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Enhancing Fastening of Threaded Joints With Sustainable Lubricants From Waste Cooking Oil

Mineral-based lubricants commonly used in industrial tightening increase reliance on fossil resources and can contribute to environmental impacts, motivating the development of high-performance alternatives from renewable and waste feedstocks. This work evaluates waste-cooking-oil-derived lubricants for repeated tightening of high-strength threaded joints under severe loading. Two different processing routes for waste cooking oil are explored: hydrogenation to obtain a grease-like product, and chemical functionalization via epoxidation followed by ring opening to produce a polar polyol. The functionalized waste cooking oil lubricant delivers stable friction and torque responses over multiple tightening cycles, approaching the performance of a commercial MoS₂-containing paste and outperforming a conventional mineral engine oil as well as the minimally processed and hydrogenated waste cooking oil products in repeatability. In-depth wear analyses of the contact surfaces complement the tightening results and clarify the mechanisms behind lubricant performance. Overall, the findings indicate that appropriately tailored waste cooking oil lubricants can meet demanding joining requirements while supporting more sustainable fastening processes. [DOI: 10.1115/1.4072433]

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1 Introduction

Threaded fasteners remain one of the most widespread joining solutions in mechanical systems because they combine high-load-carrying capability with reversibility: assemblies can be dismantled for inspection, repair, or reuse without necessarily damaging the connected parts. This feature is increasingly relevant in design strategies aimed at extending service life and improving end-of-life recoverability. In practice, however, repeated assembly operations can compromise joint reliability, since the tribological condition of the contact interfaces evolves with wear and surface modification over successive tightening cycles.

The preload generated in a bolted joint is governed by the applied tightening torque, but only a limited fraction of that torque is converted into useful clamping load. Indeed, a significant fraction of the input is dissipated by friction at two main locations: along the engaged threads and beneath the rotating bearing surface (bolt head or nut) [1–3]. Consequently, even modest changes in friction at either interface can lead to significant scatter in the achieved preload for a nominal torque, with direct implications for joint integrity and assembly failures [4,5]. Because friction can drift during re-tightening [6,7], owing to adhesive wear, abrasion, local heating, and transfer films, lubrication is largely adopted to protect the mating surfaces and to keep friction coefficients more stable and predictable across multiple cycles [8].

A large body of work has therefore investigated how lubrication affects the torque–tension relationship in threaded fasteners. Seminal boundary-lubrication tests by Kato et al. first clarified that lubricant choice can significantly shift the friction response in threaded contacts [9]. Building on this, Zou et al. directly quantified how mineral-oil viscosity affects friction (with lower-viscosity oils yielding higher friction) and also benchmarked grease versus oil formulations under comparable fastening conditions [10].

Croccolo et al. then linked the scatter of friction coefficients to the reliability of the torque–preload relationship in failure-oriented analyses [11], and then extended the discussion to lightweight

applications by examining how tightening procedures and lubrication conditions influence titanium screw joints [12].

To effectively enable multiple joint assembly, the most important effect of a lubricant is to preserve the relation between torque and clamping force over repeated tightening: for example, Liu et al. tracked how friction evolves cycle-by-cycle in high-strength bolts [6], while Zheng et al. investigated the corresponding loss of pre-tightening force [13]; related studies such as Zhu et al. have addressed early tension-loss mechanisms in bolted joints [14].

Within this landscape, industrial synthetic oils and molybdenum disulfide (MoS_2) greases have become a frequent benchmark in experimental investigations for their ability to reduce friction and limit wear in repeated tightening scenarios [4,10,15]. In parallel, strategies based on coatings or surface treatments have been proposed to reduce lubricant demand, although they may require redesign and can increase manufacturing cost [7].

Despite these advances, the environmental profile of conventional lubricants remains a concern, since most commercial lubricants are still derived from petroleum and can contribute to environmental contamination when released, particularly in open or loss-lubrication systems [16–18]. This has motivated a growing effort to develop bio-based lubricants derived from vegetable oils, which can offer biodegradability and improved sustainability metrics in several sectors, including automotive, manufacturing, and hydraulic systems [19–21]. The tribological performance of vegetable oils is strongly governed by molecular structure, especially fatty-acid chain length, degree of unsaturation, and functional groups, which affect viscosity, viscosity index, oxidative stability, and boundary-film formation [22–24]. Accordingly, promising results have been reported for rapeseed/canola, palm, castor, high-oleic soybean and sunflower oils, and non-edible feedstocks such as jatropha, pongamia/karanja, rice bran, and *Calophyllum inophyllum*, although chemical modification is often needed to improve oxidation stability and low-temperature behavior [24–29].

Evidence for renewable oils specifically in bolted-joint lubrication is surprisingly limited. After early observations by Kato et al., who included castor oil in the comparison with mineral

lubricants [9], systematic studies that directly benchmark vegetable oils against established mineral oils and greases in repeated tightening of threaded joints have been largely absent in the literature. Recently, the use of renewable lubricants for threaded joints has been investigated by Crococolo et al., who demonstrated how vegetable oils can outperform mineral oils in fastening zinc-plated bolts on steel plates [30]. Particularly, they compared the performance of sesame, sunflower, castor and coconut oil. In more recent work, the same group demonstrated how the effect of these lubricants changes with the nature of the mating materials [31].

However, as highlighted by the same authors, the use of virgin vegetable oil has a drawback in terms of sustainability, especially when edible oils are used [30]. Recent comparative Life Cycle Assessment (LCA) studies highlighted the advantage of waste cooking oil (WCO) over virgin oils [32–34]. To date, WCO is mainly adopted for the production of biodiesel [35–37], whose tribological behavior after subsequent partial hydrogenation has been reported [38]. An exploratory study to use as-received cooking oil as a lubricant for threaded joints undergoing high temperatures has been recently presented by Fini et al. [39].

