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Laser surface texturing of both thin polytetrafluoroethylene coatings and stainless steel substrates for improving tribological properties

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ABSTRACT

Hilbert curve patterns were laser-textured on both stainless steel substrates and polytetrafluoroethylene (PTFE) thin coatings by applying 15% and 5% of the 2.3-W full laser power, respectively. The nanomechanical and tribological behavior of both smooth and laser-textured PTFE coatings on both smooth and laser-textured stainless steel substrates were studied. It was found that laser-texturing thin PTFE coatings reduced the hardness of PTFE coatings and prevented the coating from tearing under nanoindenter scratches. Furthermore, laser-texturing on both PTFE coatings and the stainless steel substrate improved the coating wear life 29 times compared to the wear life of the control sample without any laser textures.

1. Introduction

Polytetrafluoroethylene (PTFE) is a polymer composed of carbon and fluorine atoms and is popular for its distinguished properties such as thermal resistance, chemical stability, low surface energy, and low maintenance cost [1,2]. The polymer is chemically inert due to the strong C–F bonds between the carbon and fluorine atoms of the PTFE [3]. Among all of the desirable properties, PTFE has a low coefficient of friction (COF), which makes its coating a great solid lubricant for surfaces that are in rubbing contact [4]. Besides low friction, PTFE's other properties, including corrosion resistance, drag reduction, superhydrophobicity, and self-cleaning, make the lubricant exceptional for industrial applications [5].

One major downside of PTFE, when used as a coating, is its low wear resistance, which limits its tribological applications [6]. However, laser-texturing of hard metal and metal composite substrate surfaces was found to improve the wear resistance of the PTFE coatings deposited on them [7–11]. For example, laser-texturing the Hilbert curve pattern on stainless steel showed significant improvement in the durability of thin polydopamine (PDA)/PTFE coatings [11]. Micropatterns on the surfaces produced by laser-texturing function as solid lubricant reservoirs and/or wear debris traps that prolong the wear life of the lubricant coating [11]. The roughness created by the textures also provided interlocking to the PTFE coating, thus preventing coating global delamination [12].

While laser surface texturing is capable of providing precise control of the micro/nanopatterns and is environmentally friendly, it has rarely been used to texture PTFE coatings for tribological application. Most studies on laser-texturing of PTFE were on texturing bulk PTFE or PTFE sheets and were for improving surface hydrophobicity by altering the micro/nanostructure of PTFE particles [3,13–19]. Liang et al. laser machined PTFE surfaces and presented a fibrous homogeneous and heterogeneous surface structure after laser machining. They also suggested a hair-like structure for laser-treated PTFE that was positively affected by laser fluence [16]. Kietzig et al. demonstrated that increasing laser power increased the density and length of hair-like structures [17]. Li et al. applied femtosecond laser treatment on PTFE sheets and fabricated rough microstructures to obtain superhydrophobic surfaces for oil and water separation [13]. Toosi et al. fabricated biaxial and uniaxial nano and micro patterns on PTFE surfaces and showed that PTFE hydrophobic morphologies depend on the laser parameters [14]. Also, Fan et al. laser textured submicron grooves on PTFE surfaces and showed that the intervals between textured grooves affected the superamphiphobicity of PTFE surfaces [15].

Only one study was found that investigated both the superhydrophobicity and the wear resistance of laser-textured PTFE coating on laser-textured Al_2O_3/Ni composites. A 5-time improvement in the wear life of the laser-textured PTFE coating on the laser-textured surface compared to the smooth PTFE coating on a smooth surface was reported [5]. However, in that study, only simple laser texture patterns of dimples and grooves were investigated and the coating had a very large thickness

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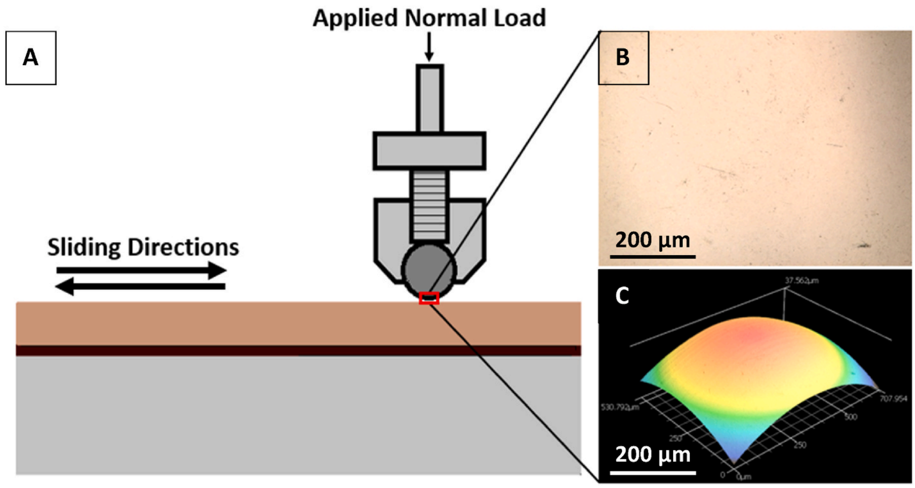

Fig. 1. Tribological test setup and optical images of the Cr-Steel counterface ball: A) ball-on-plate configuration, B) 2D optical image of the counterface ball with the curvature removed, and C) 3D optical image of the counterface ball.

Table 1
List of different sample types.

Sample Label	Substrate	Underlayer	Top layer
SS/PDA/PTFE	Smooth stainless steel	PDA	Smooth PTFE
SS/PDA/LT PTFE	Smooth stainless steel	PDA	Laser-textured PTFE
LT SS/PDA/PTFE	Laser-textured stainless steel	PDA	Smooth PTFE
LT SS/PDA/LT PTFE	Laser-textured stainless steel	PDA	Laser-textured PTFE

of 30 μm. Furthermore, the texture depths on the coating and substrate were very large, 20 and 55 μm, respectively. Additionally, no adhesive underlayer was used. It has been shown that using polydopamine (PDA) as an adhesive underlayer significantly improves the adhesion of PTFE coatings to the substrate and hence increases its wear life by reducing coating global delamination [20].

The objective of the current study is to understand the effect of laser-texturing shallow grooves on both the substrates and very thin (1.5 μm-thick) PDA/PTFE coatings on the coating tribological properties. Hilbert curve was chosen because it is a single, continuous curve that doesn't cross over itself, which makes the texturing process efficient and the resulting texture uniform. The pattern also combines the benefit of both parallel and perpendicular grooves so that lubricant can remain entrapped for longer periods, which could benefit situations where servicing worn components is expensive or difficult. Additionally, the anisotropic arrangement of the grooves helps ensure that the contact area for wear involves both parallel and perpendicular grooves. In this study, the same Hilbert curve pattern was used to laser-texture the stainless steel substrate and the thin PTFE coating, and the wear and friction of the smooth and laser-textured PTFE coatings on smooth and laser-textured stainless steel substrate were investigated.

2. Materials

316 stainless steel substrates (25.4 mm × 25.4 mm × 0.76 mm in size) and Cr steel balls (6.35 mm in diameter) were purchased from McMaster-Carr, IL, USA. Grit 320 sandpaper, 6 μm polycrystalline diamond suspension, and 0.06 μm amorphous colloidal silica suspension were purchased from Buehler, IL, USA. Dopamine hydrochloride and tris (hydroxymethyl) aminomethane were purchased from Sigma Aldrich, MO, USA. Also, aqueous PTFE dispersion (Teflon Dispersion DISP30) with 60 wt% of PTFE was purchased from Fuel Cell Earth, TX, USA.

