. 4 Wear life and coefficient of friction results for PDA/PTFE and PDA/PTFE + Cu. The bar chart shows averages with individual measurement results shown. The average COF is calculated based on the COF before film failure

0.065 and 0.069, respectively. However, on average, PDA/PTFE + Cu films were able to withstand 19,854 rubbing cycles, while PDA/PTFE films only lasted for 10,082 cycles. It is evident that the wear life of the film has doubled using a considerably low Cu concentration of 0.01 wt%.

SEM micrographs of the wear tracks produced on the top performing PDA/PTFE and PDA/PTFE + Cu samples are shown in Fig. 5. It is clear from Fig. 5a, e that the substrate in both samples was abraded at the center of the wear track, as is evident from the deep grooves displayed in the image. These grooves are more clearly shown in Fig. 5b, f, at a higher magnification. In Fig. 5c, g, a smooth area adjacent to the grooves is displayed. In this area, the film has been compacted by the sliding counterface, and the substrate polishing lines (perpendicular to the sliding direction), to which the film has conformed to, are clearly visible. Figure 5g shows a plate-like slab of PTFE which has likely deposited back onto the surface of the sample after the formation of a transfer film on the counterface. These re-deposited slabs help replenish the film in areas that are susceptible to failure. Figure 5d, h shows images that were captured at the periphery of the wear track to show the topography of the buildup which accumulated at the edges of the contact area during the tribological test. Both sample types show fibrils and striations that are typically observed in PTFE films. However, a key difference for PDA/PTFE + Cu, shown in Fig. 5h, is the presence of micro-cracks. These micro-cracks result in reduced debris size. The Cu nanoparticles present within the film inhibit crack propagation, preventing large amounts of film from being removed with each pass of the counterface, and thus contributing to a reduction in wear.

3.3 Wear Progression

Figure 6 shows SEM micrographs of the wear track for each wear progression test. PDA/PTFE, shown in Fig. 6a–d, had a wear track width of 80 μm after 1 cycle. The wear

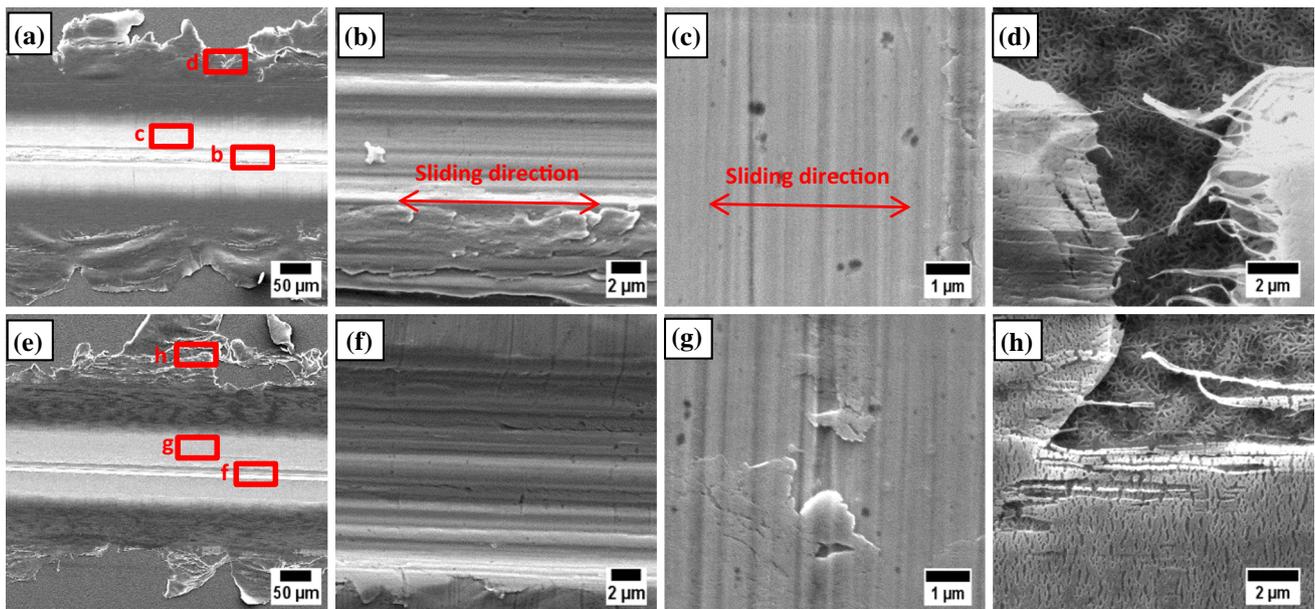


Fig. 5 SEM micrographs of the wear track on PDA/PTFE film showing **a** entire width of wear track, **b** wear track center grooves, **c** area adjacent to center grooves, **d** buildup and on PDA/PTFE + Cu