This article presents the results of a long-term project in which new lubricants have been synthesized starting from WCO using different strategies and used for the lubrication of threaded joints, with particular reference to an automotive application. The macroscopic effects of the lubricants on the relation between tightening torque and clamping force are monitored over multiple consecutive cycles by measuring the apparent friction coefficients; besides, a microscale analysis of wear on the mating surface is conducted to provide a more in-depth understanding of the role of lubrication in threaded joints. Synthetic oils and greases were included for comparison with the literature and current industrial practice, demonstrating that the renewable alternatives proposed can achieve similar or even better properties compared to mineral lubricants.

2 Methods

2.1 Preparation of the Specimens. To enable tribological testing on the rig, custom specimens were extracted from a connecting rod by electrical discharge machining (EDM). An 8-mm-thick plate was cut from the cap and used as the bearing surface, while the entire threaded region of the shank was removed and used as the nut during testing. The connecting rod material is a quenched-and-tempered steel with a surface hardness of 41 HRC.

Table 1 Density and viscosity of oils from WCO [44]

Property	WCO	H-WCO	PO ₁₀₀
Density at 20 °C (g · cm ⁻³)	0.908	0.994	0.96
Viscosity at 25 °C (Pa · s)*	0.041	1.043	0.656
Viscosity at 40 °C (Pa · s)*	0.027	0.166	0.245
Viscosity at 100 °C (Pa · s)*	0.004	0.008	0.024
Kinematic viscosity at 40 °C (cSt)	30.34	175.1	264.58
Kinematic viscosity at 100 °C (cSt)	4.61	9.78	26.85
Viscosity Index**	38	N.A.***	133

*Value at 100 s⁻¹; ** ASTM D2270 and ISO 2909, applicable for oils with kinematic viscosity at 100 °C > 2 cSt; *** not applicable to non-Newtonian fluids.

Testing was performed using an ISO 5855-2 MJ10 × 1 screw (length 50 mm) made of quenched-and-tempered chromium–molybdenum alloy steel, strength class 14.9. The screw features a 0°2' underhead inclination.

2.2 Lubricants. Tribological testing was carried out on a focused set of lubricants spanning mineral paste and oil, and selected bio-based candidates derived from WCO. As the baseline representative of the real assembly condition, the threaded joint was lubricated with Castrol MOLUB-ALLOY Paste PL (denoted MP), a mineral paste containing dispersed MoS₂ and formulated for boundary lubrication under the low-speed/high-load conditions typical of tightening. For comparison against a widely used fully formulated lubricant, mineral engine oil, Shell Helix Racing 10 W-60, UK (denoted 10 W60), was also included.

On the renewable side, WCO dehydrated and filtered (the minimally processed feedstock) was tested to establish the performance of an easy-to-produce bio-lubricant. Then, two different approaches were used for the refinement of the WCO, namely (a) WCO partial hydrogenation and (b) WCO full epoxidation followed by ring-opening reactions leading to a WCO-derived polyol.

The hydrogenation led to H-WCO, a solid hydrogenated derivative with grease-like consistency whose handling is closer to the MP. More details on this lubricant can be found in Ref. [38].

H-WCO was obtained by catalytic hydrogenation of WCO in the presence of Raney nickel, yielding a solid grease-like product in 92% yield, as detailed in Ref. [40]. Gas-chromatographic analyses after derivatization under alkaline conditions revealed the following fatty-acid molar composition: C16:0 (10.0%), C18:0 (24.1%), C18:1 (60.0%), C18:2 (5.8%), and C20:0 (0.1%).

Finally, PO₁₀₀, a polyol obtained from fully converted epoxidized WCO and subsequent ring-opening, was investigated as a more polar, chemically functionalized bio-lubricant designed to enhance interfacial film formation and wear protection under demanding contact conditions. Detailed information on the production route for this oil was previously published in Refs. [41–43].

A complete physicochemical and rheological characterization of the WCO-derived lubricants is reported in Ref. [44]. Since these properties may affect lubricant distribution, film retention, and friction evolution during repeated tightening, the most relevant data for the lubricants investigated in this work, namely WCO, H-WCO, and PO₁₀₀, are summarized in Tables 1 and 2. These include density, dynamic viscosity at different temperatures, kinematic viscosity, viscosity index, and thermal/oxidative stability assessed by thermogravimetric analysis in air.

The data highlight marked differences among the three WCO-derived lubricants. WCO is the least viscous bio-based lubricant and behaves as a low-viscosity oil. H-WCO exhibits much higher viscosity and a grease-like consistency, which makes its handling closer to that of the benchmark MP paste. PO₁₀₀ also shows relatively high viscosity, especially at 40 °C and 100 °C, together with a high viscosity index. These properties are relevant for the tightening, since viscosity and rheological behavior can affect lubricant supply, squeezing from the contact, and persistence of the lubricating film during repeated tightening cycles.

2.3 Tightening Tests. The tightening tests were performed on a dedicated testing rig by Kistler GmbH, a schematic representation of which is shown in Fig. 1.

Table 2 Results of TGA in air for oils from WCO [44]

Property	WCO	H-WCO	PO ₁₀₀
Onset temperature (°C)	279.7	229.6	229.6
Peak temperatures (°C)	370, 414.8, 474.8	369.9, 410, 479.8	370.9, 409.4, 529.6
Residue (%)	4.36	2.48	0.76