3. Methods

3.1. Sample preparation

Stainless steel substrates were polished to mirror-finish in three steps with sandpaper, diamond suspension, and colloidal silica suspension for 2 min, 12 min, and 15 min, respectively, using an AutoMet 250 Grinder-Polisher (Buehler, IL, USA) with a head speed of 60 rpm, a base speed of 150 rpm, and normal load of 6 N. Stainless steel was chosen as the substrate because it is a commonly used material in many applications. The polished samples were then laser-textured using an A5 Series femtosecond laser (Oxford Lasers Ltd., UK) with 15% of the 2.3-W full laser power, 600 Hz laser frequency, and 1200 R.A. divider. Next, samples were washed in deionized (DI) water plus detergent, acetone, and isopropyl alcohol in a sonication bath for 20 min each. Then, PDA/PTFE coatings were deposited on the stainless steel substrates using a previously reported procedure [21,22]. Briefly, PDA was deposited on smooth and laser-textured stainless steel substrates at a temperature of 60 °C in a rocking bath containing dopamine hydrochloride mixed with a tris buffer solution. A rocking rate of 25 rpm and a rocking angle of 7° for 45 min was used. In this step, the pH of the solution was brought to 8.5 using Trizma base mixed with DI water. Finally, PTFE was coated on

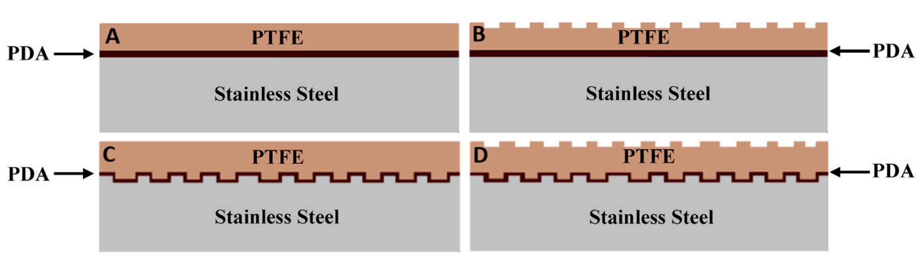

Fig. 2. Schematic of different sample types (Not to scale): A) SS/PDA/PTFE, B) SS/PDA/LT PTFE, C) LT SS/PDA/PTFE, and D) LT SS/PDA/LT PTFE.

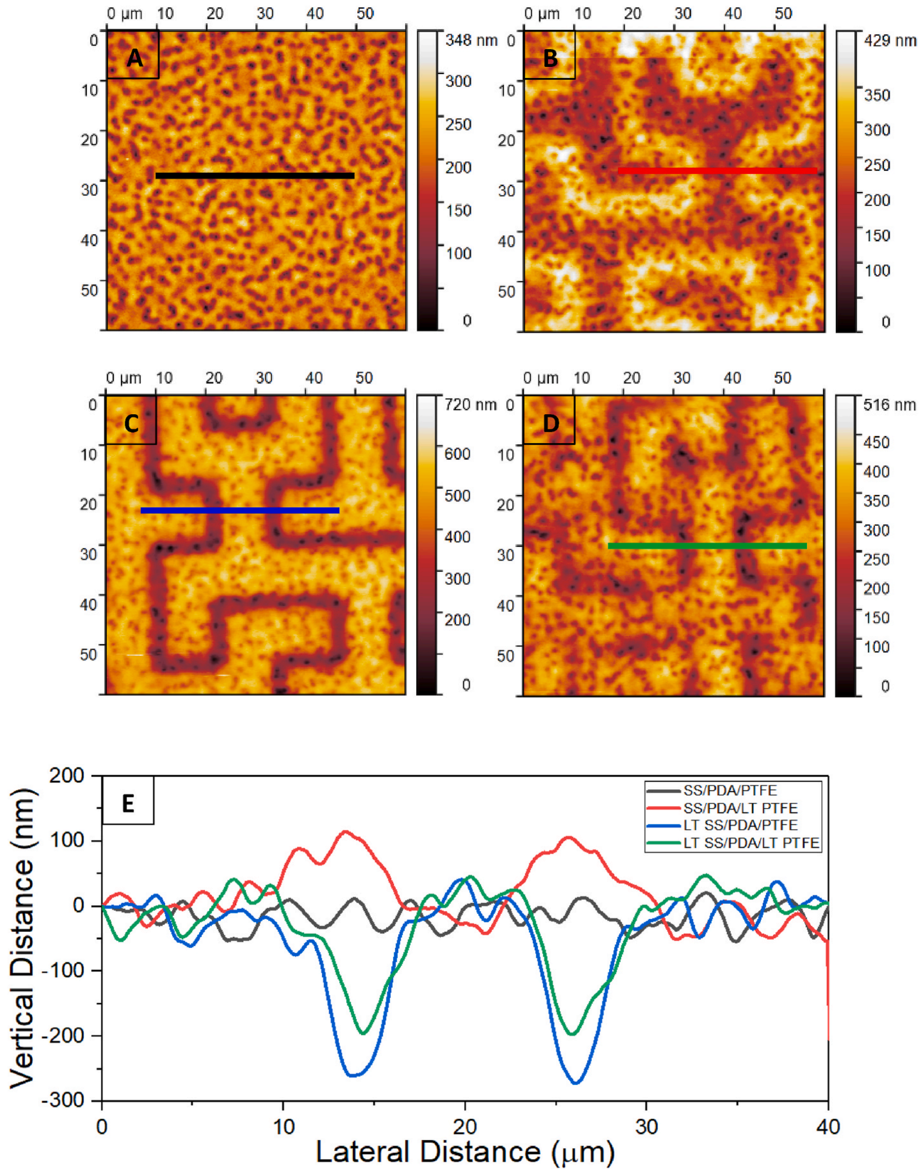

Fig. 3. AFM images of surface topography of PTFE coatings in: A) SS/PDA/PTFE, B) SS/PDA/LT PTFE, C) LT SS/PDA/PTFE, D) LT SS/PDA/LT PTFE, and E) profile of PTFE topography across the lines on images A-D.

PDA deposited stainless steel substrates using a dip coater (KSV Dip Coater, MD, USA) with a dipping and withdrawal speed of 10 mm/min, and soaking time of 1 min [23]. PDA/PTFE coated stainless steel samples were then heated at 120 °C and 300 °C and annealed at 372 °C for 4 min each. Hilbert curve pattern was laser-textured on the resulting PTFE coating with 5% of the 2.3-W full laser power. In addition, for performing nanoindentation and nanoscratch tests, square areas of 150 µm × 150 µm were laser-textured on the PTFE coatings using the same laser parameters.

3.2. Sample characterization

A 3D laser scanning confocal microscope (VK-X260, Keyence Corporation) was used to measure the surface of smooth and laser-textured substrates and image the wear tracks and the counterface balls after the durability tests. An atomic force microscope (AFM, Dimension Icon, Bruker) having a ScanAsyst air AFM tip (Bruker) with a 0.4 N/m spring constant was used to measure the surface morphology and surface roughness of PDA/PTFE coatings. A stylus contact profilometer (Dektak 150, Bruker Nano Surfaces) was used to measure the coating thickness and the profile of wear tracks after durability and progressive wear tests. X-ray photoelectron spectroscopy (PHI Versaprobe XPS system, Physical Electronics) was used to analyze the surface chemistry of PTFE coatings with and without laser texturing. A nanoindenter (TriboIndenter, Hysitron) equipped with a spheroconical indenter with a 5-µm-radius diamond tip was used to perform indentations to a maximum load of 100 µN with loading and unloading rates of 2.5 µN/s and a holding time of 2.5 s. These indentations were used to measure the modulus of elasticity and hardness of the smooth and laser-textured (LT) PTFE coatings. Additionally, the nanoindenter and the 5-µm-radius diamond tip were used to perform load-controlled scratch tests at maximum loads of 1000 µN, 2000 µN, and 3000 µN with an 8-µm scratch length.