film showing **e** entire width of wear track, **f** wear track center grooves, **g** area adjacent to center grooves, and **h** buildup

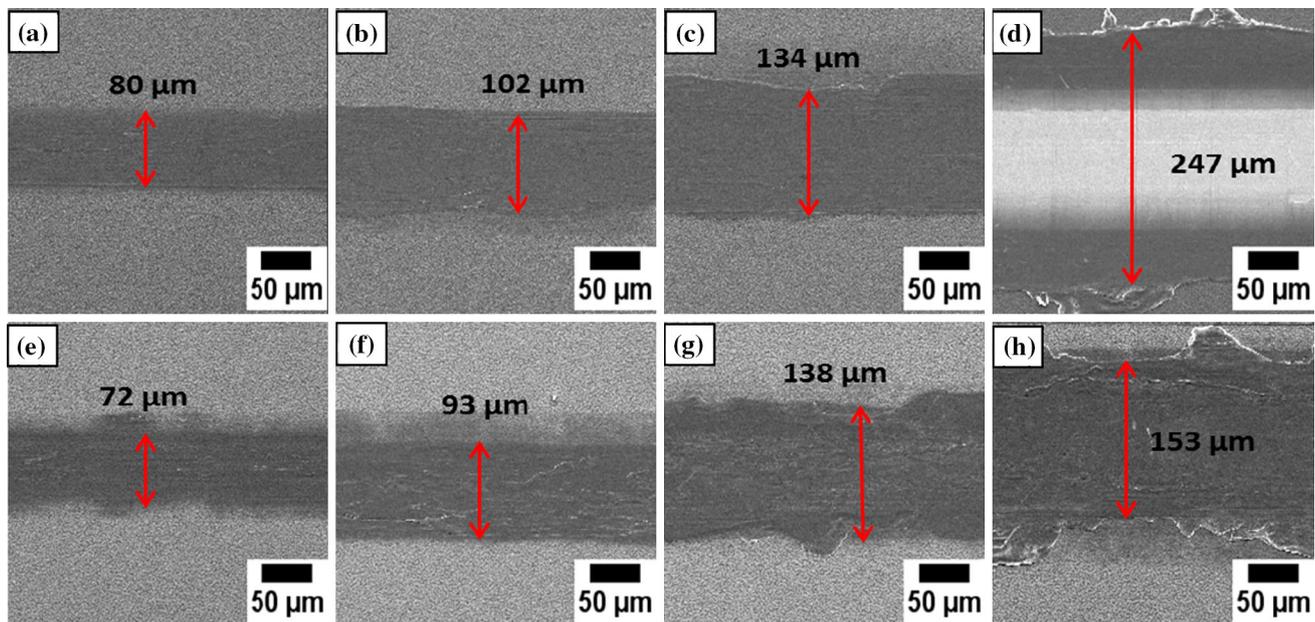


Fig. 6 SEM micrographs of the wear tracks on PDA/PTFE film tested for **a** 1 cycle, **b** 10 cycles, **c** 60 cycles, and **d** 1000 cycles and wear tracks on PDA/PTFE + Cu film tested for **e** 1 cycle, **f** 10 cycles, **g** 60 cycles, and **h** 1000 cycles

track width then increased to 102 μm after 10 cycles, 134 μm after 60 cycles, and finally 247 μm after 1000 cycles. PDA/PTFE + Cu, shown in Fig. 6e–h, had a wear track width of 72 μm after the first cycle, 93 μm after 10 cycles, 138 μm after 60 cycles, and 153 μm after 1000 cycles. Based on the wear track width, the initial wear of both sample types is quite similar; however, between 60

and 1000 cycles, the wear of the PDA/PTFE + Cu film is much less than that of PDA/PTFE.