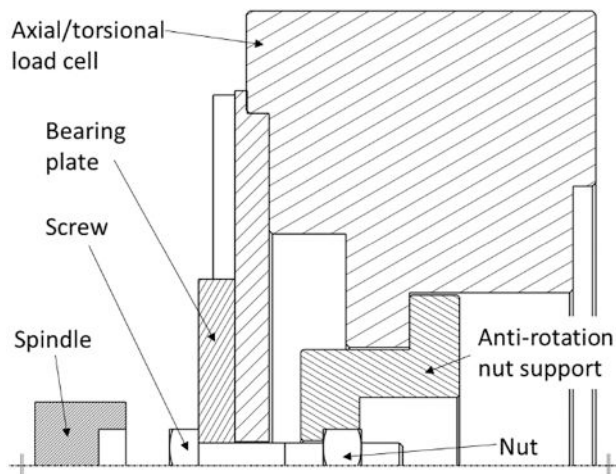


Fig. 1 Schematic representation of the tribological testing rig

In this setup, the spindle records the overall tightening torque, T . Because the plate is rigidly coupled to a torsional load cell, that sensor measures the torque transmitted through the bolt shank T_{th} , i.e., the contribution associated with thread friction. The axial load cell simultaneously measures the clamp force F , generated during tightening. The relation between the measured quantities can be expressed as in Eq. (1). [45]

$$T = F \frac{p}{2\pi} + T_{th} + T_b \quad (1)$$

where p is the screw pitch, and T_b is the bearing torque, namely the torque associated with underhead friction. Since T , F , and T_{th} are experimentally measured, T_b can be obtained from Eq. (1). The friction coefficients at the bearing surface under the head, μ_b , and in the threads, μ_{th} , can then be calculated using Eqs. (2) and (3), respectively:

$$T_b = F \mu_b R_b \quad (2)$$

$$T_{th} = F \frac{\mu_{th} R_t}{\cos \beta} \quad (3)$$

where p is the screw pitch, R_b and R_t are the average bearing and thread radius, respectively, and β is the semi-angle of the thread profile. For the tested screws, $p = 1$ mm, $R_b = 6.65$ mm, and $R_t = 4.68$ mm.

In line with previous studies on the comparison of lubricants in threaded joints [30], the tightening tests were performed by controlling the final clamping force. Although this procedure does not reproduce the most common industrial assembly strategies, which are generally torque-controlled or torque-angle-controlled, it ensures that all lubricants are compared under the same final preload and, consequently, under comparable contact pressure conditions at the thread and underhead interfaces. This makes it possible to directly compare the torque required to reach the target preload, the resulting thread and bearing friction coefficients, and the wear generated during repeated tightening.

It should also be noted that the scatter of the tightening torque measured at a fixed clamp force provides an indication of the variability of the overall tightening factor associated with each lubricant. This variability is relevant for industrial torque-controlled tightening, since fluctuations in the frictional response would directly translate into preload scatter under a fixed applied torque. Therefore, the present results should be interpreted as a comparison of lubricant tribological performance and tightening stability under controlled-preload conditions, rather than as a direct validation of preload scatter under fixed-torque industrial assembly. Further torque-controlled or torque-angle-controlled

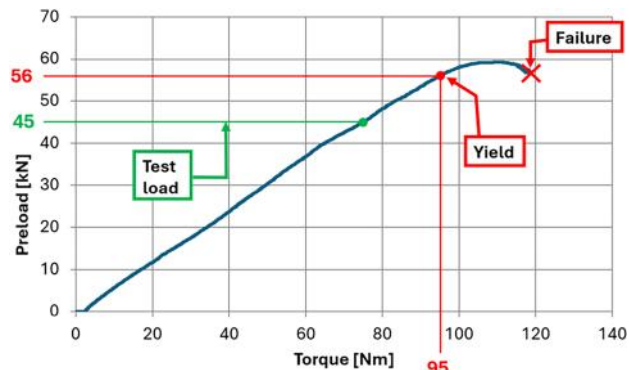


Fig. 2 Torque versus preload plot of preliminary failure test

tests would be required for full validation under production conditions.

To determine the force to be applied during the tightening test, preliminary tests were carried out by tightening the joint until screw failure. In the scope of these pretests, the joint was lubricated with MoS₂-containing grease MOLUB-ALLOY Paste PL by Castrol, to replicate the actual tightening conditions. Figure 2 shows a representative plot of this pretest.

These tests demonstrated that the yield of the screw occurs at $F_y = 56$ kN and $T_y = 95$ kN, that is to say an equivalent von Mises stress $\sigma_{y,VM} = 1100$ MPa. The target clamping force was then set to $F = 45$ kN, namely 80% of F_y . The choice is intended to ensure that the screw remains in the elastic regime at the first tightening, also considering that renewable lubricants may exhibit higher friction coefficients than the reference synthetic grease.

For each test run, the bolt was initially seated manually until the head contacted the plate and then backed off by three full turns (1080 deg). After a 0.5-s synchronization delay, a pre-tightening step was performed at 20 rpm to reach a preload of 15 kN. Following a 2-s dwell, the joint was tightened at a constant speed of 10 rpm until the target preload of 45 kN was attained. This force was maintained for 2 s, after which the joint was untightened at 10 rpm. A 20-s pause was introduced between consecutive cycles to mitigate frictional heat accumulation. The protocol represents an adaptation of the Volkswagen standard VW 01131-1[46].

Prior to testing, each fastener was manually lubricated on both the bearing surface and the threaded region according to a standardized procedure. Paste/grease-like lubricants, namely MOLUB-ALLOY Paste PL and H-WCO, were applied with a flat blade and spread over the threads and underhead bearing surface to obtain a continuous visible layer, ensuring that the lubricant reached the thread roots and the whole bearing contact region. Liquid lubricants, namely WCO, 10 W60 and PO_100, were applied by dipping the screw into the lubricant; the screw was then withdrawn and kept upside down, with the shank facing upward, to avoid lubricant drainage from the underhead bearing surface. The lubricated screw was assembled on the testing rig within 5 s after application. The same operator, sequence, and timing were used for all specimens. No attempt was made to impose the same lubricant mass or nominal film thickness for all materials, because the tested lubricants have markedly different consistencies and retention behavior. Instead, the procedure was chosen to reproduce a realistic industrial lubrication practice. The different tendency of each lubricant to spread, remain on the surfaces, and be displaced during tightening was therefore considered part of its practical effectiveness.