3.3. Tribology testing

A tribometer (UMT-3, Bruker) was used to conduct linear reciprocating wear tests with a ball-on-plate configuration. Cr steel balls with 6.35 mm diameter and 0.221 µm average root mean square surface roughness were used as the counterfaces. This material was selected because it is commonly used in industrial applications. Smooth and

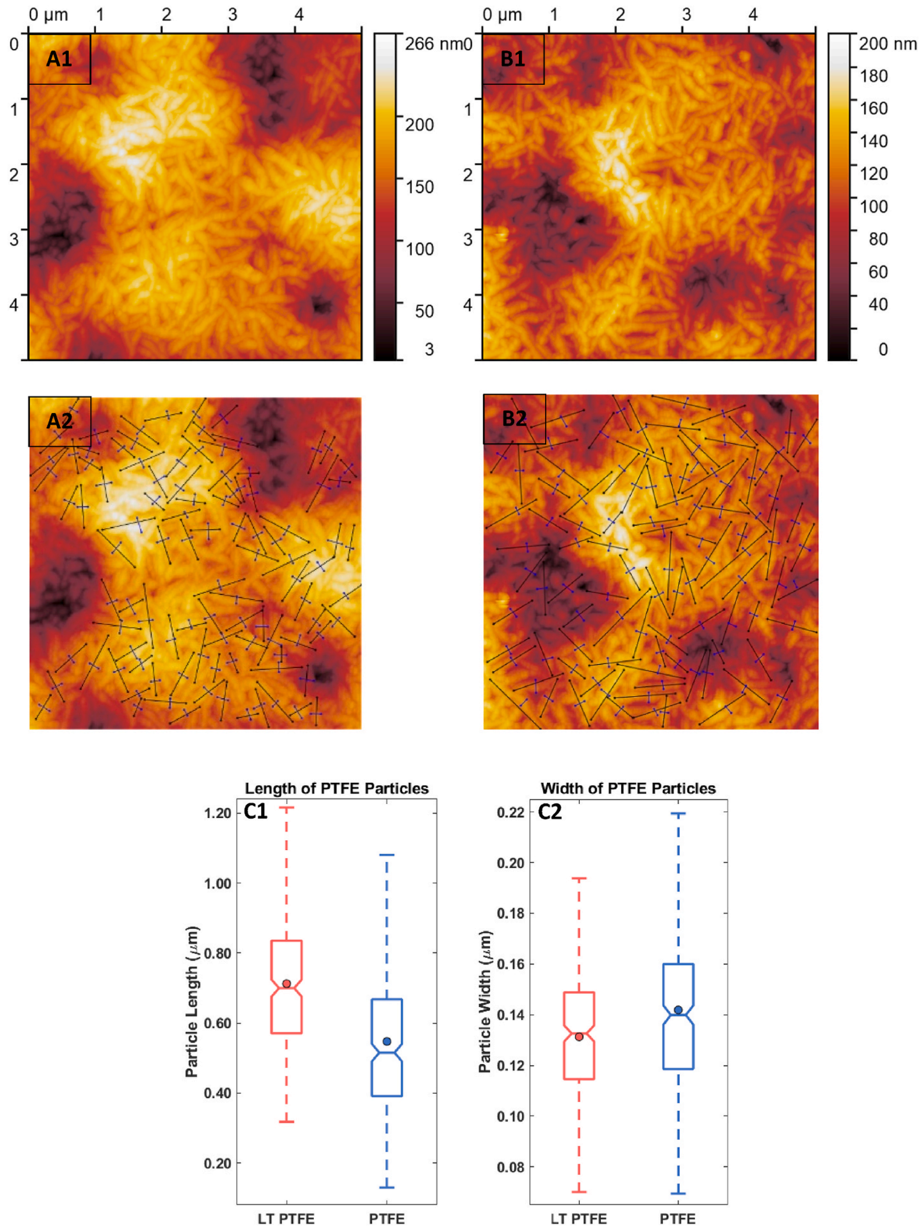

Fig. 4. AFM images of PTFE and LT PTFE coatings and PTFE particle size analysis: A1 and B1) AFM images and A2 and B2) AFM images with labeled particle lengths and widths of the PTFE and LT PTFE coatings, respectively, and boxplots of particle C1) length and C2) width of the PTFE and LT PTFE coatings, respectively.

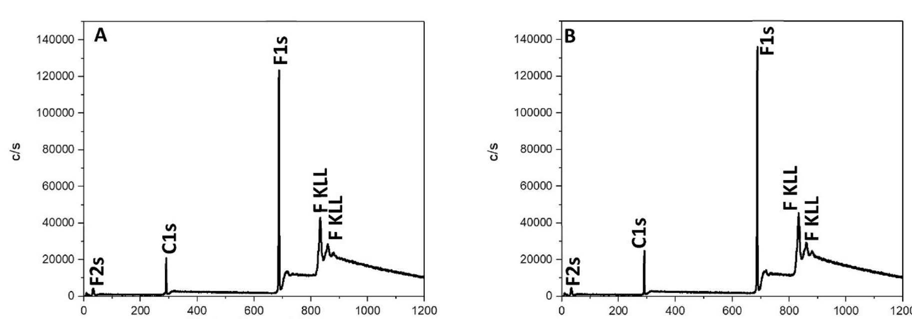

Fig. 5. XPS spextra of PTFE and laser textured PTFE coatings: A) SS/PDA/PTFE and B) SS/PDA/LT PTFE.

laser-textured PDA/PTFE coatings on smooth and laser-textured stainless steel substrates were used as the plates. Fig. 1 shows the test setup and optical images of the counterface ball. The tests were run with 2 N normal loads, 5 mm stroke length, and 10 mm/s sliding speed. Friction forces greater than 0.65 was set to be the failure criteria and the temperature and humidity were kept constant at 25 °C and 25%,

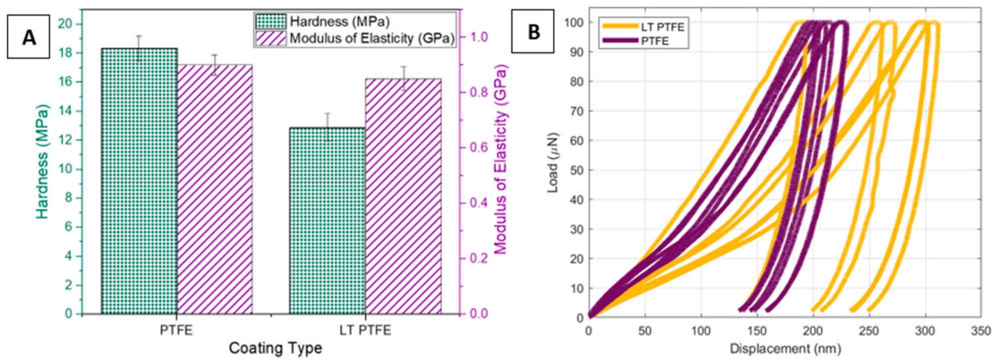

Fig. 6. Hardness, modulus of elasticity, and load-displacement curves from indentations on SS/PDA/PTFE and SS/PDA/LT PTFE coatings: A) hardness and modulus of elasticity and B) load-displacement curves.

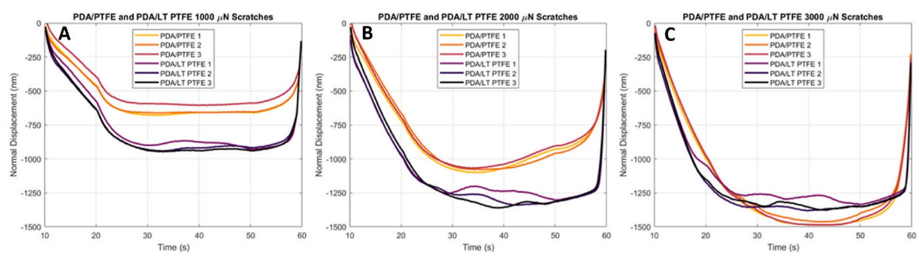

Fig. 7. Normal displacement vs. time for the nanoindenter scratches performed on SS/PDA/PTFE and SS/PDA/LT PTFE coatings using a 5 μm diamond indenter: A) 1000 μN normal load, B) 2000 μN normal load, and C) 3000 μN normal load.