The slower wear progression of PDA/PTFE + Cu films, determined from the width of the wear tracks, was confirmed through cross-sectional profiles of the wear tracks obtained using a Dektak stylus profilometer. The cross-sectional profiles permitted the observation of the wear

depth at 1, 10, 60, and 1000 rubbing cycles. Figure 7a shows the cross-sectional profiles for the PDA/PTFE film, and Fig. 7b shows the cross-sectional profiles for the PDA/PTFE + Cu film. The difference in wear progression after 60 cycles is again very clear. The depth of the wear track is proportionate for both sample types up to 60 cycles; however, after 1000 cycles, the depth of PDA/PTFE is approximately 1000 nm, while the depth of PDA/PTFE + Cu is only about 350 nm (very little increase from the wear depth after 60 cycles). This difference in depth confirms that PDA/PTFE + Cu has higher wear resistance than PDA/PTFE and is thus more durable.

Figure 8 shows optical microscope images of the chrome steel ball counterfaces used for the wear progression study. Figure 8a–d shows images of the chrome steel balls rubbed against the PDA/PTFE film for 1, 10, 60, and 1000 cycles, respectively. Figure 8e–h shows images of the balls rubbed against the PDA/PTFE + Cu sample for the same number of cycles. The balls rubbed against PDA/PTFE showed little to no signs of a transfer film on the surface. The balls rubbed against PDA/PTFE + Cu films, on the other hand, had a significant amount of transfer film formed after only 10 rubbing cycles. It appears that the majority of the transfer film was deposited at the front edges of the contact area as the ball slid forward and, likewise, at the back of the contact area as the ball slid in the reverse direction. The transfer film was also present for 60 and 1000 rubbing cycles. The formation of the transfer

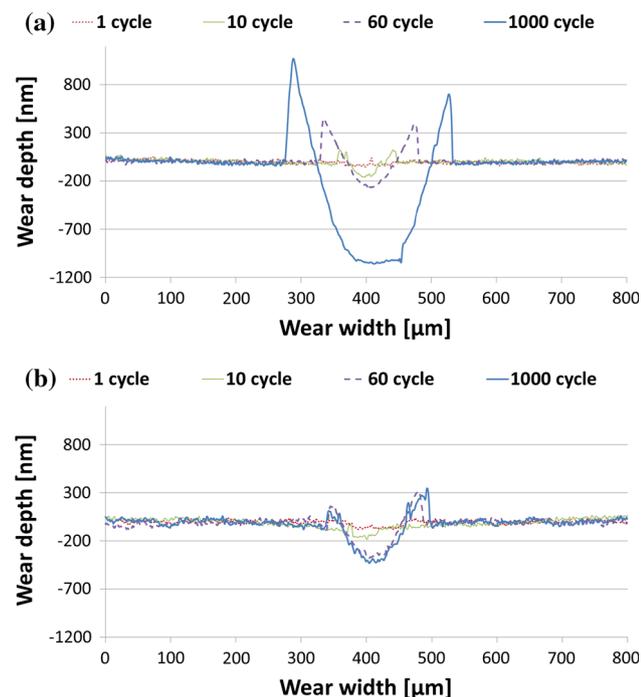


Fig. 7 Dektak cross-sectional profile of film wear track after 1, 10, 60, and 1000 cycles on **a** PDA/PTFE and **b** PDA/PTFE + Cu

film explains the drastic reduction in wear rate observed in the cross-sectional profiles of the wear tracks.

