

Tribologically superior PTFE- and ATSP-based polymer composites for hydrogen environment applications

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Abstract

The global motivation for clean hydrogen energy has grown due to the need for additional sustainable energy sources. To address this demand, it is essential to minimize hydrogen loss during its production and transportation. Polymers are increasingly used to replace metals in components such as seals, valves, and gaskets. This transition is motivated by their superior tribological properties, corrosion resistance, and ability to operate without external lubrication.

However, despite their excellent performance in air, these polymers have not been thoroughly tested in hydrogen environments. In this study, several polymer composites were developed using a vitrimer aromatic thermosetting copolyester (ATSP) and polytetrafluoroethylene (PTFE) matrices, and their tribological performance was evaluated in a specially designed tribometer under a 5% hydrogen atmosphere. Fluorine-rich transfer films were detected on the steel counterface, and the ATSP-matrix composite exhibited the lowest wear. These findings identify the materials studied as promising candidates for hydrogen-related tribological components. However, they should not be extrapolated directly to high-purity hydrogen, high pressure, elevated temperature, cyclic decompression, or long-duration service without additional validation.

Keywords: Polymer Composites; ATSP; PTFE; Hydrogen Tribology

1. Introduction

Hydrogen-based systems were developed to reduce carbon emissions, promote sustainability, and address energy-related challenges [1]. Hydrogen (H_2) offers significant advantages over conventional energy carriers like electricity, primarily due to its ability to store and deliver substantial amounts of energy and its ease of conversion to other energy forms [2]. As a transportation fuel, particularly in fuel cell vehicles (FCVs), hydrogen addresses key issues of fossil fuel-based vehicle technologies, such as reducing greenhouse gas and pollutant emissions, promoting energy independence, diversifying fuel sources, and enhancing onboard fuel efficiency, which is three times greater than that of petroleum [3]. Additionally, being the most abundant element in the universe and the richest in energy per unit mass, hydrogen has numerous applications beyond road vehicles, such as aeronautics, space, industrial, and domestic energy needs, backup energy solutions, and hydrogen village concepts [4, 5].

Renewable energy sources such as solar, wind, hydro, tidal, geothermal, and biomass are highly weather-dependent, and their intermittent nature can lead to supply-demand mismatches when connected directly to the electric grid. Therefore, storing renewable energy such as hydrogen is emerging as a key solution. Hydrogen can be generated by electrolyzers and stored in various forms based on the end use. The development of hydrogen as a cost-effective, large-scale, and long-term energy storage method encourages the growth of renewable energy industries [6, 7]. Hydrogen has the potential to become a mainstream energy carrier, but to be practical and competitive, it requires a highly reliable and safe system for delivery, storage, and end-use.

The effectiveness of these steps relies heavily on technological advancements and material development, which are crucial for establishing a viable hydrogen economy. This underscores the importance of a multi-material approach for components involved in hydrogen distribution, storage, and dispensing infrastructure to create a cost-effective and safe working environment [8, 9]. Due to its physical properties, H_2 experiences high pressure and high temperature, which pose challenges for automobile, aerospace, and agricultural applications, where external lubrication of different tribopairs is not feasible [10]. Therefore, high-performance polymers (HPPs) are appealing due to their ability to withstand extreme pressures and temperatures [11, 12, 13]. Polytetrafluoroethylene (PTFE), Polyether ether ketone (PEEK), elastomers, and related polymers are already used in seals, gaskets, valve seats, liners, and compressor components for hydrogen systems; however, despite their good performance in air, the tribological behavior of PTFE- and Aromatic thermosetting copolyester (ATSP)-based composites in hydrogen-containing environments remains poorly understood. Previous hydrogen tribology studies have focused mainly on neat or filled PTFE under high-pressure hydrogen, while ATSP-based vitrimer composites have not been systematically compared with PTFE-matrix composites under controlled

hydrogen-containing atmospheres. The revised gap statement emphasizes three limitations in the literature: material-design limitations, environmental adaptability limitations, and insufficient tribochemical characterization of transfer films formed during sliding in hydrogen-containing environments.

Hydrogen can be transported in liquid or gaseous form by pipelines or cylinders, or by mixing it with compounds such as ammonia, methanol, or organic mixtures. Synthesizing ammonia from hydrogen at production facilities, transporting ammonia, and then cracking it to produce hydrogen at the end-user facility is a recent advancement. This process, though efficient, faces several limitations, including safety, cost, and additional processes within the supply chain [14]. To efficiently transport or store hydrogen, the mode should be safe, lightweight, compact, and sufficiently large to make it economical. Traditional storage facilities face challenges with hydrogen due to its low boiling point (-253°C), extremely low density in its gaseous state (0.09kg/m^3), and exceptionally high density in its liquid phase (70.9 kg/m^3). The risk of explosion and fire is higher with hydrogen due to its inherent characteristics, such as low minimum ignition energy and high flammability [15]. To mitigate this risk, it is necessary to prevent leakage, especially as the pressure increases. This creates a challenge in developing storage facilities that exhibit minimal or no leakage. The safety of storing high-pressure hydrogen gas in pressure vessels and distributing it through pipelines depends on the materials used. Earlier pressure vessels and pipelines were entirely made of metallic alloys, which faced challenges such as hydrogen embrittlement, caused by hydrogen atoms diffusing through metal grain boundaries. Unlike metals, which undergo hydrogen-induced diffusion, polymers are chemically inert and do not exhibit this phenomenon [16, 17, 18, 19, 20].

As a result, polymeric materials are increasingly used in applications involving direct contact with high-pressure hydrogen in storage vessels and pipelines. Recently, pressure vessels have been developed with a polymeric composite shell covered with a plastic liner (Type IV), and pipelines are now being constructed with a steel tube and a plastic liner. The liners provide the necessary gas tightness, and plastic liners are lightweight, cost-efficient, corrosion-resistant, and prevent hydrogen embrittlement compared to metallic liners. The latest Type V pressure vessel, made entirely of composites without a liner, is 20% lighter than Type IV vessels but can only handle low pressures and requires further advancements [15, 21].

Table 1 summarizes the main components used in hydrogen gas production until reaching the fuel cell of the FCV, and the most commonly used material grades for the respective function [22]. To achieve high-pressure gas storage, advancements in sealing components are needed. Elastomeric materials are commonly used in sealing components and other applications, such as control valves, flexible hoses, and connectors, that are exposed to high-pressure hydrogen during service. PTFE, PEEK, or rubber O-rings are predominantly used as sealing components and must provide high endurance and seal integrity over long durations [23, 24].

Table 1. Polymeric materials used in hydrogen applications.

Component	Description	Materials commonly used
Compressed H ₂ pressure vessels	Type IV	HDPE or Polyamide (PA) liner on glass or epoxy resin container
Piping, tubing		HDPE, Polypropylene (PP), Poly(vinyl chloride) (PVC), Chlorinated poly(vinyl chloride) (CPVC)
Compressors	Seals, coatings	PTFE, PEEK
Connectors	O-rings, gaskets	PTFE, Nitrile rubber, Fluor elastomer (FKM)
	Pistons	PEEK
Valves	Seals and gaskets	PTFE, FKM, PA, PEEK, silicone
	Valve seats	PA, PTFE, Polychlorotrifluoroethylene (PCTFE), Polyimide (PI)

Sealing components exposed to high-pressure hydrogen gas may swell, altering their dimensions and increasing contact with the housing. Additionally, changes in surface properties resulting from high-pressure hydrogen exposure can shorten the lifetime of sealings and compromise their intended gas-tightness. Valves, compressors, liners in storage tanks, and pipes, made from polymeric materials, undergo cyclic gas exposures and temperature changes, causing repeated back-and-forth motions between the polymeric and metallic surfaces [22, 25]. Sawae et al. developed a pin-on-disk tribometer for in situ testing in an autoclave under high-pressure hydrogen conditions to study sliding friction and wear. They tested PTFE disk specimens filled with graphite and bronze, exposing them to 40 MPa of high-pressure hydrogen at 100°C. The COF results showed no significant changes across different hydrogen pressures, high-pressure helium, or atmospheric conditions. However, all PTFE grades exhibited lower wear behavior when exposed to high-pressure hydrogen compared to other conditions [21, 22].

In similar tests, Nakashima et al. observed a PTFE transfer film on the metal counterpart, and XPS showed a higher peak corresponding to metal fluoride. Hence, they concluded that under high-pressure hydrogen exposure, steel counterparts do not form protective metal oxide layers. Instead, the pure metal is exposed to active PTFE radicals, which form stable metal-fluoride bonds as a transfer film. This transfer film reduces subsequent wear caused by relative motion. Further improvements in characterization techniques and in-situ tribological experimental setups are needed to gain a deeper understanding of the factors influencing the wear and friction behavior of polymers exposed to hydrogen conditions [28, 29].

A new class of materials, a vitrimer aromatic thermosetting copolyester (ATSP), has been shown to improve wear performance when used as a reinforcing additive for PTFE. Previous studies have demonstrated that incorporating ATSP into PTFE, either as a matrix or as a filler, leads to a

reduction in wear rates for engineering applications in a wide range of temperatures [26, 30, 31, 32]. Molybdenum disulfide (MoS₂) as reinforcement for PTFE and ATSP, especially around 10 wt. %, has been known to provide superior strengthening characteristics and improved tribological performance [33, 34, 35]. Carbon nanotubes (CNTs) as reinforcements (1-5%) have already been proven attractive due to their high Young's modulus, tensile strength, biocompatibility, and good tribological properties [36, 37, 38].

In this study, the effect of hydrogen on the tribological properties of PTFE-based and ATSP-based composites was evaluated. The sliding experiments are performed on four different PTFE-ATSP composites in air and in a 5%H₂-95%N₂ environment to measure their COF and wear. The wear is studied using optical microscopy, profilometry, scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS). Also, to better understand tribochemical reactions, Fourier transform infrared spectroscopy (FTIR) and X-ray Photoelectron Spectroscopy (XPS) were performed inside the wear tracks.