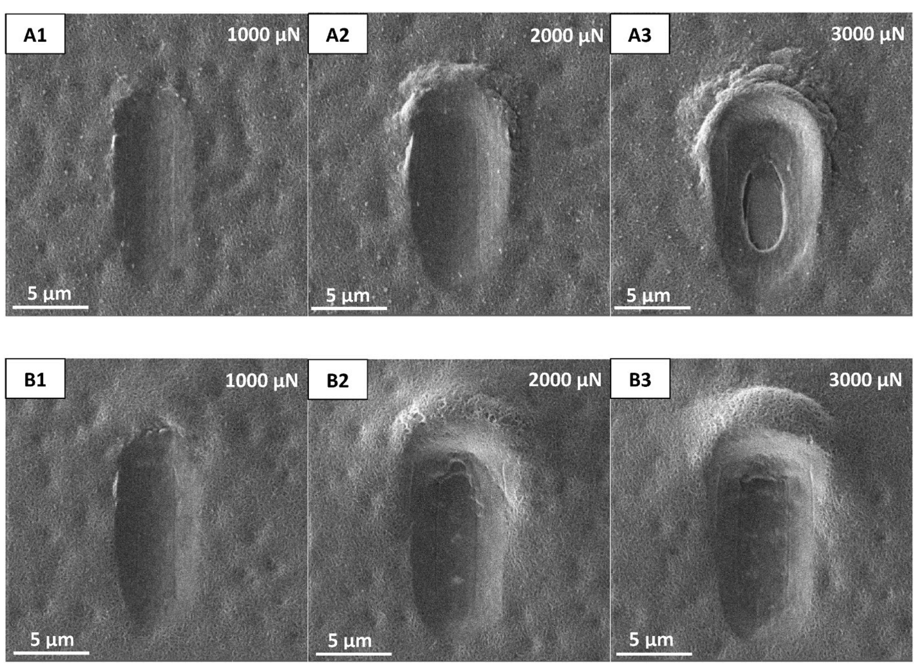

Fig. 8. SEM images of the nanoindenter scratches performed on A) SS/PDA/PTFE and B) SS/PDA/LT PTFE coatings with: A1, B1) 1000 μN normal load, A2, B2) 2000 μN normal load, and A3, B3) 3000 μN normal load.

respectively.

4. Results and discussions

4.1. Coating surface topography and roughness

Four types of samples, i.e., smooth and laser-textured PTFE coatings on smooth and laser-textured stainless steel substrates, were fabricated and labeled as shown in Table 1. Fig. 2 shows the schematics of sample types in Table 1. Hilbert curve patterns were laser-textured on stainless steel substrates and PTFE coatings. Laser power of 15% of the 2.3-W full laser power was used for laser-texturing the hard stainless steel substrates. This laser parameter resulted in textured grooves with 1.27 $\pm$ 0.4 μm depth and 5.0 $\pm$ 0.6 μm width on the substrates. The average surface roughness of smooth stainless steel and laser-textured stainless steel was measured as 0.08 $\pm$ 0.01 μm and 0.17 $\pm$ 0.06 μm, respectively.

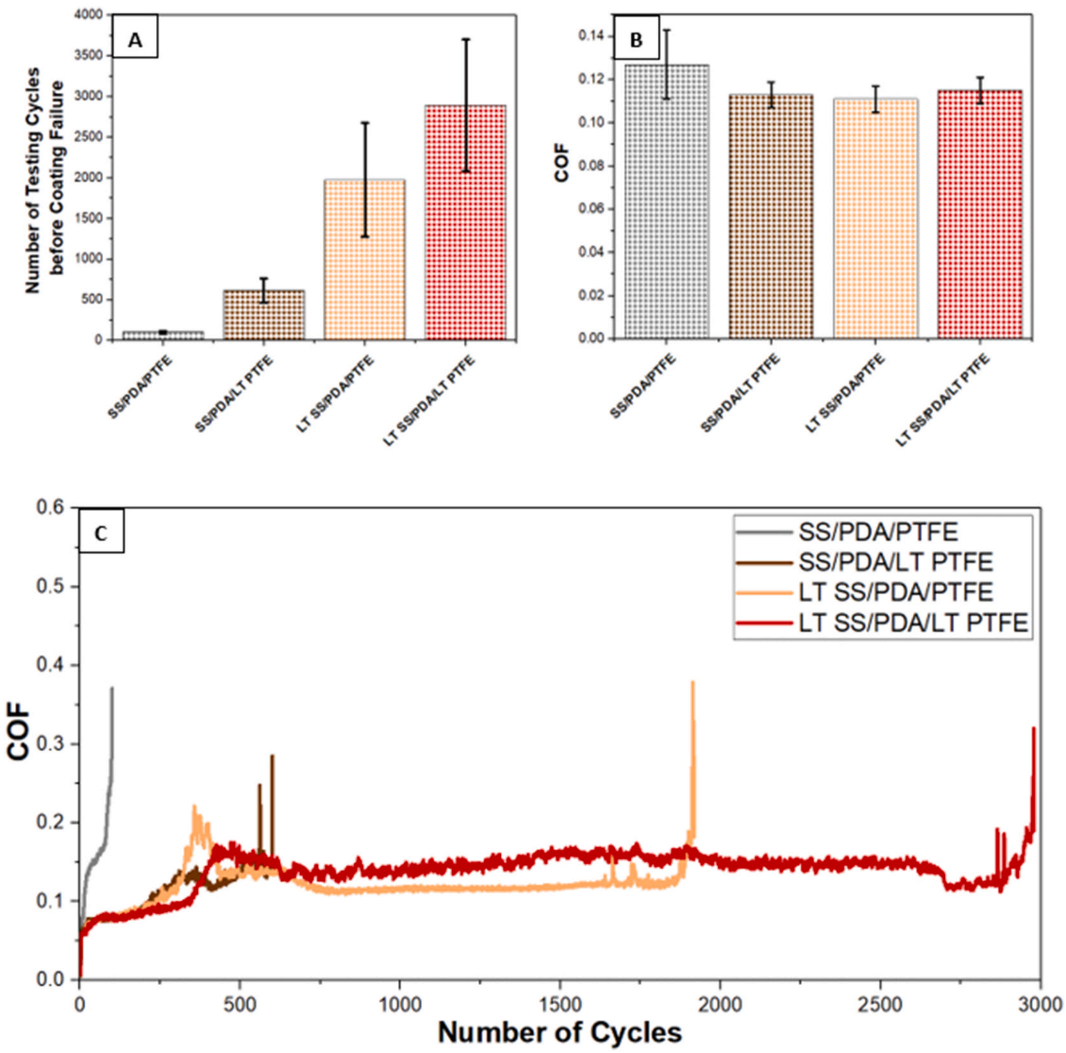

Fig. 9. Durability and COF of smooth and laser-textured PTFE coatings with PDA underlayer on smooth and laser-textured stainless steel substrates: A) Number of testing cycles before coating failure, B) average COF over the test duration, and C) representative COF as a function of the number of testing cycles during the durability tests.

To avoid affecting the spindle-like structure of PTFE nanoparticles, a very low laser power of 5% of the 2.3-W full laser power was used for laser-texturing the PTFE coating. Fig. 3 illustrates the AFM images of the surface topography of PTFE coatings of the four sample types. The surface profile of the PTFE coating topography at the horizontal line in each AFM image is plotted in Fig. 3E. The average surface roughness of PTFE coatings of the SS/PDA/PTFE, LT SS/PDA/PTFE, SS/PDA/LT PTFE, and LT SS/PDA/LT PTFE samples measured from the AFM images was 40.85 nm, 105.44 nm, 73.35 nm, and 111.17 nm, respectively. As expected, the roughness was higher for the samples with the substrate textured.