Three bolts were tested for each lubricant, and five consecutive tightening cycles were performed on each bolt without re-lubrication, in accordance with established practice in the literature [8,12,30]. The evolution of friction coefficients and generalized forces over successive cycles was recorded.

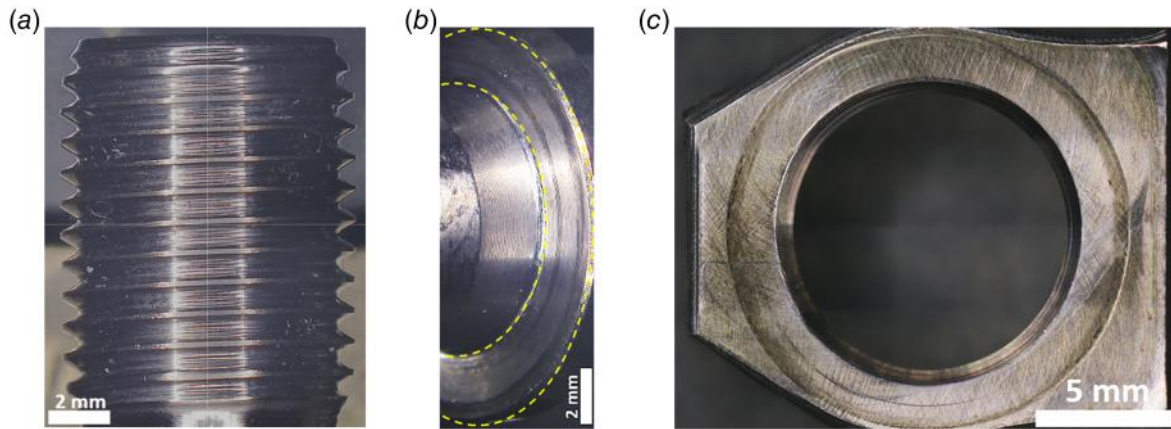


Fig. 3 Representative images of investigated surfaces (before tightening): (a) screw threads, (b) screw underhead; (c) connecting rod underhead (bearing plate)

2.4 Wear Assessment. Before and after the tightening tests with the different lubricants, the surfaces of the mating components (Fig. 3) were investigated by profilometry (chromatic confocal profilometer, Nanovea JR25, USA), 3D digital optical microscopy (3D-OM, Hirox HRX-01, Japan), and field-emission gun scanning electron microscopy (FEG-SEM, Tescan Mira 3, Czech Republic) coupled with energy-dispersive spectroscopy (EDS, Bruker XFlash 630M, USA), focusing on the screw threads and bolt underhead surfaces (Figs. 3(a) and 3(b)), as well as the bearing plates (Fig. 3(c)). Three screw/plate couples per lubricant were analyzed to obtain statistically relevant results.

A tailored experimental protocol was developed to analyze the surface damage in threaded joints after multiple tightenings. To ensure the cleanliness of both screws and plates, ultrasonic cleaning in isopropyl alcohol was performed before and after testing. Being aware that cleaning may remove weakly adhered lubricant residues, loose debris, or poorly attached tribofilms, we compared worn surface morphology before and after cleaning: the results indicated that, in this case, the main morphological features were preserved.

The screw threads were first analyzed by 3D-OM and FEG-SEM for qualitative surface morphology evaluation. For quantitative

wear assessment, two threads in each screw (n. 2 and n. 9 counting down from the screw head) were selected as representative of the most and least mechanically stressed parts by the tightening process [47], respectively. The selection of these threads was based on a systematic SEM screening of all threads. For each specimen, all threads were imaged in left and right-side views and in front view, both at low and medium magnification. The specimen was then rotated by 90 deg, and the imaging sequence was repeated to minimize orientation-dependent bias and to ensure that damage features along the full screw thread were captured. Based on a visual inspection of the whole SEM dataset, threads 2 and 9 were selected for quantification due to the most reproducible damage morphologies across specimens and lubricants. Based on the above and in line with the FEM results in Ref. [46], thread 2 was selected as representative of an early, highly loaded engagement region, whereas thread 9 was chosen as representative of a later, lower-load position; together they capture two distinct loading regimes ensuring reliability and repeatability in wear assessment. The topographies of the selected threads were acquired by 3D-OM and processed using Mountains software. Two main features were considered for comparisons: (i) groove worn area (GWA, Fig. 4(a)) and (ii) plastically deformed area (PDA, Fig. 4(b))

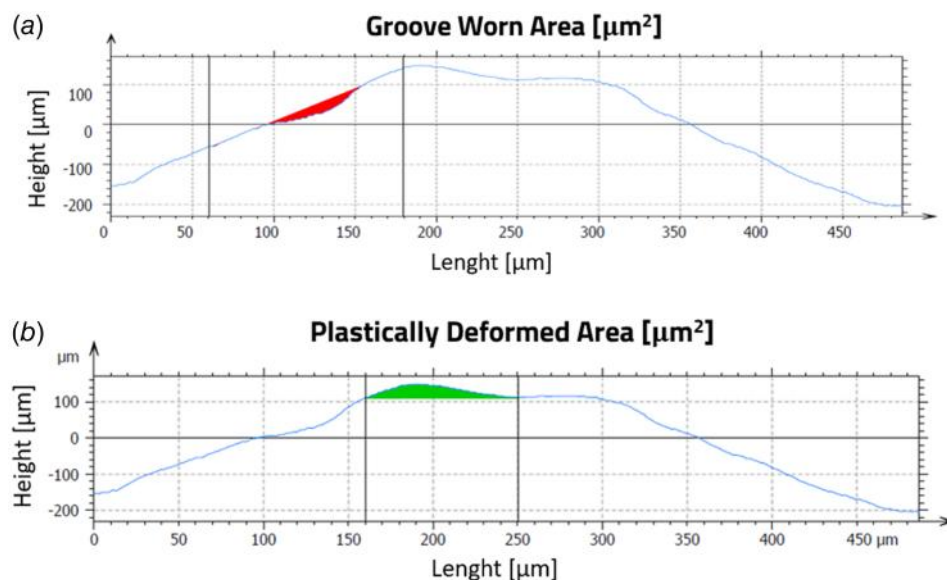


Fig. 4 Representative line profiles (3D-OM) for wear assessment of the screw threads: (a) area of the wear groove (GWA) in the thread grip zone; (b) portion of plastically deformed material (PDA) due to tightening

Fig. 4(b)) at the edge of the thread. Wear was quantified by averaging values from two 90-deg positions for each selected thread.