2. Materials and methods

2.1 Sample preparation

Three different composites with PTFE matrix, PTFE +15%ATSP, PTFE +25%ATSP, and PTFE +15%ATSP +2.5% MoS₂, and one ATSP-matrix composite, ATSP +5%PTFE +2%CNT, were fabricated for tribological testing, as shown in **Table 2**. The choice of these composites and the matrix-to-filler ratio were based on previous work and preliminary experiments. Addition of 15-25% ATSP filler to PTFE matrix has been shown to improve the wear resistance of pure PTFE by one to two orders of magnitude without a significant increase in friction for oil-less engineering applications [30]. The goal was to ensure that their performance was comparable to or exceeded that of commercially available HPPs designed for similar applications. Teflon® PTFE 7A X

(Chemours Company FC LLC) with an average particle size of 38 μm was used as a matrix material for three of the composites in this work. The ATSP powder (NOWETM VC-201) was supplied by ATSP Innovations Inc. MoS₂ particles (average particle size tenths of nm to 50 μm) and CNT (5 to 50 nm wide and a few micrometers long) were purchased from PBY Plastics. The matrix and fillers were mixed in a high-shear mixer to achieve the intended weight proportions listed in **Table 2**. The mixtures were placed in a mold to produce compression-molded parts at room temperature under 35 MPa air pressure. The molded parts had a diameter of 76.2 mm and a thickness of 18 mm. After molding, the parts were removed from the mold and placed in an oven to air-sinter at a maximum temperature of 375°C for 4 hours, following a specific protocol recommended by the manufacturer. The cooling process was controlled at a rate of 40°C per hour. The ATSP resins used were produced from cross-linkable aromatic copolyester oligomeric systems, specifically a mix of carboxylic acid- and acetoxy-end-capped oligomers, resulting in the final oligomer system. The cured resin was then ground and sieved to produce ATSP powder suitable for use as a filler, with a particle size kept below 90 μm . The composites were shaped into cylindrical pins with a diameter of 6.3 mm and length of 6.3 mm (**Fig. 1(b)**). The sliding surfaces of the pins were polished on a rotating polishing wheel with increasing grit number polishing pads using DI water to wash away debris. After the pins were polished, they were sonicated in IPA and air dried to remove any loose debris. The RMS surface roughness (R_q) of the pins was kept below 5 μm . The tribological counterpart used in this study is stainless steel (SS) 316 disks, chosen for their extensive commercial use owing to their high strength, good corrosion resistance, and chemical inertness. The chemical composition of the SS 316 is Fe (69%), Cr (16–18%), Ni (10–14%), Mb (2–3%), C (0.08%), and trace amounts of other elements. 25.4 mm (1 inch) square side disks with surface roughness (R_q) of 0.35 ± 0.06 μm are used for tribological testing. One set of

PTFE + 15% ATSP composite pins was aged in a chamber pressurized at 13.8 MPa with 5% H₂-95%N₂ for 6 hours before conducting sliding tests to measure the effect of hydrogen aging on the tribological performance. The micro-Vickers hardness (HV) values of the pin samples are presented in **Table 2**.

Table 2. Pin materials and their bulk hardness.

Pin Material	Hardness (HV)
PTFE+15%ATSP	3.50
PTFE+25%ATSP	3.73
PTFE+15%ATSP+2.5%MoS ₂	3.78
ATSP +5%PTFE +2%CNT	7.80

2.2 Tribological testing

A High-Temperature Tribometer (HTT) depicted in **Fig. 1(a)** is a highly adaptable tribometer capable of performing in-situ tribological experiments across a broad temperature range, from -200 to 1000°C, within controlled atmospheres such as N₂ and CO₂. It supports various pin-on-disk configurations, including flat pin-on-disk, ball-on-disk, and curved pin-on-disk, facilitating plane, point, and line contacts, respectively. The HTT also provides both unidirectional motion (up to 1000 rpm) and oscillatory motion (up to 5 Hz) to measure kinetic and static friction. It uses an electromagnetic force mechanism to apply a normal load of up to 45 N and records both normal force and lateral frictional force, enabling the calculation of in-situ COF. Using a vacuum pump and ports, the chamber can be evacuated and then filled with gas, such as N₂. The system features a transparent furnace. This furnace uses infrared radiation to heat the sample by reflecting nearly

all of it onto a thin, gold-coated quartz tube. Detailed information about the HTT is available in Ref. [39].

The experiments were conducted using a pin-on-disk configuration in the HTT chamber (**Fig. 1**). In this setup, a 1-inch-square SS 316 disk is secured to the bottom rotating surface of the disk holder. The counterface, a flat cylindrical pin, is mounted in a shaft on the top of the tribometer using a pin holder. The in-situ normal and friction forces were recorded during each experiment using an Analog-to-Digital converter and a LabVIEW user interface. Unidirectional experiments were conducted under unlubricated sliding conditions, atmospheric pressure, and at room temperature. A constant normal load of 32 N, equivalent to a nominal contact pressure of 1 MPa, was applied at the contact interface of the pin, and a sliding speed of 0.25 m/s was maintained for one hour. This combination of normal load and sliding speed was chosen to simulate the extreme conditions encountered by seals and O-rings in hydrogen-environment applications. Care was taken to ensure that the polymer blends did not exceed the maximum PV limit of the PTFE or ATSP composites under unlubricated testing conditions. The HTT chamber was vacuumed and filled with 5% H_2 -95% N_2 gas at atmospheric pressure for the experiments. Each experiment was repeated three times to ensure repeatability. Before each experiment, both the SS 316 samples and composite polymer pins were subjected to ultrasonic cleaning with acetone and isopropyl alcohol, respectively, for 10 minutes. Subsequently, they were rinsed using isopropyl alcohol and dried using warm air to remove impurities.

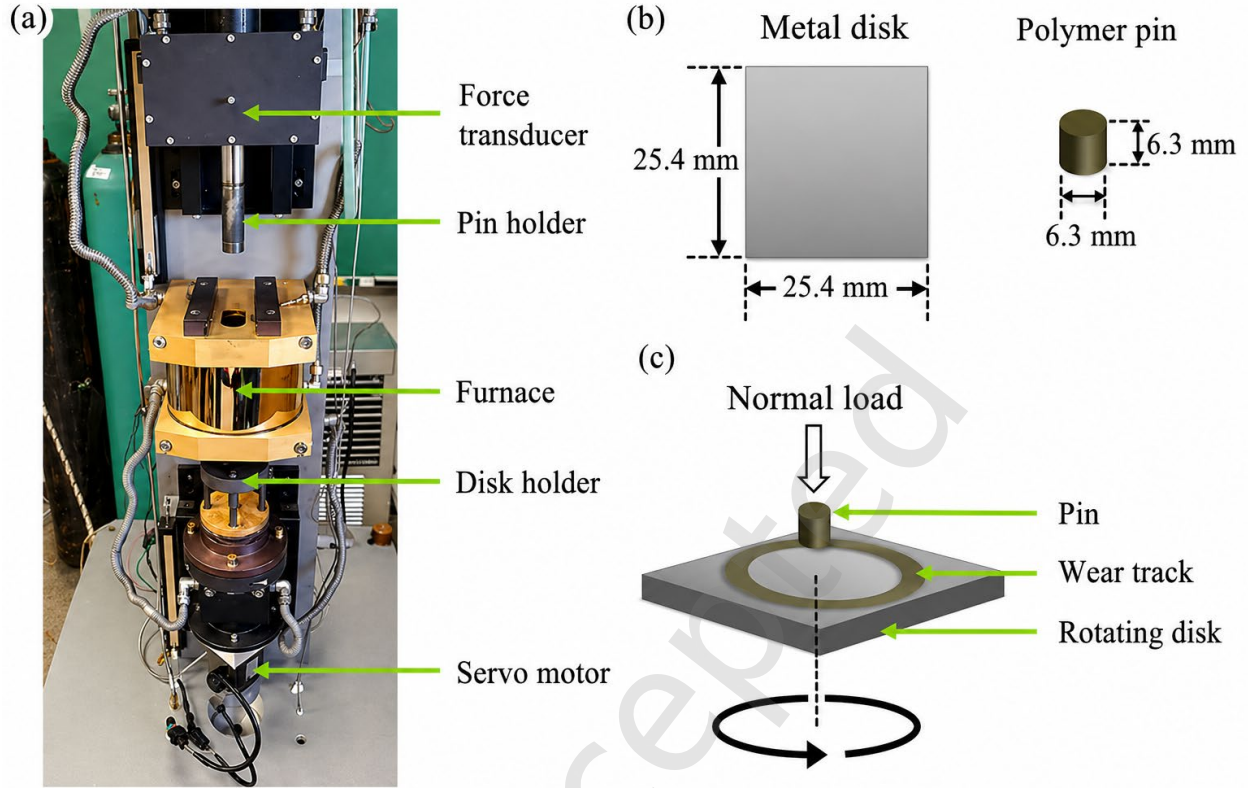


Figure 1. (a) Overall view and components of the HTT, (b) sample dimensions, and (c) flat pin-on-disk configuration.

2.3 Surface characterization and wear calculations

After the tribological tests were completed, the samples were investigated under an optical microscope to study the wear regions in detail. SEM and EDS were employed for further analysis. Linear scans across the wear tracks on the disks were performed using a contact profilometer to visualize material transfer on the counterpart disk. The profilometer stylus had a 2 μm radius, and a 0.1 mN load was applied during the scans. For the line scans, any tilt or curvature in the profile was corrected to remove surface waviness, and the area outside the wear track was adjusted to the “zero” line to accurately measure the presence of any transfer films or transfer layer thickness. A point to note here is that a line scan of the same disk could appear rougher when taken across the machining marks than when taken parallel to them. The mass of the composite pins was measured

using a scale with a precision of 0.01 mg before and after the experiments to calculate the wear rate of the pin material. Material transfer from the pin was measured by the following wear rate equation:

$$k = \frac{\Delta V}{W_n \cdot s} \quad (1)$$

Where k is the wear rate in (mm³/N.m), ΔV is the polymer volume loss (mm³), W_n is the normal applied load (N), and s is the total sliding distance (m).

In addition to further elucidating the tribochemical reactions occurring within the wear tracks, FTIR and XPS were employed. While FTIR provides information on chemically modified polymer functional groups, XPS identifies elemental composition and whether those functional groups are chemically bonded to the metal surface.