It is interesting to observe that the morphology of the laser-textured PTFE coating (Fig. 3B) is quite different from that of the PTFE coating on the laser-textured substrate (Fig. 3C). As shown in Fig. 3E, the PTFE along the Hilbert curve on the SS/PDA/LT PTFE sample (lighter color in Fig. 3B) was about 120 nm taller than the remaining PTFE areas that were not affected by the laser. In contrast, the PTFE over the Hilbert curve patterned on the LT SS/PDA/PTFE substrate was deeper (darker color in Fig. 3C). This is due to the texture grooves on the surface of stainless steel substrates. PDA and PTFE would deposit inside the grooves resulting in a deeper surface topography than the areas outside the grooves [11]. However, it should be noted that the surface profile was less than 300 nm deep compared to the measured depth of 1.27 μm for texture grooves on stainless steel. This is due to the existence of PTFE inside the texture grooves [11]. On the other hand, on SS/PDA/LT PTFE sample, the PTFE coating was bumped up about 100 nm in the areas affected by laser energy.

Also, it can be seen that when both stainless steel substrate and PTFE were laser-textured (Fig. 3D), the surface profile depth was decreased to 200 nm (Fig. 3E) because of the combined effects of the texture grooves on the substrate and the raising up of the coating by the laser texturing of the coating. Although the texture patterns on the PTFE coatings were not completely lined up with those on the stainless steel substrate because the latter was covered by the PDA/PTFE coatings, comparing Fig. 3D with Fig. 3B and C suggests that the texture patterns on PTFE coatings overlapped the texture pattern on the stainless steel substrates since the texture pattern in Fig. 3D does not have a clear and sharp border like the texture pattern in Fig. 3B and C. As a result, overlapping the texture patterns on the PTFE coating with that on the stainless steel substrate decreased the overall depth of surface profile on LT SS/PDA/LT PTFE sample compared to that on LT SS/PDA/PTFE (Fig. 3E).

Fig. 4 shows representative AFM images of the PTFE and LT PTFE coating surfaces and particle size analysis results based on three AFM images of each coating type. The results were obtained using a custom MATLAB script that allowed the user to measure the length and width of 100 PTFE particles in each image (Fig. 4A2 and 4B2). All measured data were collected in the boxplots in Fig. 4C1 and 4C2. Statistical difference

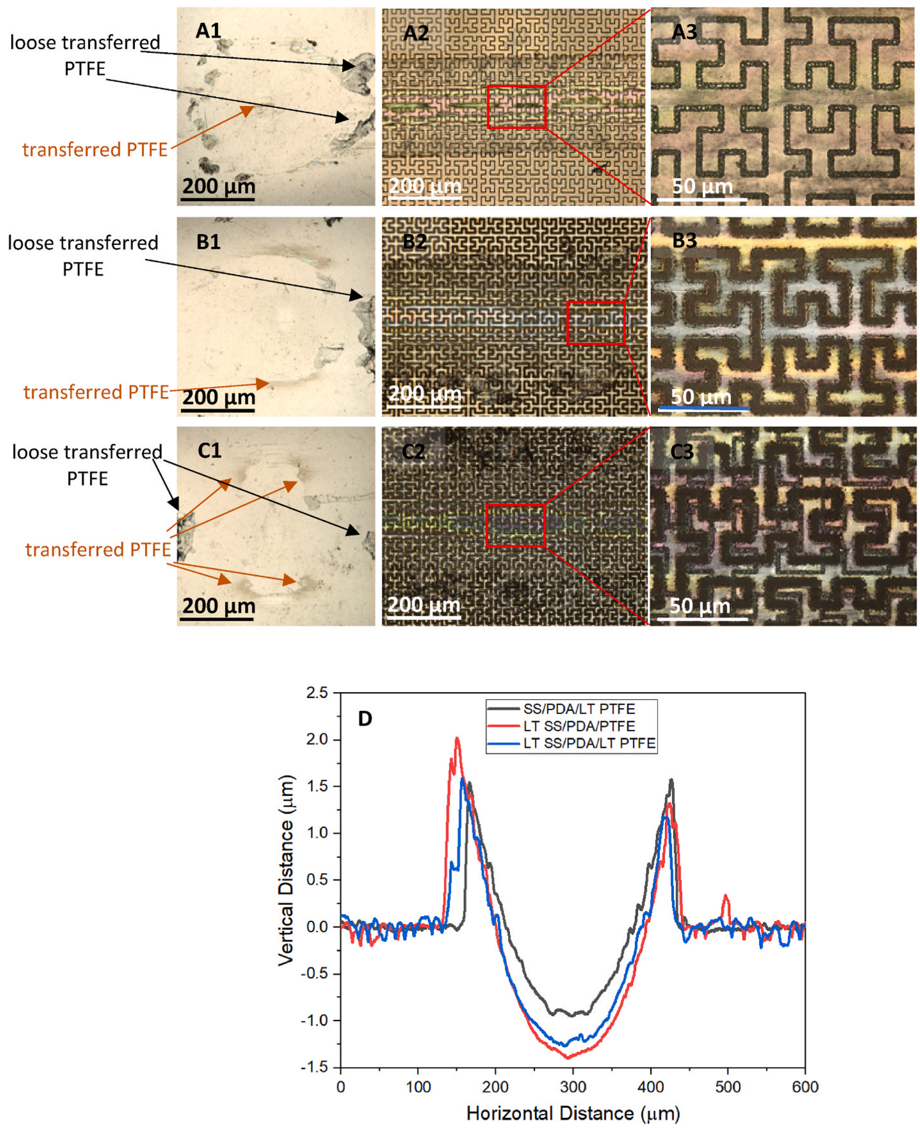

Fig. 10. Optical images of the counterface balls and wear tracks after 400-cycle progressive tests for: A) SS/PDA/LT PTFE, B) LT SS/PDA/PTFE, C) LT SS/PDA/LT PTFE (A3-C3: Zoom-in images of the red boxes on A2-C2), and D) profile of the wear tracks shown in A-C. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

between each dataset was calculated using a two-tailed paired *t*-test of each length and width data pair. The means of both length and width were found to be significantly different with a $p < 0.0001$. As shown in Fig. 4C1, and 4C2, the average lengths of the spindle-like PTFE particles on the laser-textured area were longer while the average widths were narrower. This could be due to the heat from the laser beam. Applying heat on PDA/PTFE coating increases the length of spindle-like PTFE particles and combines them to form a cross-network structure [21]. The networks formed by longer PTFE particles required more space than those formed by shorter PTFE particles. Therefore, they protruded and bumped up compared to the coating not affected by the laser beam. That is why the line profile of LT PTFE in Fig. 3E was higher than that of the neighboring areas.

4.2. *The effect of laser texturing on the coating chemistry*

XPS analysis was performed on SS/PDA/PTFE and SS/PDA/LT PTFE samples to investigate the effect of laser-texturing on the surface chemistry of PTFE. As shown in Fig. 5, both samples had the same photoelectron energy peaks meaning that the surface material on both PTFE and LT PTFE coatings had the same elemental and chemical composition. Hence, it could be concluded that laser-texturing the PTFE coating did not affect its chemical bonds because of the very low laser power used.

4.3. *The effect of laser texturing on the mechanical properties and scratch resistance of the coatings*

To further investigate the effect of laser-texturing on the PTFE coating, the mechanical properties of PTFE and laser-textured PTFE were measured through nanoindentation. As shown in Fig. 6A, laser-texturing significantly reduced the hardness but did not significantly change the modulus of elasticity of PTFE coating. Fig. 6B shows the load-displacement curves of five indentations performed on PTFE and LT PTFE. LT PTFE deformed more easily than PTFE under loading, and greater plastic deformation was observed for LT PTFE after unloading. It can be concluded that laser-texturing the PTFE coating made it less resistant to plastic deformation. The variations in the Young's modulus and hardness of the coatings were caused by the porous nature of the coatings.