Upon completion of the tightening tests, selected screws were sectioned to remove the shank, providing direct access to the screw underhead region for subsequent characterization. The screw underheads and bearing plates underwent surface-roughness characterization by profilometry. Four cross-oriented topographies, rotated by 90 deg, were considered for each bearing-plate UH (Fig. 5(a)), while two cross-oriented topographies were considered for the screw UHs (Fig. 5(b)).

Within each zone, 2–3 microscale surface topographies corresponding to regions of interest (ROIs) of $500\ \mu\text{m} \times 500\ \mu\text{m}$ were extracted. For the nut UHs, two ROIs were acquired (Fig. 6(a)): (i) internal (INT, closest to the pin hole of the UH); (ii) external (EXT, in the outer part of the connecting rod surface, usually damaged most quickly during service [48]). In the case of the screw UHs, three ROIs were considered (Fig. 6(b)): (i) INT, closest to the shank; (ii) MID—between INT and EXT; (iii) EXT—in the outer part of the screw UH. Each ROI was acquired at the same position before and after testing. A more severe region is expected in the EXT regions due to the inclination of the screw UH mentioned in Sec. 2.1.

The ROIs were acquired by using a scan step of $5\ \mu\text{m}$ in X and Y coordinates, with a scan frequency of 1000–2000 Hz, depending on source-light intensity, and an averaging factor of about 15. Each full raster corresponded to 100 profile scans per specimen. The average values of the surface-roughness parameters S_q , S_p , S_v , and S_z (ISO 25178-2:2012 [49]) were extracted for each ROI.

The root mean square height parameter (S_q) was used as the main wear index.

Measurements of the angle between the shank and the UHs were performed by cross-sectioning the screws to evaluate potential changes resulting from stresses induced by the tightening torque. Cutting reproducibility was achieved by a clamping frame; the sections were mounted in epoxy resin and polished by conventional metallographic procedures. Measuring the underhead angle required destructive metallographic cross-sectioning. To limit the number of destructive tests while retaining statistical relevance, one representative specimen was analyzed for each lubrication condition, and results were compared to a reference specimen “before tightening”. To minimize uncertainty introduced during sectioning, UHs were first cut out from the shank using a low-speed cutting procedure to produce a flat reference surface. The UH was then clamped in a vice with full flush contact between the jaws and the underhead extremities to prevent tilt and ensure an orthogonal (square) cut. Sectioning was performed as close as possible to the geometric mid-plane of the underhead to avoid diagonal cuts that would bias the measured angle. Afterwards, two independent cross sections per specimen were prepared by mounting in epoxy resin and polishing by conventional metallographic procedures. On each cross-section, the UH angle was measured on both sides of the profile, resulting in 4 measurements per specimen. The UH angle was defined and measured by ImageJ software on the polished cross sections. Polished cross sections were analyzed by OM, and shank/UH angles were measured by ImageJ/Fiji Software. Subsequently, screw cross sections were also used to