3. Results and discussion

3.1 Friction

The tribological results of the PTFE and ATSP composites are depicted in **Fig. 2**. Each test was repeated at least three times to verify the repeatability and reliability of the friction measurements. The results of the COF vs time depicted in **Fig. 2** are from a single experiment of each type, to better show the trends. **Fig. 2(e)** shows the average and the standard deviation of the COF for multiple tests. **Fig. 2(a)** shows the COF vs time for the PTFE + 15% ATSP composite against the SS 316 disks in air, H₂, and aged pins in the H₂ environment. No significant difference in friction performance was observed between the air and H₂ experiments, with the H₂-aged sample showing an increase in COF. A slight increase in the COF trend is also observed for the other PTFE composites in the presence of H₂, with COF values remaining stable at 0.2-0.25. The ATSP composite pin results are shown in **Fig. 2(d)**. The COF is slightly higher than that of the PTFE

composites, averaging 0.3, and shows a slight reduction under H_2 , which is opposite the trend with the PTFE composites. The stability of the COF in the PTFE composites after a short run-in period can be attributed to PTFE transfer films on the metal disk surface. This material transfer process is governed by adhesive forces at the metal-polymer composite interface and by cohesive forces within the polymer composite [40, 41].

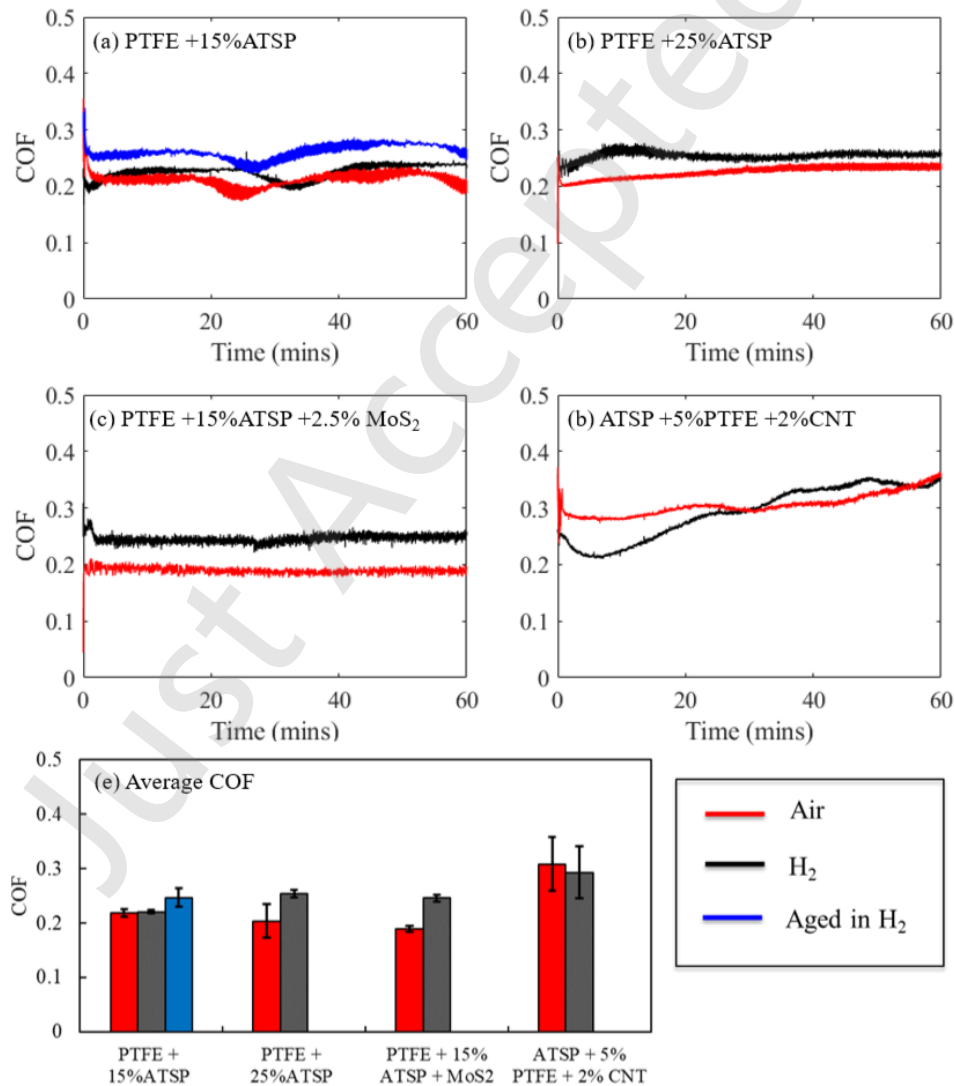


Figure 2. (a-d) In-situ COF for the tribological tests for the four composites in air and H_2 , and (e) Average COF with $\pm$ one standard deviation (SD) obtained from multiple experiments.

3.2 Wear

The wear rates calculated by mass loss of the composite pins are depicted in Fig. 3. Each bar represents the average wear rate ± 1 SD across multiple experiments at each set of conditions. As expected, the ATSP-matrix composite showed the lowest wear rate, which was one order of magnitude lower than that of the PTFE-matrix composites. This can be partially attributed to its hardness (ATSP exhibits extremely high wear resistance), as documented in previous works [11, 42, 43, 44]. For the ATSP +5%PTFE +2%CNT composite, the wear rate in air was 6×10^{-7} mm³/N.m, and in H₂ was 1.6×10^{-6} mm³/N.m, which is excellent for such applications. The effect of hydrogen on the wear was noticeable, but not significant in any of the composites. The highest wear rate was measured for the PTFE + 15% ATSP composition, and the wear rate decreased with increasing ATSP content and with the addition of MoS₂. Still, the highest wear rate was less than 2×10^{-5} mm³/N.m, suggesting a good performance by all the composites tested. The wear rates of the PTFE-based composites are lower than the wear rates observed by Sawae et. al. in high-pressure hydrogen for unfilled PTFE (6×10^{-5} to 8×10^{-4} mm³/N.m) [27]. The aging of the PTFE+15% ATSP in H₂ environment showed no major difference in the wear performance. Overall, the wear of the PTFE and ATSP-based composites appears unaffected by the hydrogen environment at room temperature and atmospheric pressure.

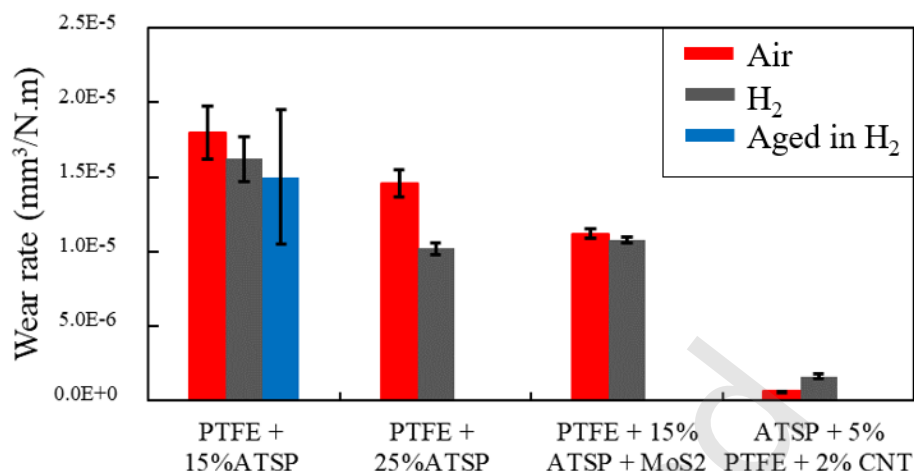


Figure 3. Wear rate comparison of the composite polymer pins. Error bars: $\pm$ one standard deviation (SD) obtained from multiple experiments.

Fig. 4 shows optical images of the disks, depicting the wear-track region after testing with PTFE + 15% ATSP composite pins in air, in H₂, and with aged pins in the H₂ environment. The figure also shows line scans across the wear tracks on the disks to measure any material transfer. At least four (90° apart) 10 mm-long scans across the 6.3 mm wear tracks were taken on all sides of the disks. The scans best representing the samples are shown in the figures. The wear track is easily recognizable in the optical images as a slightly darker region. The wear scans show that a stable transfer film approximately 2 μm thick was observed on the PTFE +15% ATSP disk in a H₂ environment. The formation of the transfer films (compared to tribolayers, which are lumpy, discontinuous, and less than 0.1 μm thick) has been observed and reported before, and it is attributed to the increase in interfacial adhesion between the asperities of the tribopairs compared to the cohesive strength of the polymers during sliding [45]. Such a recognizable tribofilm could not be observed or measured in air. In the H₂-aged composite, the valleys on the wear track appear to be filled with pin material, suggesting the presence of a transfer layer. The presence of transfer

films and layers in PTFE-ATSP blends has already been demonstrated, yet claims that even when the metal disk counterpart has low surface roughness, contact profilometry cannot detect any measurable transfer film [32, 44].

Similar behavior was observed in the PTFE +15%ATSP+2.5% MoS₂ experiments (**Fig. 5(a)**), and a stable transfer film approximately 1 μm thick was measured on the wear track after the H₂ environment tests. **Fig. 5(b)** depicts the optical images and the profilometric scans of the wear track after sliding against the PTFE +25%ATSP pins. Although there are signs of slight material deposition on the disk in the H₂ environment, the profilometric scan resembles what is characteristic of a transfer film. The higher ATSP content compared to the previous two composites results in a lower wear rate in this pin.

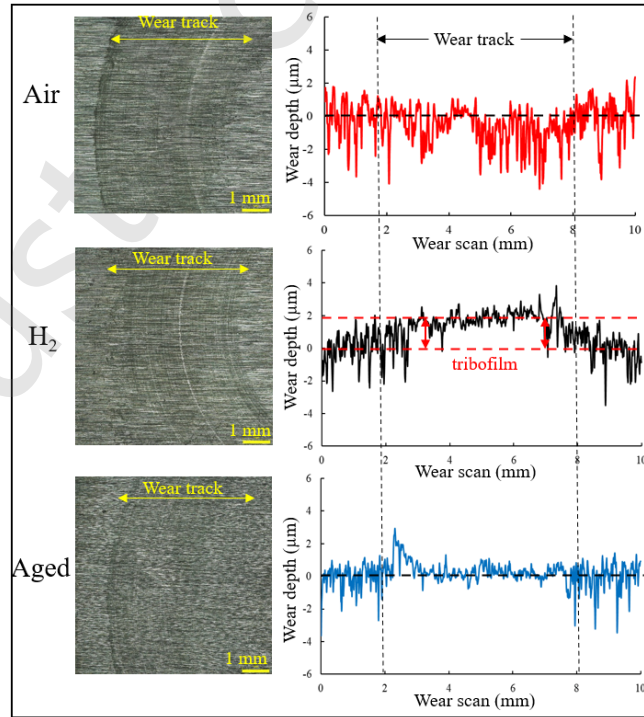


Figure 4. Optical microscopy images and representative line scans across the wear tracks on the disks tested against PTFE + 15% ATSP composite pins.