Scratch tests were performed on PTFE coatings of SS/PDA/PTFE and SS/PDA/LT PTFE samples using a 5-µm-radius diamond indenter at

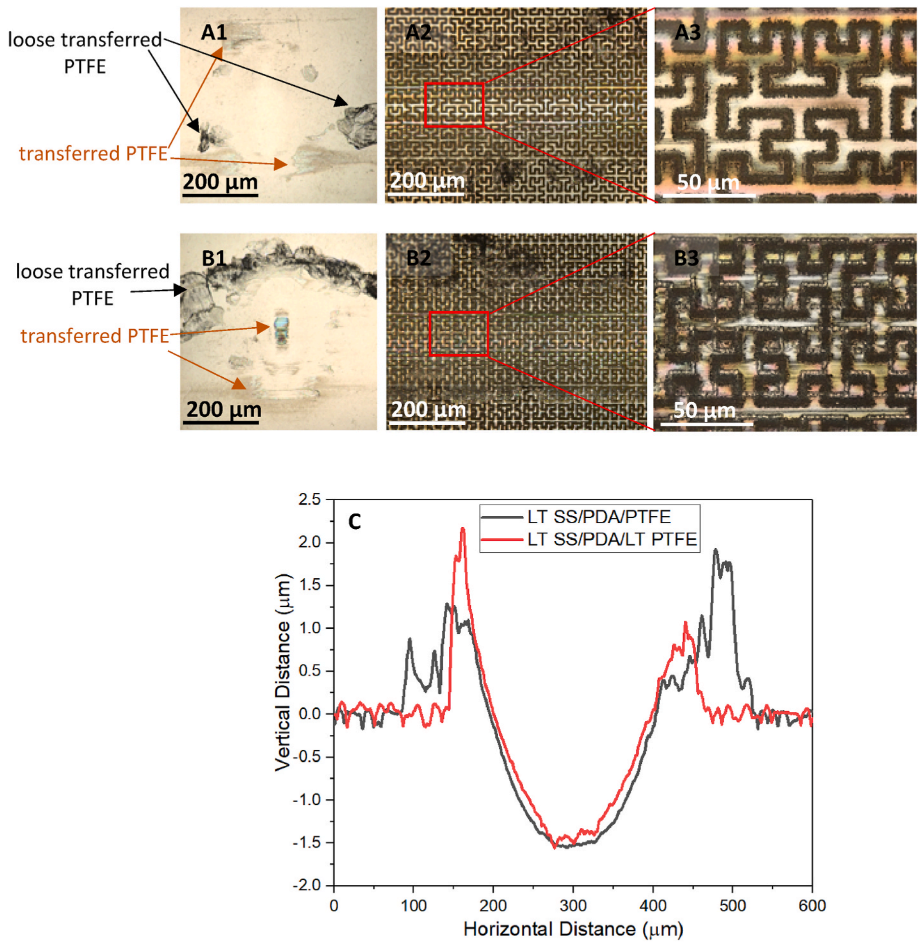

Fig. 11. Optical images of the counterface balls and wear tracks after 900-cycle progressive tests for: A) LT SS/PDA/PTFE, B) LT SS/PDA/LT PTFE (A3-B3: Zoom-in images of the red boxes on A2-B2), and C) profile of the wear tracks shown in A and B. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

normal loads of 1000 μN, 2000 μN, and 3000 μN. Fig. 7 shows the displacement curves during scratch. In Fig. 7, the first 10 s of the plots (10 s–20 s) correspond to the loading period where the normal load was applied at constant rates of 100, 200, and 300 μN/s and the tip did not move laterally. From t = 20 s to t = 50 s, the constant normal load of 1000 μN, 2000 μN, or 3000 μN was held, and the tip scratched on the surface at a constant velocity of 0.27 μm/s and for a length of 8 μm. From t = 50 s to t = 60 s, the load was reduced to zero with a constant unloading rate equal to the loading rate. As shown in Fig. 7A and B, the displacement of the LT PTFE coating on the SS/PDA/LT PTFE sample was larger than the displacement of the PTFE coating on the SS/PDA/PTFE sample at 1000 μN and 2000 μN loads. As discussed before, the LT PTFE coating was softer than the PTFE coating and hence it could be displaced easier during loading. The reduced hardness of the LT PTFE coating enabled it to be compacted more effectively than the PTFE coating. However, the LT PTFE coating showed less displacement than the PTFE coating throughout the scratches at 3000 μN load (Fig. 7C, t = 20 s to t = 50 s). This can be explained from the SEM images of the coatings after scratch in Fig. 8.

Fig. 8A3 and 8B3 show that, at 3000 μN scratch load, the PTFE coating on the SS/PDA/PTFE sample failed, but the LT PTFE coating on the SS/PDA/LT PTFE sample did not. During the 3000 μN scratches on the PTFE coating (Fig. 8A3), the indenter tip tore the coating and exposed the stainless steel substrate, thus resulting in a larger displacement than that of the SS/PDA/LT PTFE sample, as shown in Fig. 7C. In contrast, the improved compaction of the LT PTFE coating on the SS/PDA/LT PTFE sample enabled a resilient layer of coating near the substrate to withstand these higher-load scratches and prevented contact between the tip and stainless steel substrate (Fig. 8B3). This enhanced scratch resistance of the LT PTFE coating was attributed to the better-compactable PTFE nanostructures that result from laser texturing. The compaction of PTFE has previously been shown to improve its tribological performance during nanoindenter scratch testing [24].

4.4. The effect of laser texturing on the coating tribological properties

Fig. 9 shows the tribological test results. It can be seen from Fig. 9A that the PTFE coatings without laser texturing on the smooth substrate had the shortest wear life of 101 cycles. Laser texturing the PTFE coating and the substrate increased the coating wear life to 614 and 1977 cycles, respectively. Notably, laser texturing both the coating and the substrate increased the coating wear life to about 2900 cycles, which is 29 times that of the untextured PTFE coating on the smooth substrate. These improvements can be explained by surface roughness caused by laser texturing. Laser texturing the PTFE coating slightly increased the coating surface roughness and thickness on the Hilbert curves. This led to more compaction of the coating along the Hilbert curves and thus increased coating durability. It should be noted that compaction played a major role in forming a durable coating that is more resistant to wear regardless of the initial coating hardness. The lower initial hardness caused the LT PTFE coating to deform more easily initially and compacted more due to being taller than the neighboring untextured areas, which led to better wear resistance. Also, the heat from the laser beam

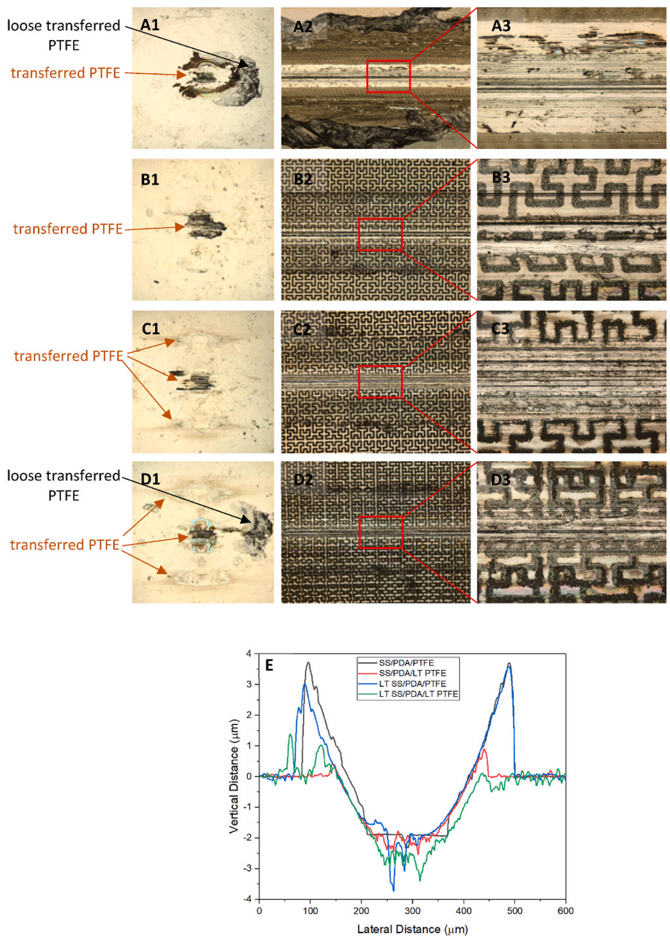

Fig. 12. Optical images of the counterface balls and wear tracks after test failure for: A) SS/PDA/PTFE, B) SS/PDA/LT PTFE, C) LT SS/PDA/PTFE, D) LT SS/PDA/LT PTFE (A3-D3: Zoom-in images of the red boxes on A2-D2), and E) profile of the wear tracks shown in A-D. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

elongated the PTFE nanoparticles and improved their network connectivity, resulting in a more cohesive coating. Therefore, the LT PTFE coating was more resistant to wear.