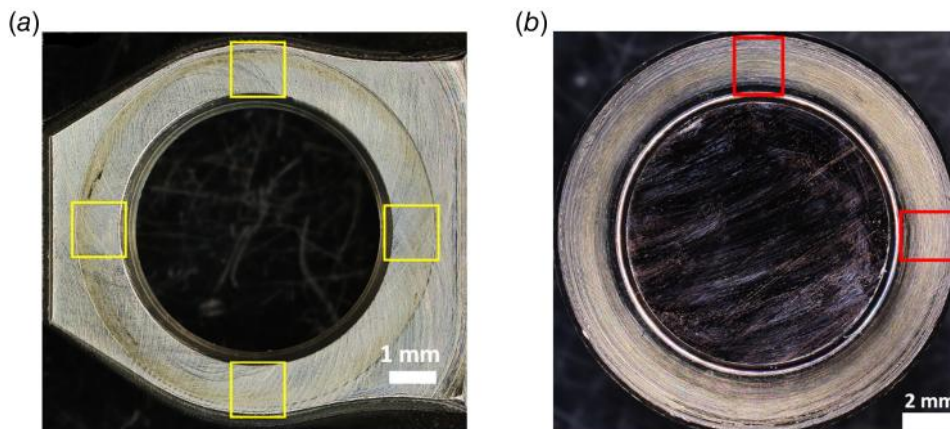


Fig. 5 Examples of area sampling for (a) bearing-plate UHs and (b) screw UHs

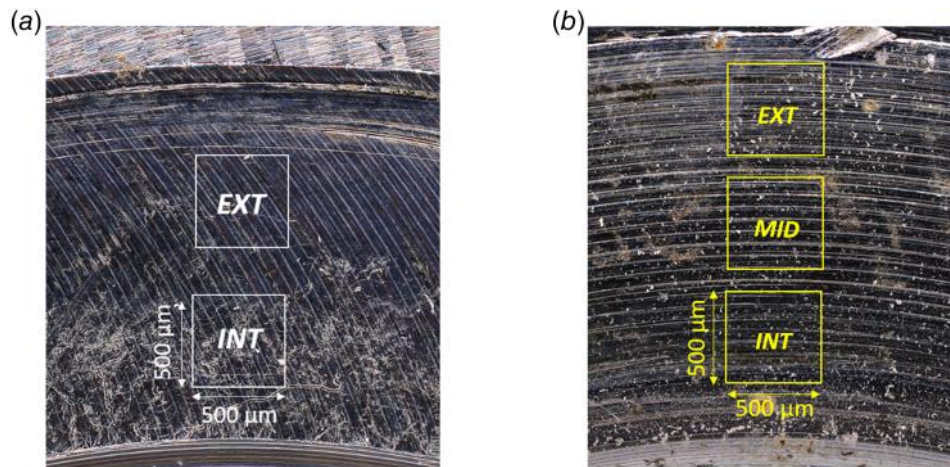


Fig. 6 Examples of UH area sampling: (a) bearing plate; (b) screw

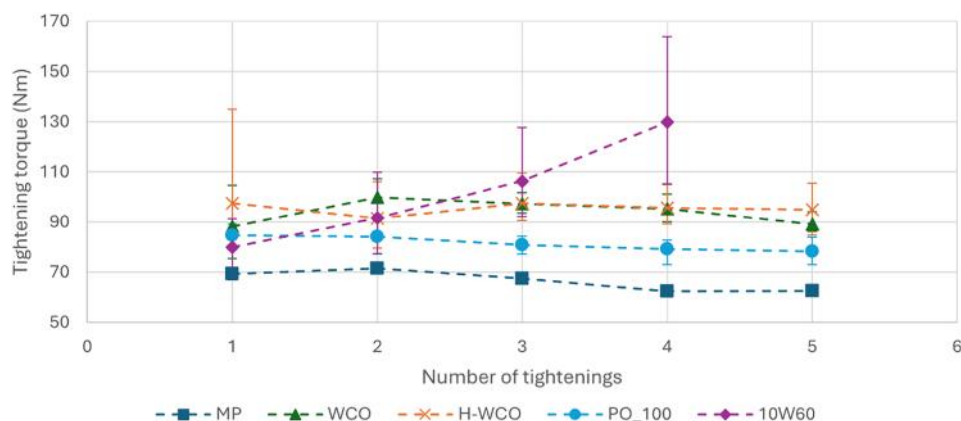


Fig. 7 Average tightening torque at consecutive tightening cycles for different lubricants (error bars represent minimum and maximum values)

Table 3 Maximum torque and equivalent stress on the screw

		Tightening cycle				
		1	2	3	4	5
MP	T (Nm)	69.3	71.5	67.4	62.2	62.6
	σ_{VM} (MPa)	839	854	828	795	797
H-WCO	T (Nm)	97.5	91.5	97.4	95.6	94.9
	σ_{VM} (MPa)	1035	991	1034	1020	1016
WCO	T (Nm)	88.1	99.8	97.4	95.2	89.0
	σ_{VM} (MPa)	967	1052	1033	1018	973
PO_100	T (Nm)	84.7	84.1	80.8	79.2	78.2
	σ_{VM} (MPa)	943	939	916	905	899
10 W60	T (Nm)	79.9	91.6	106.3	129.9	125.3*
	σ_{VM} (MPa)	909	991	1100	1273	1232*

*Values at failure.

reveal microstructural and micro-mechanical changes induced by tightening: etching by Nital 2 (2% HNO_3 in ethanol), observation via OM (Zeiss Axioscope 7, Germany), and FEG-SEM/EDS and microhardness measurements ($\text{HV}_{0.02}$, ISO 6507-1:2023 [50]) were carried out. $\text{HV}_{0.02}$ measurements were conducted only on the best- and worst-performing screws in each lubricant category (i.e., oil and grease), by indenting both the bulk and two random locations underneath the wear track [50].

3 Results and Discussion

3.1 Results of Tightening Tests. Figure 7 shows the evolution of tightening torque over repeated tightening cycles for the different lubricants; the corresponding average values are reported in Table 3 together with the equivalent von Mises stresses.

The equivalent von Mises stresses reported in Table 3 confirm that, during the first tightening cycle, all lubricants remained below the yield limit identified in the preliminary tightening-to-failure tests, supporting the selection of the 45-kN clamp load for the experimental campaign. This is consistent with the preliminary-test rationale, where the selected clamp load was chosen to maintain a safety margin with respect to yielding.

Table 4 reports the ranges of thread, bearing, and total friction coefficients. Due to the limited number of tests and to better highlight the actual scatter of the tests, the minimum and maximum values recorded in testing are reported.

As shown in Fig. 7, MP exhibits highly stable performance over repeated tightening cycles and requires the lowest tightening torque. Consistently, Table 4 indicates comparable values of the

Table 4 Range of friction coefficients at different tightening cycles

Lubricant	Tightening	μ_{th} min	μ_b max	μ_{tot}			
				min	max	min	max
MP	1	0.11	0.12	0.11	0.12	0.11	0.12
	2	0.11	0.11	0.12	0.12	0.12	0.12
	3	0.11	0.11	0.11	0.11	0.11	0.11
	4	0.10	0.10	0.10	0.10	0.10	0.10
	5	0.09	0.11	0.10	0.10	0.10	0.10
H-WCO	1	0.13	0.22	0.10	0.25	0.11	0.24
	2	0.16	0.21	0.11	0.16	0.13	0.18
	3	0.18	0.22	0.13	0.16	0.15	0.19
	4	0.18	0.21	0.13	0.15	0.15	0.18
	5	0.18	0.21	0.12	0.16	0.14	0.18
WCO	1	0.14	0.20	0.10	0.18	0.13	0.18
	2	0.18	0.19	0.12	0.18	0.14	0.18
	3	0.15	0.19	0.14	0.18	0.16	0.17
	4	0.15	0.18	0.16	0.17	0.15	0.17
	5	0.14	0.16	0.14	0.16	0.15	0.16
PO_100	1	0.15	0.18	0.12	0.14	0.13	0.15
	2	0.14	0.17	0.12	0.14	0.13	0.15
	3	0.14	0.16	0.12	0.13	0.13	0.14
	4	0.13	0.15	0.11	0.13	0.12	0.14
	5	0.13	0.15	0.11	0.13	0.12	0.14
10 W60	1	0.14	0.18	0.10	0.14	0.12	0.16
	2	0.15	0.20	0.11	0.18	0.13	0.19
	3	0.19	0.22	0.12	0.22	0.16	0.22
	4	0.21	0.23	0.16	0.22	0.18	0.22
	5	—	—	—	—	—	—

thread and bearing friction coefficients (μ_{th} and μ_b), confirming that MP is effective in both the underhead and threaded interfaces. This outcome is expected: as noted in the Introduction, MP is formulated for high-load, low-speed operation, and was therefore selected as the reference lubricant.