The optical images and profilometric scans of the SS316 disks run against the ATSP +5%PTFE +2%CNT are shown in **Fig. 5(c)**. No signs of wear are visible in the wear track, consistent with this composite showing the lowest wear rate. The ATSP-matrix pin is resistant to adhesive and abrasive wear, demonstrating excellent performance. The lower wear of the ATSP-matrix composite is related not only to its higher bulk hardness but also to the way this hardness affects contact mechanics. At a fixed nominal pressure, the harder composite resists asperity penetration and plastic indentation, supports a larger fraction of the load elastically, and limits the development of the real contact area. Consequently, subsurface deformation, plowing, and abrasive microcutting are reduced. ATSP and CNT reinforcement also restrict matrix deformation and crack propagation, while PTFE and MoS₂ modify interfacial shear and debris accommodation. These mechanisms explain why the ATSP + 5% PTFE + 2% CNT composite show extremely low wear despite a coefficient of friction slightly higher than that of the PTFE-matrix blends. Thus, hardness primarily governs resistance to deformation and material removal, whereas friction also depends on the chemistry, continuity, and shear strength of the transfer layer.

To better understand the wear mechanism, SEM and EDS analyses were performed on the worn regions.

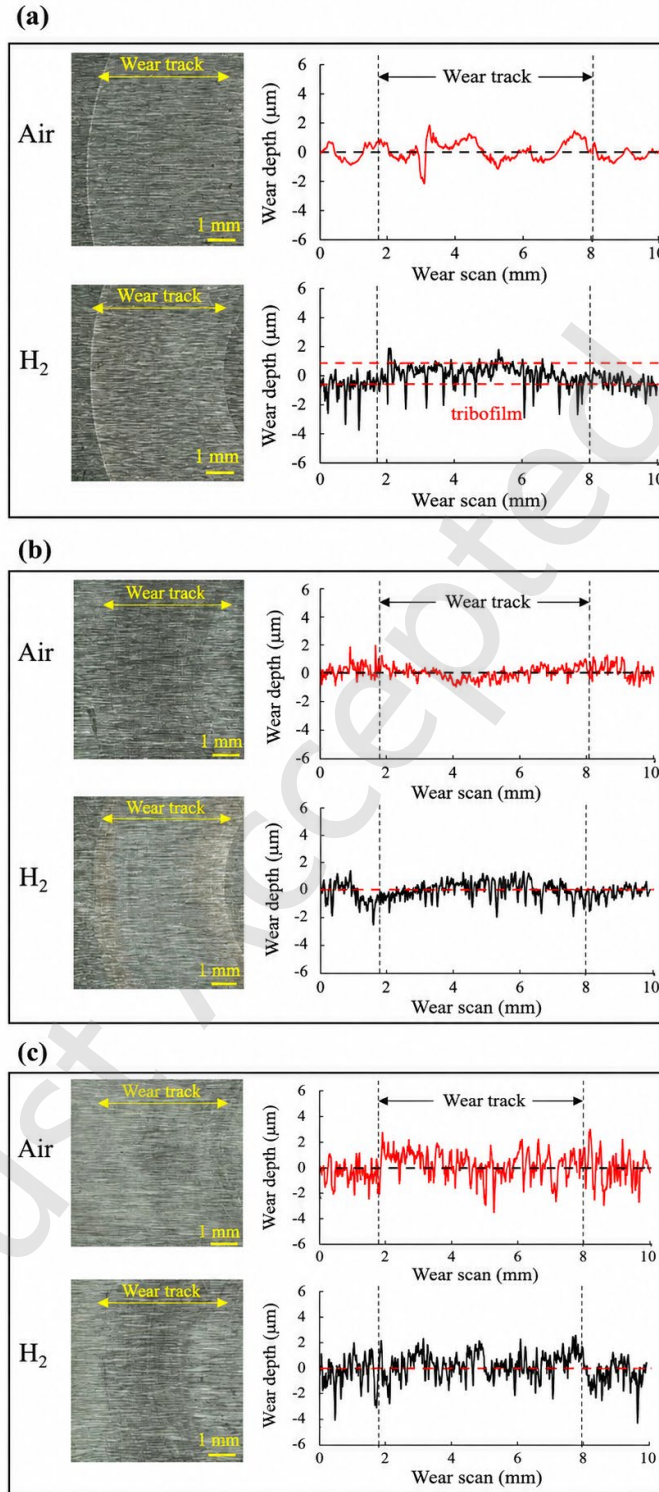


Figure 5. (a) Optical microscopy images and representative line scans across the wear tracks on the disks tested against PTFE + 15% ATSP + 2.5% MoS₂ composite pins. (b) microscopy images and line scans across the wear tracks on the disks tested against PTFE + 25% ATSP composite pins. (c) microscopy images and line scans across the wear tracks tested on the disks against ATSP + 5%PTFE + 2%CNT composite pins.

3.3 SEM and EDS

SEM and EDS were performed at least twice on each worn area at multiple magnifications. Only the best visually representative images were shown. **Fig. 6 (a)** through **(f)**, **Fig. 6 (g)** through **(l)**, and **Fig. 6 (m)** through **(r)**, present the SEM and EDS images of the worn regions of the SS 316 disks after sliding against the PTFE + 15% ATSP pins in air, H₂, and aged pins in the H₂ environment, respectively. In **Fig. 6(a)**, the wear track is discernible, which shows a slight amount of material transfer (**Fig. 6(b)**). When EDS was performed on the same region shown in **Fig. 6(b)**, a high content of fluorine (33 wt.%) was found on the wear track. The fluorine patches are attributed to the fragmentation of PTFE composite pins inside the wear track. In the H₂ environment (**Fig. 6(k)**), a higher F content (46 wt.%) was observed in the wear-track region, which was attributed to the thick, stable transfer film. These results also support the formation of a metal fluoride layer on steel metal atoms by PTFE radicals in a high-pressure hydrogen environment [29]. A similar higher-F-rich (43 wt.%) material transfer film was found on the aged pin tests in an H₂ environment (**Fig. 6(q)**). A point to note here is that the direction of the machining marks did not affect the results, as multiple areas were evaluated on the wear track, and the results agreed. The formation of stable tribofilms at the interface is responsible for the lower COF and wear of these composites, due to the lower shear strength between the polymer-polymer interface. It has been shown that once these transfer films form during the running-in transient period, further wear of the PTFE and ATSP composites ceases [43, 45].

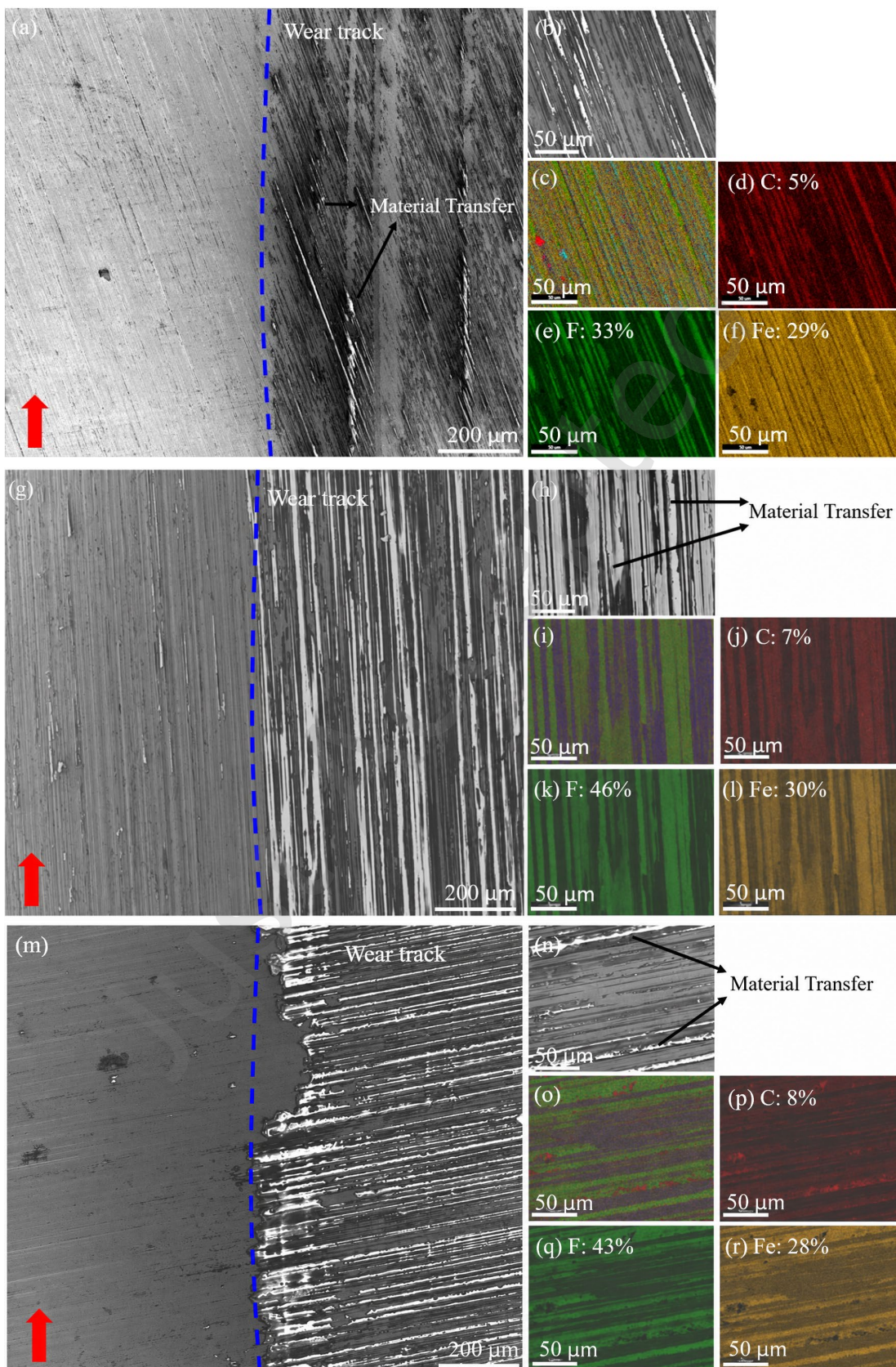


Figure 6. (a) SEM image of the SS 316 disks after testing against PTFE +15%ATSP pin in air (red solid arrow points to the sliding direction, and blue dashed line separates the inside (right) and outside (left) wear track region), (b) magnified image inside the wear track where EDS was taken, and EDS maps showing (c) all elements combined along with (d) C, (e) F, and (f) Fe elements. (g) SEM image of the SS 316 disks after testing against PTFE +15%ATSP pin in H₂, (h) magnified image inside the wear track where EDS was taken, and EDS maps showing (i) all elements combined along with (j) C, (k) F, and (l) Fe elements. (m) SEM image of the SS 316 disks after testing against the Aged PTFE +15%ATSP pin in H₂ (n) magnified image inside the wear track where EDS was taken, and EDS maps showing (o) all elements combined along with (p) C, (q) F, and (r) Fe elements.