3.4 Scratch Test

The results for the scratch tests with a linearly increasing normal load are shown in Figs. 9 and 10. The PDA/PTFE film, shown in Fig. 9, was detached from the substrate after the test progressed for a distance of approximately 15 mm and the normal load reached a value of 370 mN. Optical images of the scratch show the different stages of wear. Figure 9a depicts the characteristic wear morphology in the initial segment of the scratch between 1 and 5 mm where the COF is relatively stable and has a value of about 0.09. Figure 9b depicts the characteristic wear morphology in the second segment between 5 and 15 mm. In this segment, the COF intermittently jumps between 0.07 and 0.11. This stick–slip behavior is caused by the film building up in front of the counterface as it slides against it, producing areas where the film has accumulated into folds or corrugations. At first, the COF increases because the ball is pushing against the film, accumulating material in front of it as it slides forward. Once the accumulation of the film in front of the ball is large enough, it forces the ball to slide over the buildup, exhibiting slip behavior. This stick–slip behavior continuously repeats for this section of the scratch. The areas of accumulated material in the scratch are shown in Fig. 9b. The distance between these areas of accumulated film is approximately 100 μm which is comparable to the distance between the peaks and valleys representative of stick–slip behavior in the COF plot in Fig. 9d. The commensurate distance confirms that the accumulation of material is the cause of the stick–slip behavior. Figure 9c shows the characteristic wear morphology between 15 and 20 mm. Here, the stick–slip behavior is more pronounced and it is evident from the optical image that there are segments of the scratch where the PTFE top coat has detached from the PDA layer.

Figure 10 shows the scratch produced on the PDA/PTFE + Cu film. Figure 10a depicts the characteristic wear morphology in the scratch between 0 and 2.5 mm. In this segment, the COF is stable but shows a slightly increasing trend from 0.08 to 0.10. Figure 10b depicts the characteristic wear morphology between 2.5 and 8 mm where the film accumulates into corrugated segments, similar to what is observed in Fig. 9b. The ploughed areas in between the corrugated segments have less film remaining; however, the PDA layer has not yet been exposed. In this section of the scratch, the COF intermittently jumps between 0.07 and 0.12. Figure 10c depicts the characteristic wear morphology between 8 and 18 mm. In this section, the corrugations in the film are evenly and closely spaced producing an even surface and a stable COF that remains at approximately 0.10. Figure 10d shows the characteristic wear morphology between