By contrast, the behavior of 10 W60 deteriorates markedly with increasing cycle number. This trend is corroborated by ν , where both μ_b and μ_{th} rise sharply during multiple tightenings. The stress increase observed in Table 3 for this lubricant in the later cycles, up to 1273 MPa, exceeds the yield limit and highlights the progressive loss of lubrication efficiency under repeated tightening. Notably, friction data for the fifth consecutive tightening with 10 W60 are not reported in Table 3 because this tightening cycle could not be completed. All three screws lubricated with 10 W60 failed before reaching the target clamping force, at

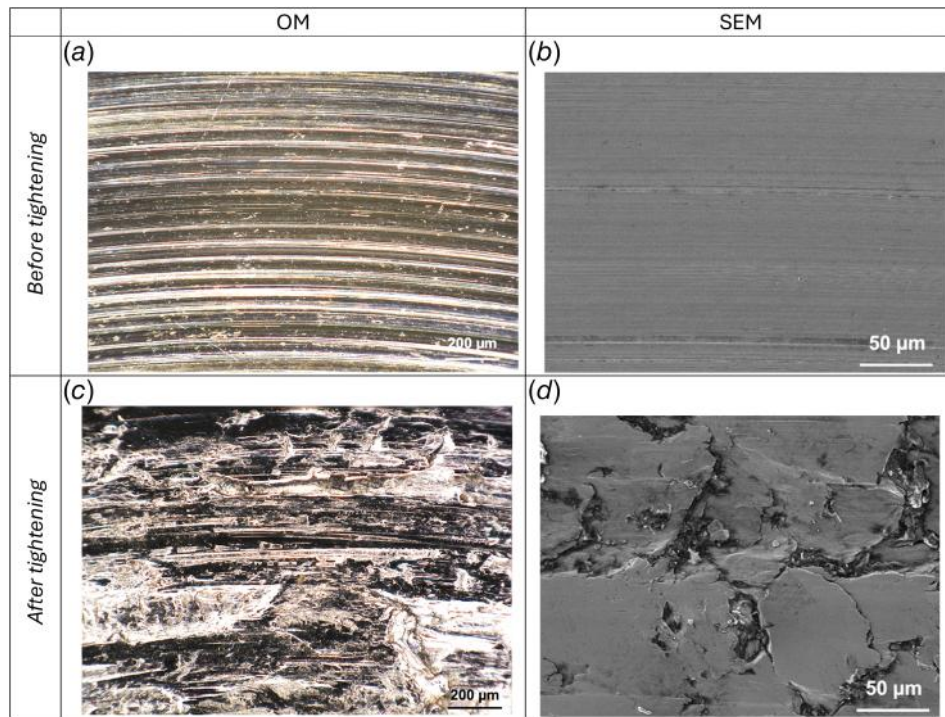


Fig. 8 Images of the screw UHs before and after tightening using mineral oil (10 W60): (a) and (c) Optical images, (b) and (d) SEM images

clamping forces between 40 kN and 43 kN. The corresponding average tightening torque and equivalent stress are reported in Table 3. The failure was not caused by fracture of the screw shank or threaded region, but by severe adhesion phenomena (sticking/galling) at the screw underhead/bearing-plate interface. This produced plastic deformation of the screw head, preventing further tightening and requiring drilling of the screw head for removal without damaging the mating surface. Figure 8 compares the bearing-plate underhead surfaces before tightening (Figs. 8(a) and 8(b)) and after tightening followed by disassembly (Figs. 8(c) and 8(d)), showing the severe surface damage associated with the observed failure mode. This finding suggests that 10 W60 is unsuitable for the load and speed conditions characteristic of tightening.

The WCO-derived lubricants show intermediate performance between MP and 10 W60, but their response depends strongly on the adopted synthesis route. WCO and H-WCO yield similar friction coefficients, which remain substantially higher than those obtained with MP. Although their average trends over successive cycles appear relatively stable in Fig. 7, the wide error bands indicate limited repeatability and increased uncertainty. This variability may be attributed to non-uniform lubricant distribution within the contact regions and progressive lubricant loss during cycling.

The comparison of tribological results with those of Table 1 shows that viscosity alone does not determine the tribological response. Although H-WCO is the most viscous WCO-derived lubricant, it does not provide the most stable friction behavior. Conversely, PO₁₀₀ shows the most repeatable response among the renewable lubricants, displaying a more stable and repeatable evolution of friction in both the bearing and thread regions. This behavior may be tentatively associated with the presence of hydroxyl groups, which could promote interactions with metal-oxide species on the steel surface, improving the lubricating action. In particular, the hydroxyl (–OH) groups of the fatty chains can coordinate with metal atoms on the contact surface or interact through weak van der Waals forces, thus promoting stronger retention of the lubricant film on the surfaces involved. However, this interpretation remains hypothetical, as direct

surface-chemical analyses such as XPS, and FTIR spectroscopy were not performed in the present study.

Accordingly, the torque levels observed for PO₁₀₀ in Fig. 7 are only slightly higher than those measured for MP, despite MP being a purpose-designed lubricant for this application. This result highlights the potential of epoxidation-derived routes to produce high-performance, sustainable lubricants suitable for demanding tightening conditions. This point is even more compelling when considering that MP contains a dispersion of MoS₂, which is highly effective in improving tribological performance under low-speed, high-load conditions. Accordingly, the inclusion of solid lubricating additives in PO₁₀₀ could represent a promising direction for future research.