Figs. 7(a) through (g) and (h) through (n), present the SEM and EDS maps of the wear tracks on SS 316 disks after sliding against the ATSP +5%PTFE +2%CNT pins in air and H₂ environments, respectively. Wear regions from both tests show some amount of polymer presence, indicative of the C, F, and O regions. Specifically, the sample tested in air contains C (6%), F (17%), and O (14%), whereas the sample tested in H₂ contains C (14%), F (17%), and O (18%). The lower presence of the composites on the disk surface not only agrees with the low wear rates observed in the ATSP-based composite but also confirms that ATSP matrix composites do not form transfer films. It can be seen in **Fig.7(a)** that most machining marks of the SS disk are not covered with material transfer from the composite ATSP polymer pin (as opposed to the PTFE matrix composites), which confirms that only transfer layers are formed.

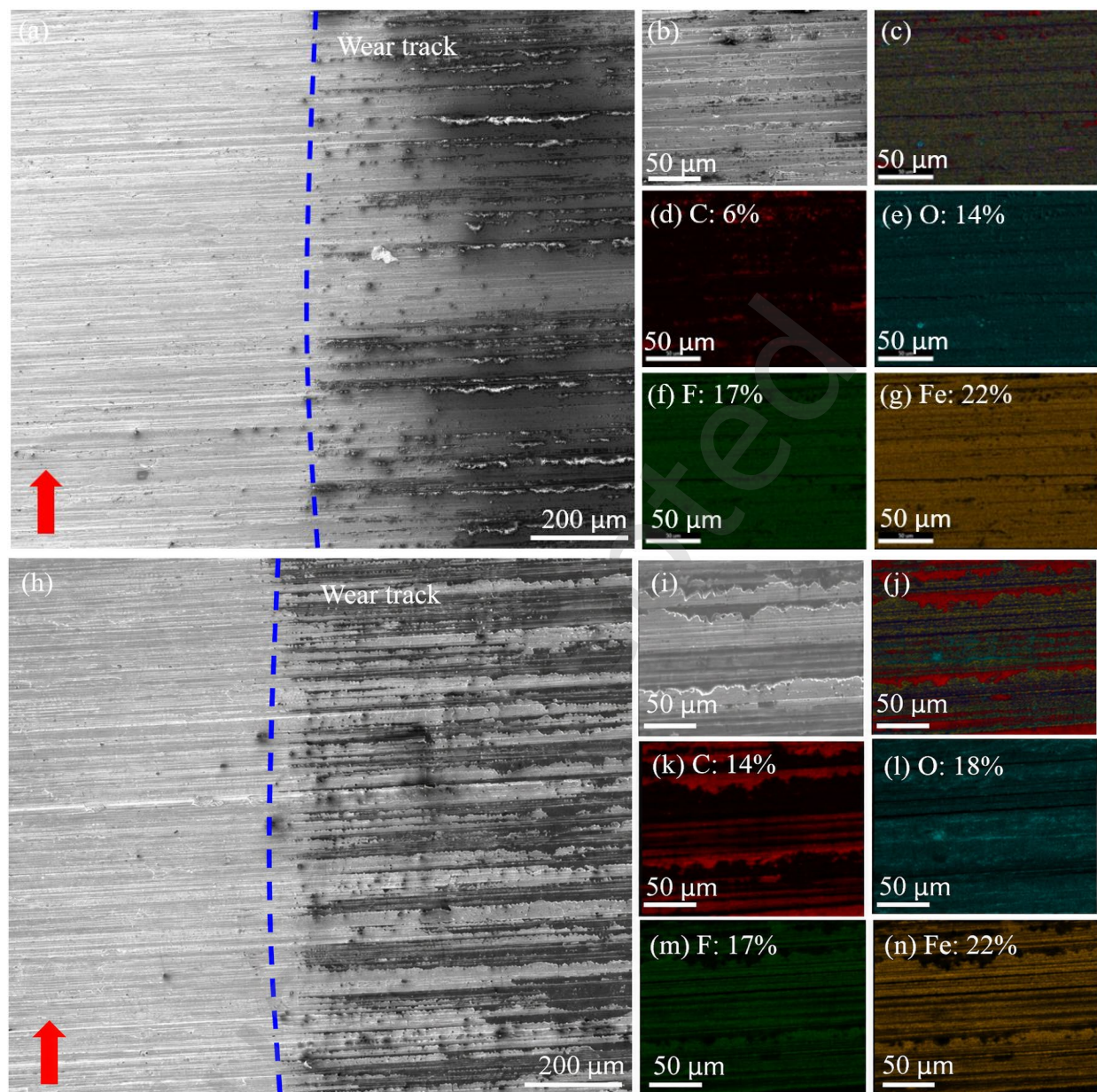


Figure 6. (a) SEM image of the SS 316 disks after testing against the ATSP +5%PTFE +2%CNT pin in air (red solid arrow points to the sliding direction, and blue dashed line separates the inside (right) and outside (left) wear track region), (b) magnified image inside the wear track where EDS was taken, and EDS maps showing (c) all elements combined along with (d) C, (e) O, (f) F, and (g) Fe elements. (h) SEM image of the SS 316 disks after testing against the ATSP +5%PTFE +2%CNT pin in H₂ (i) magnified image inside the wear track where EDS was taken, and EDS maps showing (j) all elements combined along with (k) C, (l) O, (m) F, and (n) Fe elements.

3.4 FTIR analysis

FTIR analysis of the pins and disks was performed to elucidate tribochemical reactions and to further clarify the friction and wear behavior of the different polymer composites examined in this study. **Fig. 8** shows the normalized FTIR spectrum of the PTFE+15%ATSP composite pin tested in H_2 , where strong characteristic peaks are observed at 1150 and 1205 cm^{-1} [46, 47, 48]. Note that the spectrum is similar after tribotesting, indicating that the material transfer maintains the chemical composition of the pin. These prominent peaks can be attributed to stretching vibrations of CF_2 . Compared to the sharp peak at 1150 cm^{-1} , it can be observed that the shoulder at 1205 cm^{-1} corresponds to C–O–C stretching vibrations from the aromatic ether linkages in ATSP, indicating its presence and potential interfacial interaction with the PTFE matrix. Peaks at 1005 and 1055 cm^{-1} belong to C–F stretching vibrations from the PTFE backbone, whereas those at 1612 and 1743 cm^{-1} are skeletal vibrations of aromatic (benzene) rings, and C=O stretching vibration bond, respectively [38]. Once PTFE+15%ATSP pins slide against stainless steel, it can be observed that the spectra inside the disk wear track shows a sharp peak at 1150 and a shoulder at 1205 cm^{-1} (as shown in **Fig. 9**), however, both are of lower intensity compared to **Fig. 8**. Peaks of less intensity can be associated with the transfer film of the PTFE+15%ATSP composite to the SS disk during sliding. This indicates the presence of transferred material, likely PTFE and/or ATSP debris that accumulated and bonded to the stainless-steel surface during sliding. In addition, chemical compounds during the tribotesting of PTFE+15%ATSP correspond to CF_2 vibrations of PTFE and ester structures from ATSP being formed into the surface of the SS 316 disk, as previously shown in **Figs. 6** and **7**, where the EDS spot was mainly composed of F due to the fragmentation of the PTFE. The FTIR peaks observed in the spectra agree with the profilometric measurements and the SEM-EDS microscopy images, which show a transfer film on the surface of the SS. The FTIR inside the wear track of the SS disk after testing of PTFE+15%ATSP pins in air and of the aged

pin in H₂ shows similar FTIR spectra, indicating that the presence of H₂ is not significantly affecting the tribotested materials.

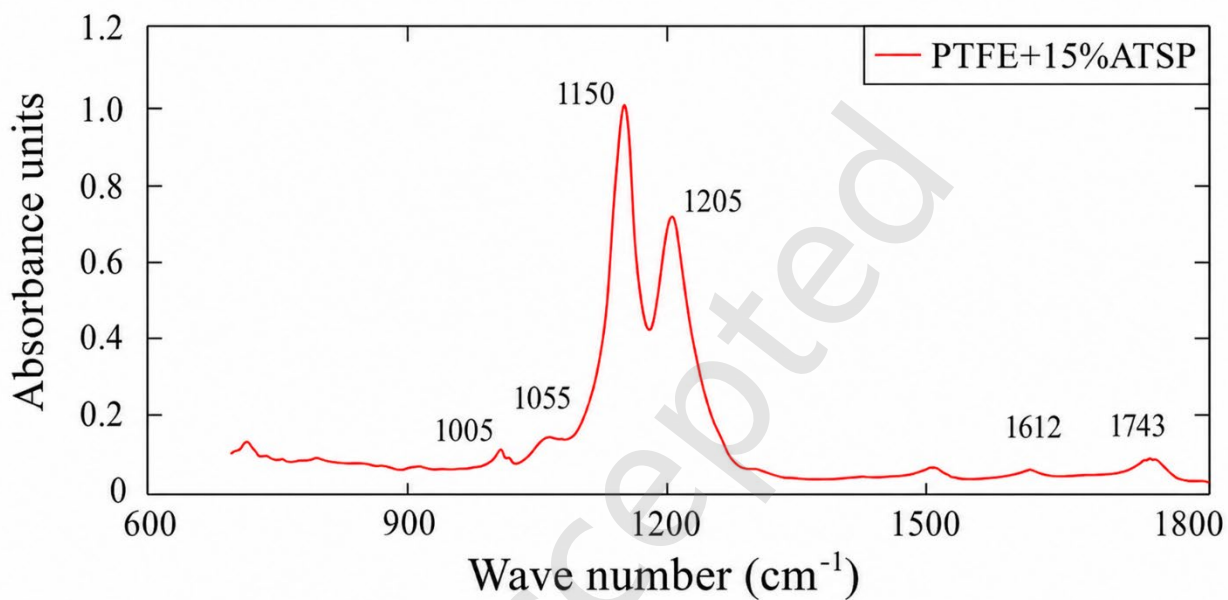


Figure 8. FTIR spectrum of PTFE+15%ATSP pin tested in H₂. Similar spectra were measured for the untested pin and pin tested in air.