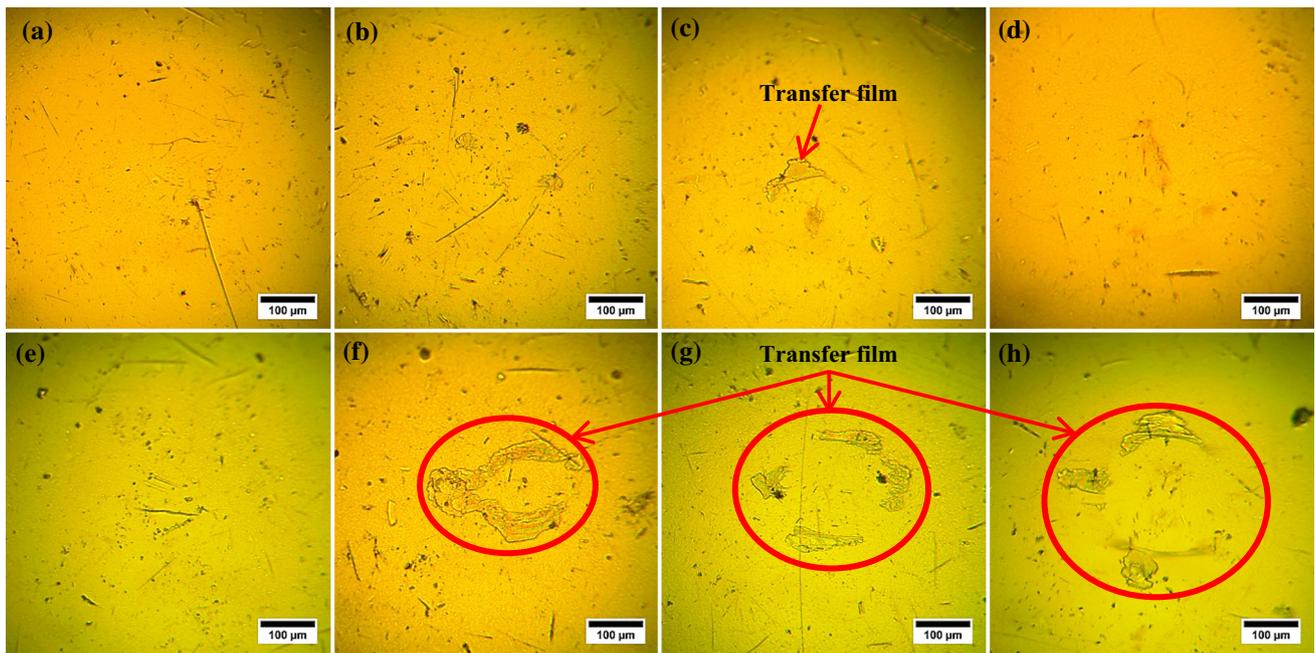
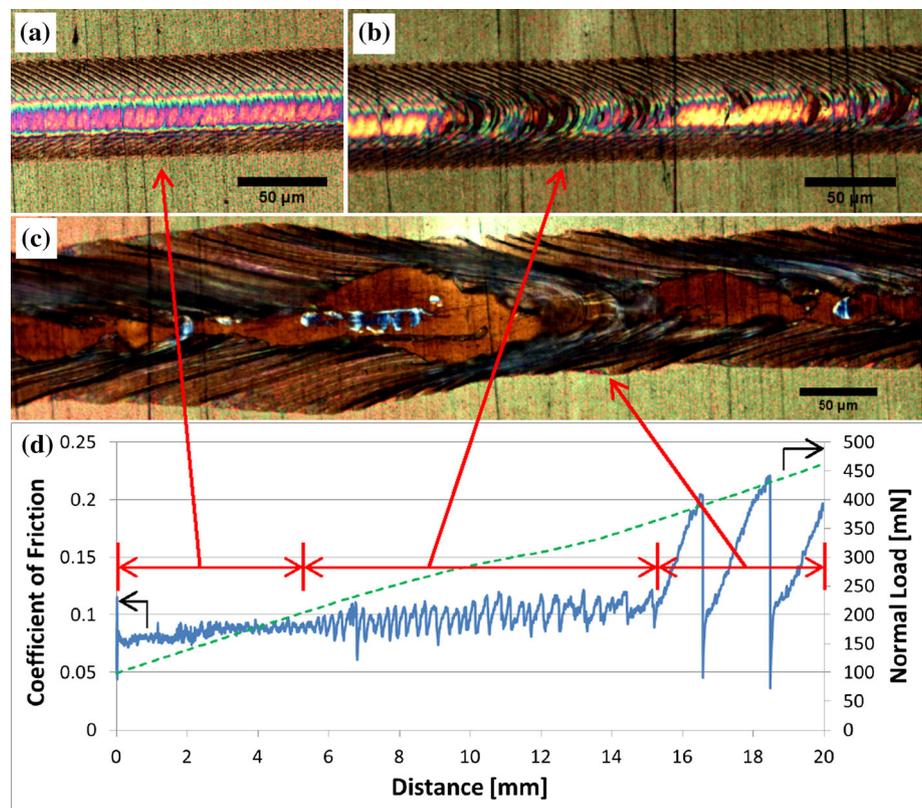


Fig. 8 Optical microscope images of the chrome steel balls rubbed against PDA/PTFE films for **a** 1, **b** 10, **c** 60, and **d** 1000 cycles and chrome steel balls rubbed against PDA/PTFE + Cu films for **e** 1, **f** 10, **g** 60, and **h** 1000 cycles