Laser-texturing the substrate has been shown to provide a reservoir for the PTFE lubricant and helped recover the tests from failure [11]. The roughness created by the textures on the substrate also provided interlocking to the PTFE coating and thus prevented coating global delamination [11].

Fig. 9B illustrates that the samples with a laser-textured substrate, laser-textured PTFE, or both had a slightly lower COF than the samples without laser texturing (SS/PDA/PTFE). The representative COF graphs of each sample type are presented in Fig. 9C. The sharp increase at the end of each graph represents the coating failure. There was an increase in the COF of the SS/PDA/PTFE sample immediately after the test began and well in advance of when the coating failed with a sharp COF increase. In comparison, this COF increase was delayed for samples with either laser-textured PTFE or stainless steel substrate, which happened around 300 cycles. This increase in the COF was due to compacting loosely connected PTFE particles by the counterface ball [11]. Since laser-textured samples had rougher surfaces, they experienced this increase later than the SS/PDA/PTFE sample.

Figs. 10–12 show the optical images of counterface ball and wear tracks after 400-cycle progressive tests, 900-cycle progressive tests, and tests conducted to coating failure, respectively. On the SS/PDA/LT PTFE sample, the PTFE coating was barely rubbed off after 400 cycles and it was compacted by the counterface ball since LT PTFE was soft. Hence, the laser-textured pattern was not rubbed off and there was some loose transferred PTFE on the counterface ball and only a slight amount of transferred PTFE at the center of the ball (Fig. 10A). This sample failed after 614 cycles at which the laser-textured pattern was rubbed off and a large amount of PTFE was transferred to the center of the counterface ball (Fig. 12B). Also, there was no loose transferred PTFE on the counterface ball after test failure since the loose material might have been transferred back to the wear track at some point during the test.

For the LT SS/PDA/PTFE sample, after 400 cycles, the PTFE coating was plowed from the center to the sides of the wear track and the substrate was slightly exposed at the center of the wear track (Fig. 10B2). This led to a slight increase in the COF, as shown in Fig. 9C. However, the test did not fail because PTFE stored inside the texture grooves replenished the wear track and brought the COF back to a low level. This can be seen in the image of the wear track after 900 cycles (Fig. 11A). This sample failed after 1977 cycles of testing when all PTFE stored inside the texture grooves were rubbed off and transferred to the counterface ball (Fig. 12C).

A combination of the phenomena observed from the SS/PDA/LT PTFE sample and the LT SS/PDA/PTFE sample was observed for the LT SS/PDA/LT PTFE sample after 400 cycles. PTFE was compacted and plowed to the sides of the wear track which slightly diminished the laser-textured pattern on PTFE. Therefore, no obvious PTFE was transferred to the center of the counterface ball, however, there was some loose transferred PTFE surrounding the contact area of the counterface ball. These loose PTFE might transfer back to the wear track later during the test to repair the wear (Fig. 10C). After 900 cycles, the laser-textured pattern of the PTFE was less visible and some PTFE was transferred to the center of the counterface ball. The substrate was almost exposed but the texture grooves on the substrate were still filled with PTFE (Fig. 11B) that would replenish the wear track and recover the test (Fig. 9C). Also, some loose PTFE materials were transferred to the counterface ball (Fig. 11B1) due to the counterface scratched part of the soft laser-textured PTFE not interlocked in the texture grooves. This sample failed after 2893 cycles at which point the laser-textured patterns on both stainless steel substrate and PTFE were completely removed in the wear track, and the PTFE was rubbed off and transferred to the counterface ball (Fig. 12D). It should be noted that although this sample ran much more number of cycles than other samples, the width of the wear track on this sample is the same as on SS/PDA/LT PTFE and LT SS/PDA/PTFE samples. This shows the effectiveness of Hilbert curve textured grooves in preventing coating wear and global delamination.

The profiles of the wear tracks in Figs. 10–12 illustrate that the depth of the wear tracks increased as the number of tribology test cycles increased. Also, Fig. 12A indicates global delamination of the SS/PDA/PTFE sample, and unlike the textured samples, a large amount of loose PTFE was transferred to the counterface ball (Fig. 12A1).

Fig. 13 demonstrates the SEM images and EDS elemental maps of the wear tracks after tribology tests to failures. As presented in the EDS maps of iron, Fe, and fluorine, F, the wear track on SS/PDA/PTFE sample (where the fluorine from the coating is mostly removed) had the largest width (Fig. 13A). This data confirms the wide and flat wear track profile in Fig. 12E that represented the global delamination of the coating inside the wear track. It can be observed that laser texturing stainless steel substrate and/or PTFE coating significantly decreased the wear track width and prevented the coating's global delamination despite their longer testing duration (Fig. 13B–D). Laser texturing reduced the wear track width by protruding (for LT PTFE, Fig. 13B and D) and/or recessing (for LT SS, Fig. 13C and D) the surface. The protruded laser-textured areas of PTFE (arrow positions in Fig. 13B and D) resulted in a higher contact pressure and increased the compaction in these areas, which led to more coherent coating. The recessed laser-textured areas of SS (arrow positions in Fig. 13C and D) increased the surface roughness and interlocking between the coating and the substrate. They also stored PTFE that replenished and recovered the removed PTFE above the

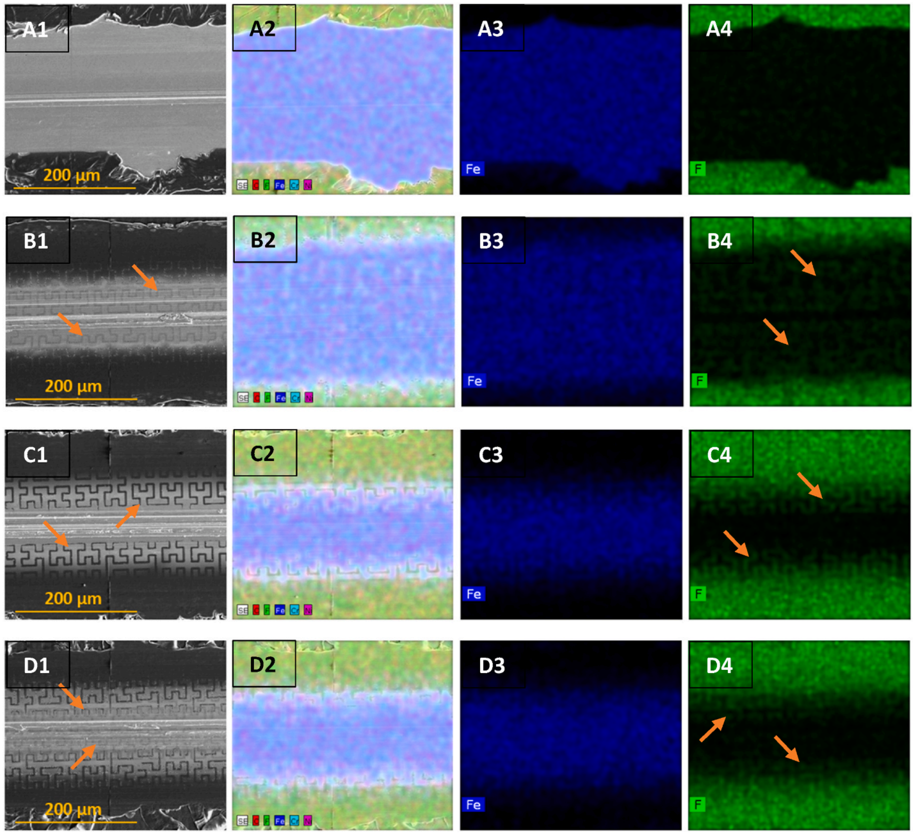

Fig. 13. SEM images (A1-D1), EDS elemental maps (A2-D2), EDS map of iron (A3-D3), and EDS map of fluorine (A4-D4) of wear tracks after test failure for: A) SS/PDA/PTFE (113 cycles), B) SS/PDA/LT PTFE (601 cycles), C) LT SS/PDA/PTFE (1918 cycles), and D) LT SS/PDA/LT PTFE (2809 cycles).

grooves within the wear track. It can be seen that laser texturing the stainless steel substrate resulted in a narrower wear track after testing more cycles than laser texturing the PTFE coating. Therefore, it was more effective than laser texturing the PTFE coating.