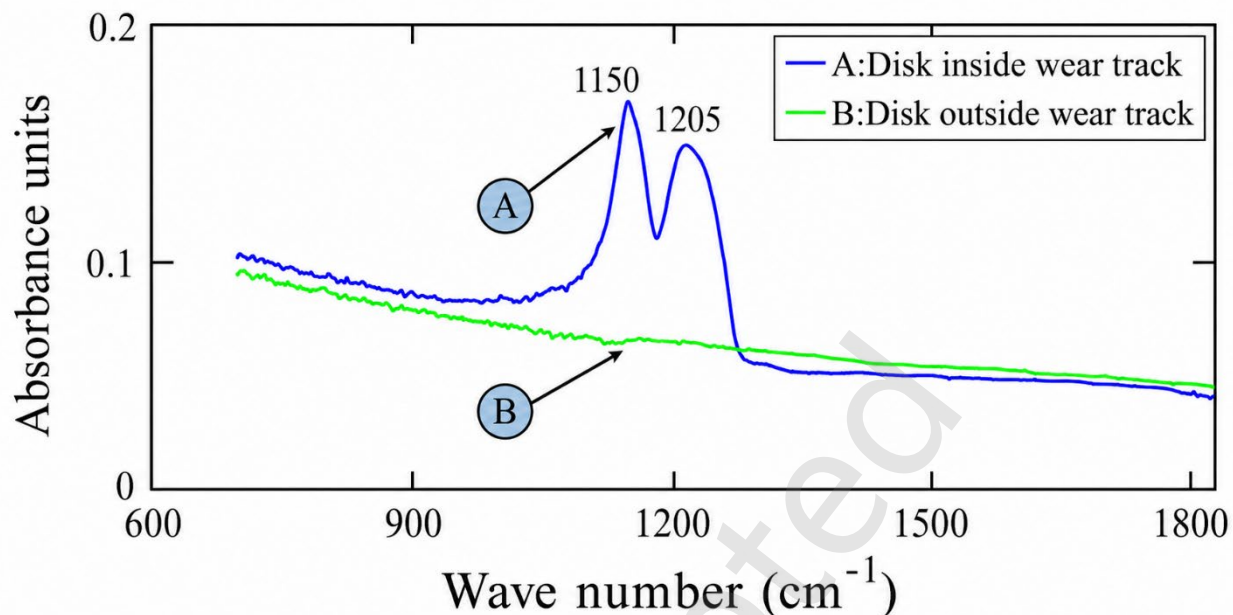


Figure 9. FTIR spectra measured on the SS 316 disk, after testing with PTFE+15%ATSP pin in H_2 atmosphere. Similar spectra are observed when tested in air, and with aged samples.

Fig. 10 shows the FTIR spectra measured on the disk (inside and outside the wear track) after testing with an ATSP+5%PTFE+2%CNT pin in a H_2 environment. Inside the wear track, peaks at 1150 and 1205 cm^{-1} are also present, but they are weaker than those measured in the PTFE+15%ATSP run in H_2 . Moreover, both peaks are much broader and are observed only within the wear track, suggesting that more complex tribochemical interactions occur. For instance, the weak, broad peak around 1150 cm^{-1} is related to skeletal vibrations of CF_2 in PTFE and might overlap, making the signal appear wider. Also, in the wavelength band near 1150 cm^{-1} , tribochemical metal-organic surface reactions may occur, as supported by the higher Fe content observed in the SEM-EDS spectra in **Figs. 6** and **7**. The weak peak at 1205 cm^{-1} might be explained considering that under normal load and sliding speed, tribochemical reactions of PTFE that cause C–C or C–F fluorinated layers (F is the higher element content in **Figs. 6** and **7** after Fe). In this

region, thermal and mechanical degradation of ATSP during tribological testing breaks ester bonds, forming fragments containing aromatic ester and ether groups with C–O and C–O–C stretching vibration bands, which ultimately form fluorinated layers in the presence of hydrogen. The overlap of these tribochemical compounds results in a broad peak in the wavelength spectrum. There were no significant differences in the FTIR spectra of ATSP+5%PTFE+2%CNT run in a H₂ atmosphere compared to air, indicating that 5% H₂ in H₂/N₂ mixture at atmospheric pressure does not play a significant role in the tribochemical reactions.

Based on the FTIR results of PTFE+15%ATSP (PTFE matrix composite) and ATSP+5%PTFE+2%CNT (ATSP matrix composite) after tribotesting, the presence of H₂ has a negligible influence on friction and wear behavior, as the spectra are similar in air and H₂ atmospheres. Also, considering the stronger CF₂ peaks measured after testing of PTFE+15%ATSP compared to those of ATSP+5%PTFE+2%CNT, it is hypothesized that the prevailing mechanism that explains the comparatively lower COF of PTFE+15%ATSP is the formation of transfer films onto the surface of SS 316. From a wear-rate perspective, for ATSP+5%PTFE+2%CNT, it is proposed that the dominant factor explaining its lower wear rate relative to PTFE+15%ATSP is its higher bulk hardness. This is supported not only by profilometric measurements and SEM/EDS images of ATSP+5%PTFE+2%CNT, which show uneven or lumpy transfer layers in the polymer composites, but also by the higher friction coefficient of ATSP+5%PTFE+2%CNT compared to PTFE+15%ATSP composites. The higher COF implies that a higher shear traction is required to abrade the harder asperities of the ATSP+5%PTFE+2%CNT during sliding against SS 316.

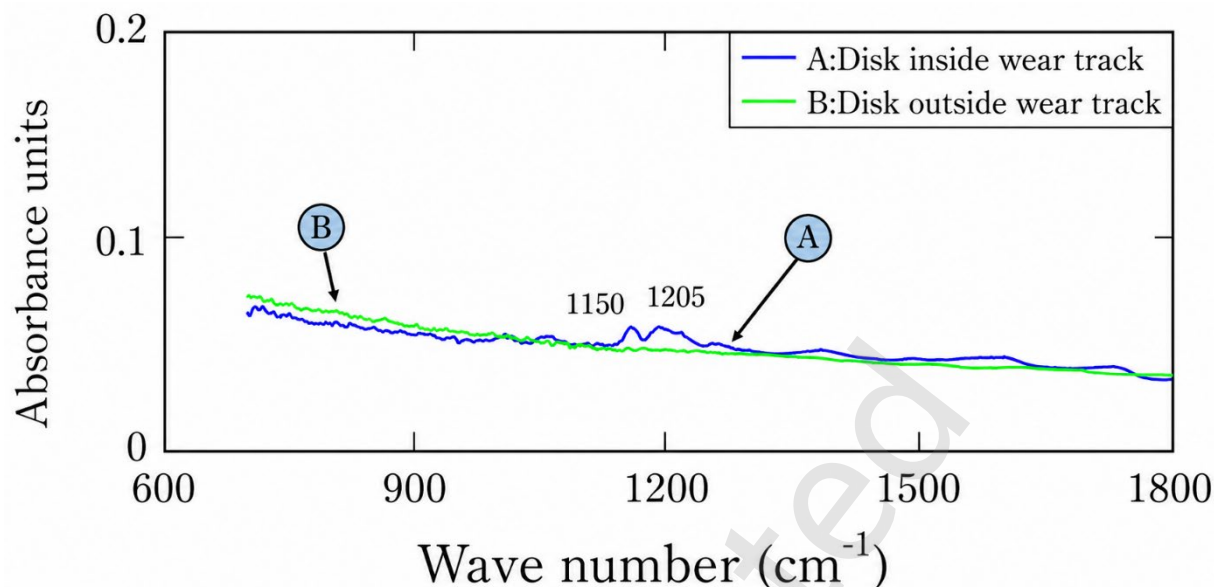


Figure 10. FTIR spectra measured on the SS 316 disk, after testing with ATSP+5%PTFE+2%CNT pin in H₂ atmosphere. Similar spectra are observed when tested in air.

3.5 XPS analysis

Tribochemical characterization of the wear tracks after testing was performed using XPS. **Fig. 11** shows the XPS spectra inside the wear track of SS 316 after testing of PTFE + 15%ATSP pins in H₂ atmosphere. The 5%H₂–95%N₂ atmosphere used in these experiments is mildly reducing and oxygen-poor. Therefore, the dominant process is not extensive oxidative degradation, but rather mechanically assisted transfer-film formation, accompanied by limited chemical reactions involving PTFE fragments, ATSP oxygenated groups, and the native oxide/hydroxide layer on SS 316. From **Fig. 11(a)**, it can be observed that the fitted C 1s spectra include contributions from C–C/C=C, C–O, C=O/O–C=O, CF₂/C–F, and CF₃, while F 1s includes C–F/CF₂ and possible Metal–F/Fe–F components (see **Fig. 11(b)**). Based on **Figs. 11(a) and (b)**, it can be ascertained that the transferred film is not pure PTFE, but it is a mixed tribolayer containing fluorinated PTFE fragments, ATSP-derived aromatic/oxygenated fragments, and metal-containing species from the

SS 316 surface. It has been reported that the presence of H_2 , during the tribotesting between steel and PTFE, tends to suppress metal oxide formation and converts Fe(III) to Fe(II), thereby inhibiting the formation of Fe–F bonds unless oxygen and water are present. Under a 5% H_2 –95% N_2 atmosphere, the expected tribochemistry is weaker than in humid air or in oxygen-containing hydrogen. The main reactions are associated with polymer transfer, PTFE chain scission, fluorinated transfer-layer formation, and interaction with pre-existing SS 316 oxides, rather than strong oxidation-driven formation of metal carboxylate chelates [50].