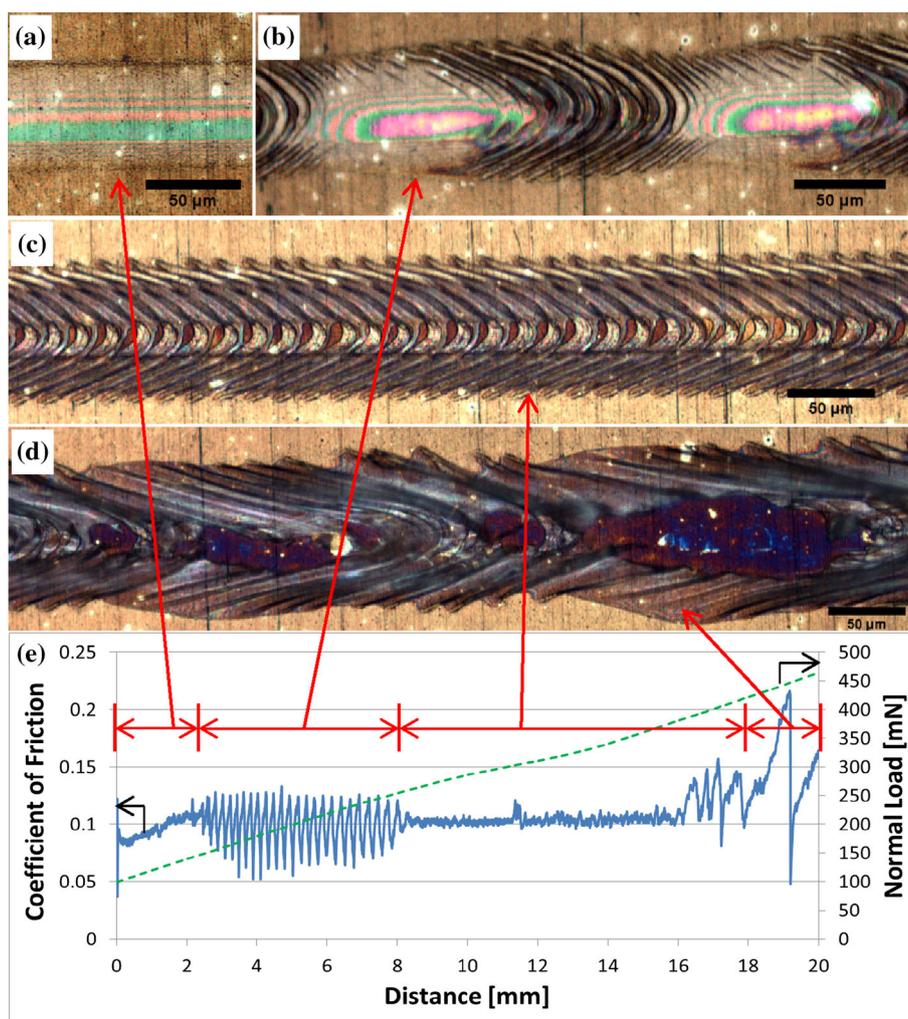
Fig. 9 Optical images showing the characteristic morphology for small representative areas in segments **a** 1–5 mm, **b** 5–15 mm, and **c** 15–20 mm of a scratch resulting from a linearly increasing load scratch test on PDA/PTFE, and **d** a plot of the COF and normal load versus distance for the same test



18 and 20 mm where the stick–slip behavior is very pronounced. Again, from the image, it is evident that the PTFE top coat has detached in certain areas of the scratch. The

areas where the PTFE film has detached expose PDA to the counterface. As the counterface slides over these areas, the COF increases sharply as shown in the plot in Fig. 10e. As

Fig. 10 Optical images showing the characteristic morphology for small representative areas in segments **a** 1–2.5 mm, **b** 2.5–8 mm, **c** 8–18 mm, and **d** 18–20 mm of a scratch resulting from a linearly increasing load scratch test on PDA/PTFE + Cu, and **e** a plot of the COF and normal load versus distance for the same test



the counterface slides over an area where PTFE is still present, the COF drops back down, producing stick–slip behavior.

The scratch tests revealed that the sample with Cu nanoparticles withstands significantly higher normal loads before the film detaches. For PDA/PTFE, the film was detached at a distance of about 15 mm and a normal load of 370 mN, whereas PDA/PTFE + Cu reached a distance of 18 mm and a normal load of about 420 mN before the film was detached from the PDA layer. These results indicate that the Cu nanoparticles may increase cohesion in the PTFE film and also improve adhesion of the PTFE film to the PDA layer.

4 Conclusion

The results indicate that the addition of Cu nanoparticles as a filler in PTFE increases the durability of PDA/PTFE films. PDA/PTFE + Cu films had a wear life that was, on

average, twice that of PDA/PTFE. It is also evident that the wear progression of PDA/PTFE + Cu films decreased significantly after 60 rubbing cycles. This became apparent through the cross-sectional profiles of the wear tracks which showed little increase in wear depth between 60 and 1000 cycles. On the contrary, the PDA/PTFE film showed a significant increase in wear depth at this stage of the tribological test. The increased wear resistance resulting from the incorporation of the Cu nanoparticles is explained by four factors: (1) increased spreading of the film resulting from a lower melting point, (2) the formation of microcracks on the film surface reducing debris size, (3) the formation of a transfer film on the counterface, and (4) enhanced adhesion between the PTFE + Cu composite and PDA, as well as enhanced cohesion in the composite film.

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recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation or Arkansas Biosciences Institute.

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