5. Conclusion

Stainless steel substrates and PTFE thin coatings were laser-textured with 15% and 5% of the 2.3-W full laser power, respectively. The coating surfaces were characterized and the mechanical and tribological behaviors of the smooth and laser-textured PTFE on smooth and laser-textured stainless steel were studied. It was shown that laser-texturing both the substrate and the PTFE increased the surface roughness of the PTFE coating. Laser-texturing PTFE bumped up the coating and made the coating softer without changing its chemical bonds. This resulted in a smaller hardness of the laser-textured PTFE compared to smooth PTFE. Consequently, laser-textured PTFE was compacted more than smooth PTFE at various loads during nanoscratch tests. Also, it was shown that better compactable laser-textured PTFE coating had better coating cohesion during the nanoscratch tests at higher loads, while PTFE without laser texture was torn by the indenter tip. The tribology test results showed that laser-texturing the PTFE coating or the stainless steel substrate improved the wear life of the PDA/PTFE coating over 6 times and 19 times, respectively, while laser-texturing both improved the wear life of the PDA/PTFE coating about 29 times. Also, laser-texturing prevented PDA/PTFE coatings from global delamination.

Author statement

Firuze Soltani-Kordshuli: Conceptualization, Writing-original draft, Methodology, Validation, Formal Analysis, Investigation, Visualization. **Charles Miller:** Conceptualization, Writing-review & editing, Investigation, Visualization. **Nathaniel Harris:** Conceptualization, Writing-review & editing, Formal Analysis, Visualization. **Min Zou:** Writing-Review & Editing, Methodology, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

[1] H. Unal, A. Mimaroglu, U. Kadıoglu, H. Ekiz, Sliding friction and wear behaviour of polytetrafluoroethylene and its composites under dry conditions, Mater. Des. 25 (3) (2004) 239–245.

[2] D.L. Burris, W.G. Sawyer, Improved wear resistance in alumina-PTFE nanocomposites with irregular shaped nanoparticles, Wear 260 (7–8) (2006) 915–918.

[3] A. Riveiro, et al., Influence of laser texturing on the wettability of PTFE, Appl. Surf. Sci. 515 (2020), 145984.

[4] F. Soltani-Kordshuli, et al., Tribological behavior of the PDA/PTFE+ Cu-SiO2 nanoparticle thin coatings, Surf. Coating. Technol. 409 (2021), 126852.

[5] H. Fan, Y. Su, J. Song, H. Wan, L. Hu, Y. Zhang, Design of "double layer" texture to obtain superhydrophobic and high wear-resistant PTFE coatings on the surface of Al2O3/Ni layered ceramics, Tribol. Int. 136 (2019) 455–461.

[6] S. Beckford, Y. Wang, M. Zou, Wear-resistant PTFE/SiO2 nanoparticle composite films, Tribol. Trans. 54 (6) (2011) 849–858.

[7] I. Etsion, State of the art in laser surface texturing, J. Tribol. 127 (1) (2005) 248–253.

[8] L. Rapoport, et al., Friction and wear of MoS2 films on laser textured steel surfaces, Surf. Coating. Technol. 202 (14) (2008) 3332–3340.

[9] C.G. Guleryuz, J.E. Krzanowski, Mechanisms of self-lubrication in patterned TiN coatings containing solid lubricant microreservoirs, Surf. Coating. Technol. 204 (15) (2010) 2392–2399.

[10] A. Voevodin, J. Zabinski, Laser surface texturing for adaptive solid lubrication, Wear 261 (11–12) (2006) 1285–1292.

[11] F. Soltani-Kordshuli, N. Harris, M. Zou, Tribological behavior of polydopamine/polytetrafluoroethylene coating on laser textured stainless steel with Hilbert curves, Friction (2022), https://doi.org/10.1007/s40544-022-0671-0. Research Article *In Press*.

[12] C. Miller, D. Choudhury, M. Zou, The effects of surface roughness on the durability of polydopamine/PTFE solid lubricant coatings on NiTiNOL 60, Tribol. Trans. 62 (5) (2019) 919–929.

[13] W. Li, Q. Yang, F. Chen, J. Yong, Y. Fang, J. Huo, Femtosecond laser ablated durable superhydrophobic PTFE sheet for oil/water separation, in: Second International Conference on Photonics and Optical Engineering, vol. 10256, International Society for Optics and Photonics, 2017, p. 102563O.

[14] S.F. Toosi, S. Moradi, S. Kamal, S.G. Hatzikiriakos, Superhydrophobic laser ablated PTFE substrates, Appl. Surf. Sci. 349 (2015) 715–723.

[15] W. Fan, J. Qian, F. Bai, Y. Li, C. Wang, Q.-Z. Zhao, A facile method to fabricate superamphiphobic polytetrafluoroethylene surface by femtosecond laser pulses, Chem. Phys. Lett. 644 (2016) 261–266.

[16] F. Liang, J. Lehr, L. Danielczak, R. Leask, A.-M. Kietzig, Robust non-wetting PTFE surfaces by femtosecond laser machining, Int. J. Mol. Sci. 15 (8) (2014) 13681–13696.

[17] A.-M. Kietzig, J. Lehr, L. Matus, F. Liang, Laser-induced patterns on metals and polymers for biomimetic surface engineering, in: Laser Applications in Microelectronic and Optoelectronic Manufacturing (LAMOM) XIX, vol. 8967, SPIE, 2014, pp. 9–16.

[18] K.T. Ahmmed, C. Patience, A.-M. Kietzig, Internal and external flow over laser-textured superhydrophobic polytetrafluoroethylene (PTFE), ACS Appl. Mater. Interfaces 8 (40) (2016) 27411–27419.

[19] A. Riveiro, et al., Laser texturing to control the wettability of materials, Procedia CIRP 94 (2020) 879–884.

[20] S. Beckford, M. Zou, Wear resistant PTFE thin film enabled by a polydopamine adhesive layer, Appl. Surf. Sci. 292 (2014) 350–356.

[21] Y. Jiang, D. Choudhury, M. Brownell, A. Nair, J.A. Goss, M. Zou, The effects of annealing conditions on the wear of PDA/PTFE coatings, Appl. Surf. Sci. 481 (2019) 723–735.

[22] Y. Zhao, M. Zou, Experimental investigation of the wear mechanisms of thin PDA/PTFE coatings, Prog. Org. Coating 137 (2019), 105341.

[23] A. Abe, D. Choudhury, M. Zou, Improved tribological performance of PDA/PTFE thin coatings with silica nanoparticles incorporated into the PDA underlayer, J. Tribol. (2021) 1–22.

[24] C. Miller, M. Zou, Microscale friction and deformation behavior of polydopamine/polytetrafluoroethylene-coated 60NiTi from nanoscratch tests, Thin Solid Films 743 (2022), 139079.