ATSP contributes aromatic and oxygenated chemical groups to the tribolayer. As it was noted in the FTIR section, peaks near 1612 cm^{-1} , assigned to aromatic ring skeletal vibrations, and 1743 cm^{-1} , assigned to C=O stretching, were identified. These are consistent with the aromatic thermosetting copolyester structure. During sliding, ATSP can undergo mechanochemical degradation, especially at ester and ether linkages, with their fragments potentially incorporated into the tribofilm. In XPS, this appears as C 1s components C–O and C=O/O–C=O near 284.8 eV (**Fig. 11(a)**), and O 1s components associated with C=O/ester and C–O/Fe–OH (~ 532.5 – 533.4 eV , in **Fig. 11(d)**). The O 1s spectrum therefore reflects both the oxygenated polymer chemistry of ATSP and the oxidized/hydroxylated SS 316 metallic counterface. As the SS 316 naturally contains a passive oxide film rich in chromium oxide, iron oxides, and mixed Fe–Cr–Ni oxides, during sliding, this passive film is mechanically disrupted and renewed, and the exposed metallic surface can interact with transferred polymer fragments. In an oxygen-poor 5% H_2 –95% N_2 environment, the oxide renewal process is limited compared with air, but it is not completely absent because stainless steel already has a native oxide film before testing, and trace O_2/H_2O may remain after chamber evacuation/filling. It can also be observed that Fe–F peaks in Fe 2p (**Fig. 11(c)**) are associated with localized reaction between fluorinated PTFE fragments and activated Fe–

containing sites on the SS 316 surface. During sliding, PTFE undergoes chain scission, producing fluorinated fragments and F-containing free chain ends. These species can interact with Fe sites exposed by mechanical wear or with Fe oxide/hydroxide sites in the passive film. The Fe–F peaks in Fe 2p are therefore evidence that the transferred PTFE was not only physically deposited, but also chemically anchored at some locations on the SS 316 surface. For instance, the Fe–F (2p 3/2) at lower (near 714–715 eV) and Fe–F (2p 1/2) at higher binding energy (near 727–728 eV), respectively, may seem unexpected because the PTFE in pure H₂ suppresses tribochemical reactions by reducing iron oxides and decreasing the Fe(III) fraction [50]. However, the current SS 316 counterface already has a native passive oxide/hydroxide film prior to sliding. Thus, in a mildly reducing 5%H₂-95%N₂, the H₂ content may limit extensive oxidation, but it does not completely remove the pre-existing oxide/hydroxide layer. In addition, the sliding process continuously exposes fresh metal and mechanically activates the interface. These conditions can allow localized Fe–F formation, especially because PTFE + 15%ATSP contains enough PTFE to provide abundant fluorinated fragments.

Fig. 12 shows the XPS spectra obtained from the SS 316 disk after testing the ATSP+5%PTFE+2%CNT pin in H₂ atmosphere. Unlike PTFE + 15%ATSP, this blended pin material contains majorly ATSP and includes CNTs, meaning that the tribolayer is not dominated only by PTFE-derived CF₂/C–F species. Instead, it contains contributions from ATSP degradation products, CNT-derived graphitic carbon, PTFE fluorinated fragments, and stainless-steel oxide/hydroxide species. In **Fig. 12(a)** in the C1 spectrum, the C–C/C=C species is the dominant carbon contribution. It comes from the aromatic ATSP backbone and the graphitic/sp² carbon structure of CNTs. CNTs strongly contribute to this peak, making the tribofilm more carbon rich. While C–O species come from ether/ester fragments of ATSP generated during sliding, which

may also include oxidized carbon species formed at the interface, $C=O/O-C=O$ is associated with ester/carbonyl groups from ATSP and possible oxidized polymer fragments. It can also be observed that $CF_2/C-F$ comes from PTFE chain scission and transfer of fluorinated fragments to the steel surface.

Considering that ATSP+5%PTFE+2%CNT blended contains only 5% PTFE. Therefore, the C 1s spectrum indicates that the tribofilm is primarily ATSP/CNT-rich, with a smaller PTFE-derived fluorinated contribution. **Fig. 12(b)** shows the F1S spectra, from which it can be observed that C-F/ CF_2 , which is the main signal, is derived from the fluorine fragmentation from PTFE transferred to the SS 316 wear track, being also the Metal-F/Fe-F, an indication of localized reaction between fluorinated PTFE fragments and Fe-containing sites in the stainless-steel oxide layer. However, in ATSP+5%PTFE+2%CNT, the Metal-F/Fe-F signal should be interpreted as localized, not as a continuous iron-fluoride film. It likely forms where PTFE-derived fluorinated fragments contact mechanically activated Fe/oxide/hydroxide sites exposed during sliding. In addition, the lower and higher binding energy Fe-O/OH 2p_{3/2} and Fe-O/OH 2p_{1/2}, respectively, are expected in the Fe 2p spectra in **Fig. 12(d)**, since SS 316 naturally contains a passive oxide/hydroxide film. During sliding, this film is locally removed, re-exposed, and mixed with polymer debris. It has been claimed that the Fe 2p_{3/2} oxide/hydroxide appears near 710–711 eV, and its formation can reduce iron oxides in an H₂-rich atmosphere, decreasing Fe(III) and inhibiting strong Fe-F formation [50]. However, in a 5%H₂–95%N₂ atmosphere, formation of Fe 2p_{3/2} oxide/hydroxide appears to be less inhibited as seen in **Fig. 12(c)**.

The O1s spectra, in **Fig. 12(d)**, show Fe/Cr/Ni oxides derived from the native SS 316 passive film and tribologically exposed oxide surface. C=O/ester and C-O/Fe-OH area attributed to Carbonyl

and ester groups from ATSP backbone, overlapping contribution from ATSP ether/ester C–O groups, and hydroxylated metal species on SS 316 fragmentation during sliding, respectively.

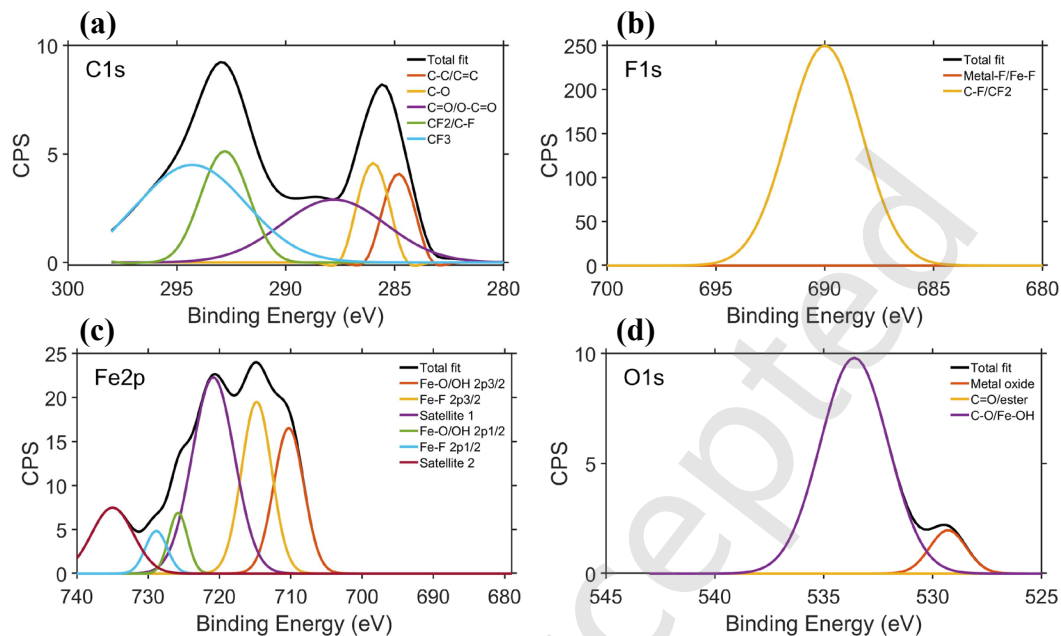


Figure 11. XPS spectra measured on the SS 316 disk wear track, after testing with PTFE + 15%ATSP pin in H₂ atmosphere.

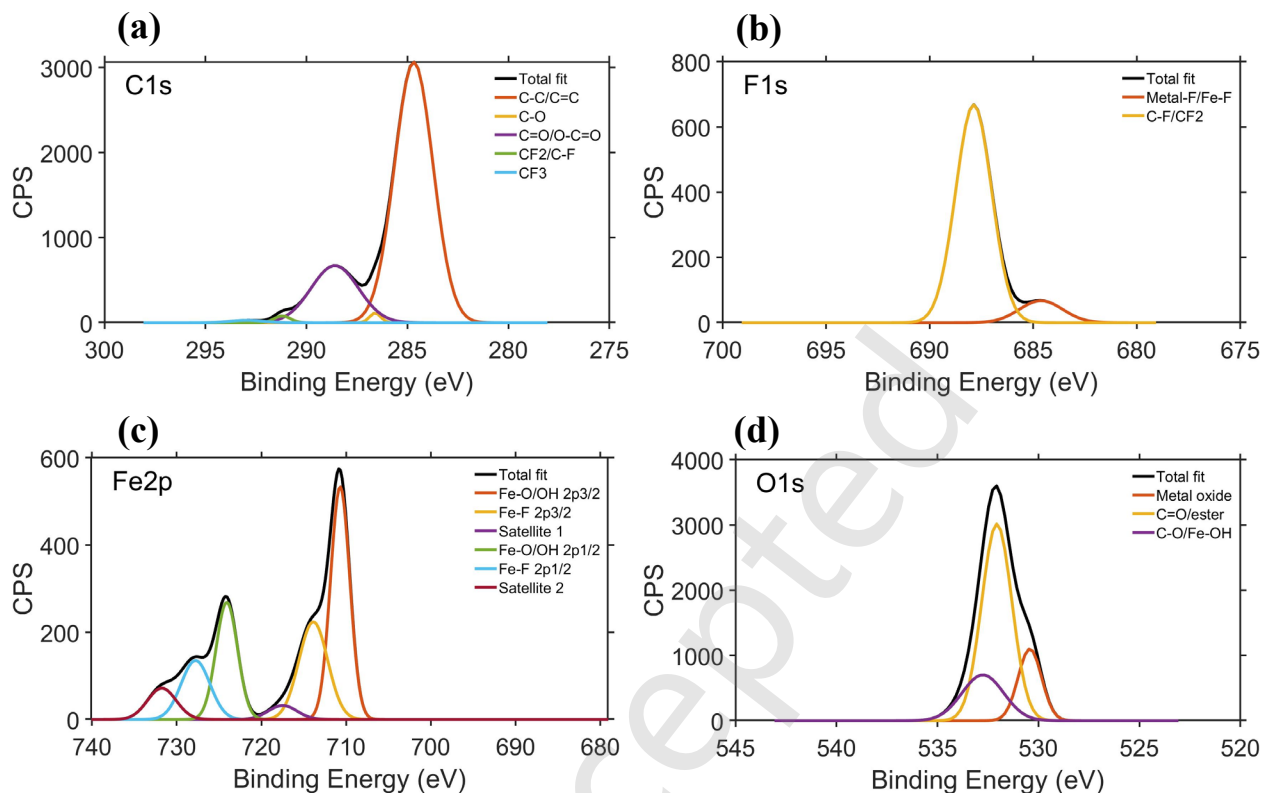


Figure 12. XPS spectra measured on the SS 316 disk wear track, after testing with ATSP+5%PTFE+2%CNT pin in H₂ atmosphere.

3.6 Discussion

To summarize the findings of this study, the wear rate is plotted against the COF for all the tribopairs in **Fig. 13**. The performance of ATSP-based composites stands out the most with the lowest wear rate; however, at a cost of slightly higher COF. This warrants further development of ATSP-based composites with PTFE and other reinforcing fillers to achieve excellent wear-resistant composites. Exposure to 5%H₂-95%N₂ may yield a higher friction coefficient than testing in air, as the atmosphere is relatively poor in oxygen and moisture. In air, the SS 316 surface can maintain or regenerate a more oxidized/hydroxylated passive layer during sliding. That oxide/hydroxide

surface can help anchor PTFE/ATSP fragments and support the development of a more stable transfer film.

A stable material-transfer film is observed on the disks tested with the PTFE-based composite pins. Once a stable transfer film forms, it reduces wear by lowering shear, making the composites highly durable. Similar behavior has been reported in self-lubricating metallic composites, where the formation of protective lubricious tribofilms plays a decisive role in reducing friction and improving wear resistance under severe sliding conditions [51]. These findings are in line with studies by Sawae, Nakashima et al. on unfilled and graphene-filled PTFE composites against metal disks in high-pressure hydrogen environments [26, 27, 29]. Their studies show that in the presence of high-pressure hydrogen, metal fluoride creation is enhanced, leading to tribo film formation, which in turn reduces the wear rate. Moreover, the wear rates measured agree with hardness measurements: harder polymer composites, such as ATSP +5%PTFE +2%CNT and PTFE +15%ATSP +2.5%MoS₂, exhibit lower wear rates than softer ones, such as PTFE +15%ATSP. Also, the transfer film evolves through four coupled stages. In the first stage, running-in produces polymer fragments by adhesive detachment, PTFE chain scission, and local mechanical degradation of ATSP ester/ether linkages. In the next stage, these fragments deposit on exposed SS 316 asperities and in machining valleys. The following stage consists of repeated sliding, compacting, smearing, and shear-aligning the debris, producing a more continuous fluorine-rich film for PTFE-matrix composites and a thinner, discontinuous transfer layer for the harder ATSP-matrix composite. In the final stage, mechanically activated steel sites and the native oxide/hydroxide layer interact locally with fluorinated and oxygenated fragments. SEM/profilometry identifies the deposited morphology, EDS verifies fluorine enrichment, FTIR confirms CF₂ and ATSP-derived functional groups, and XPS demonstrates a mixed tribolayer

containing fluorinated carbon, oxygenated ATSP fragments, metal oxides/hydroxides, and localized Fe–F bonding. Once established, this low-shear polymer-rich interface reduces direct polymer–metal contact, stabilizes friction, and suppresses further material removal.

Overall, from the FTIR and the XPS spectra, it can be claimed that for PTFE + 15%ATSP, the main mechanism is the formation of a PTFE-rich transfer film on the stainless-steel surface. The stronger FTIR peaks associated with CF_2 and C–F vibrations indicate that fluorinated fragments from PTFE are effectively transferred to the SS 316 wear track, and likely lower friction by producing a low-shear interfacial transfer film. For ATSP+5%PTFE+2%CNT, the PTFE fraction is significantly lower, meaning that the transfer film is not dominated by fluorinated PTFE species. Instead, the tribolayer is more ATSP/CNT-rich, containing aromatic carbon, graphitic carbon from CNTs, oxygenated ATSP fragments, limited PTFE-derived C–F/ CF_2 species, and metal oxide/hydroxide contributions from SS 316. This blended pin likely achieves better wear resistance mainly through higher hardness, CNT reinforcement, and a mechanically robust carbon/ATSP-rich tribolayer, rather than through a smooth PTFE-rich transfer film. In general, the present work was designed not only as a controlled screening study under a safe hydrogen-containing environment, specifically 5% H_2 –95% N_2 at atmospheric pressure, but also to determine whether hydrogen exposure and hydrogen aging significantly affect friction, wear, and transfer film formation relative to air. To clarify, the results should not be interpreted as a comprehensive map of all possible hydrogen service conditions. Instead, the findings provide an initial evaluation of PTFE and ATSP-based composites in a dilute hydrogen atmosphere and identify promising materials for further testing at higher H_2 concentrations, higher pressure, and variable humidity. To provide a complete assessment of hydrogen for storage and transportation applications, additional data on hydrogen concentration, pressure, temperature, and humidity would be required.

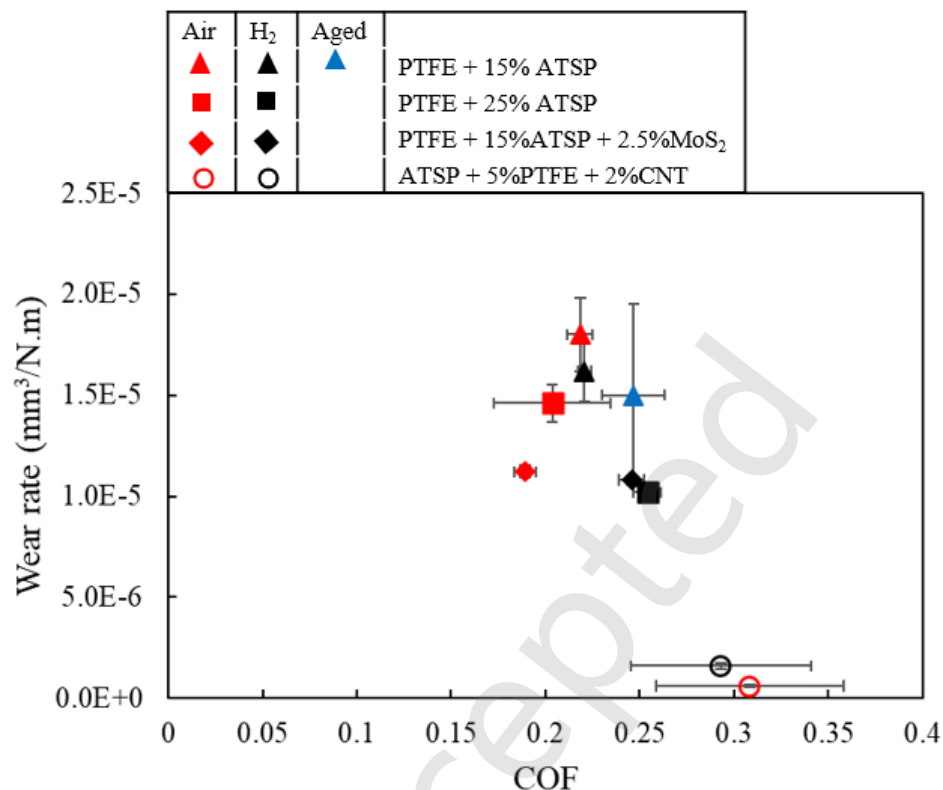


Figure 13. Summary of the COF vs wear rate of the tested composites.

4. Conclusions

Sliding experiments were performed on three PTFE-based composites and one ATSP-based composite in air and a 5% H₂-95% N₂ environment to study the impact of hydrogen on their tribological performance.

- (1) Under the specific room-temperature, atmospheric-pressure test conditions used here, changing from air to a 5% H₂-95% N₂ atmosphere produced only modest changes in friction and wear. The PTFE composites show a small negative effect under H₂, whereas the ATSP composites were not affected by H₂. This was also confirmed by FTIR spectroscopy, which showed that the spectra were not significantly affected by hydrogen, which behaves similarly to that observed in air.

- (2) The addition of ATSP in the PTFE matrix facilitates the formation of a stable material transfer film on the metal countersurface. This transfer film lowers the interfacial shear strength, thereby reducing the COF of the composites. However, from a wear performance perspective, ATSP composites exhibit significantly lower wear rates due to their higher bulk hardness.
- (3) The ATSP-based composite showed a slightly higher COF than the PTFE-based ones, but its wear rate was an order of magnitude lower. The low wear rate of the ATSP composite makes it an excellent candidate for applications where replacing parts is not possible or not cost-effective.
- (4) PTFE + 15%ATSP provides better friction reduction through a PTFE-rich, fluorinated transfer film, whereas ATSP+5%PTFE+2%CNT provides improved wear resistance through a harder, CNT-reinforced, carbon-rich tribolayer. In 5%H₂–95%N₂, the tribochemistry is dominated by mechanically assisted polymer transfer and limited interfacial reactions with the SS 316 passive film, rather than by extensive hydrogen-promoted chemical reactions. These findings identify the materials studied as promising candidates for hydrogen-related tribological components. However, they should not be extrapolated directly to high-purity hydrogen, high pressure, elevated temperature, cyclic decompression, or long-duration service without additional validation.

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