3.2 Wear Assessment. The results are presented both from a morphological point of view and in terms of quantitative assessment for each of the analyzed zones, progressing from screw threads to bolt head and nut bearing plates. Figure 9 presents representative low-magnification SEM micrographs of the first and last three screw threads, both before and after testing with each lubricant. Figure 10 provides high-magnification SEM Images illustrating the surface morphology and topography of threads selected for detailed analysis (n. 2 and n. 9, counting down from the screw head) according to the protocol described in Sec. 2.4.

Microscopic examinations revealed that the first threads engaged in contact generally exhibited the highest wear (Figs. 9 and 10), as the contact pressure and maximum stresses are predominantly distributed over the first six threads, according to [51]. A comparison between threads n. 2 and 9 indicates that all samples at the n. 2 position exhibited varying degrees of surface morphology changes. The observed damage ranged from mild to pronounced burr formation on the threads to severe alterations in surface morphology, which in extreme cases (e.g., H-WCO—Figs. 10(c), 10(i), 10(o), and 10(u)) resulted in significant geometry changes compared to the pre-tightening condition. The most extensive damage was concentrated at the edges of the screw threads and was likely caused by peak contact pressures arising from the abrupt termination of material on one side of the contact pair

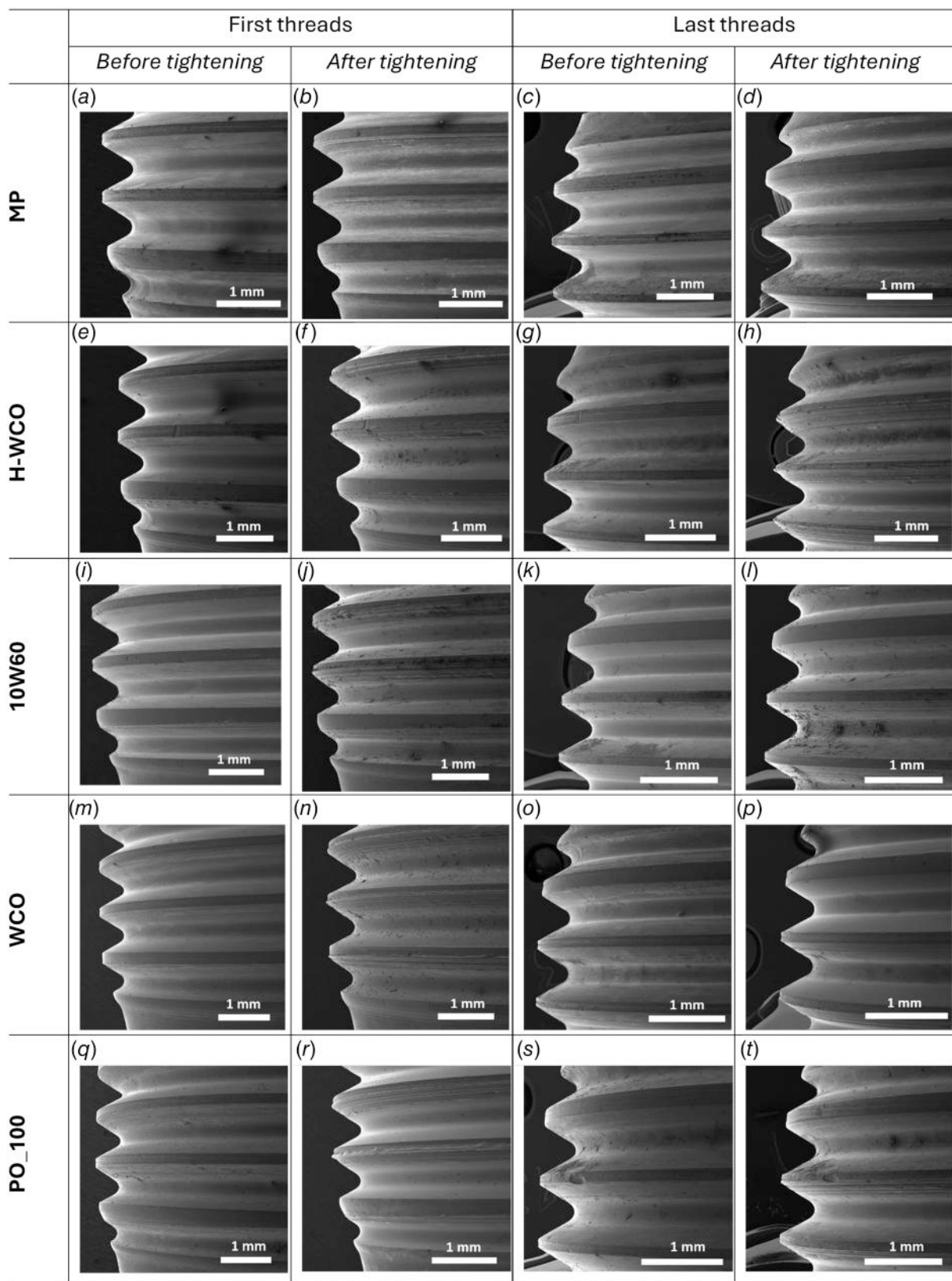


Fig. 9 SEM micrographs of first and last screw threads before and after tightening using: MOLUB paste (MP) grease, solid hydrogenated (H-WCO) grease, mineral oil (10 W60), WCO dehydrated and filtered (WCO), and polyol 100% (PO_100)

[52]. H-WCO was the only lubricant that caused severe damage extending up to thread n. 9. Quantitative information on thread damage is reported in Fig. 11, where groove worn area (GWA, a) and plastically deformed area (PDA, b) at the edge of the

thread, measured by 3D-OM, are compared. Most of the WCO-derived lubricants showed intermediate performance between MP (benchmark grease, best performer) and 10 W60 (benchmark mineral oil, worst performer), except for H-WCO, which was

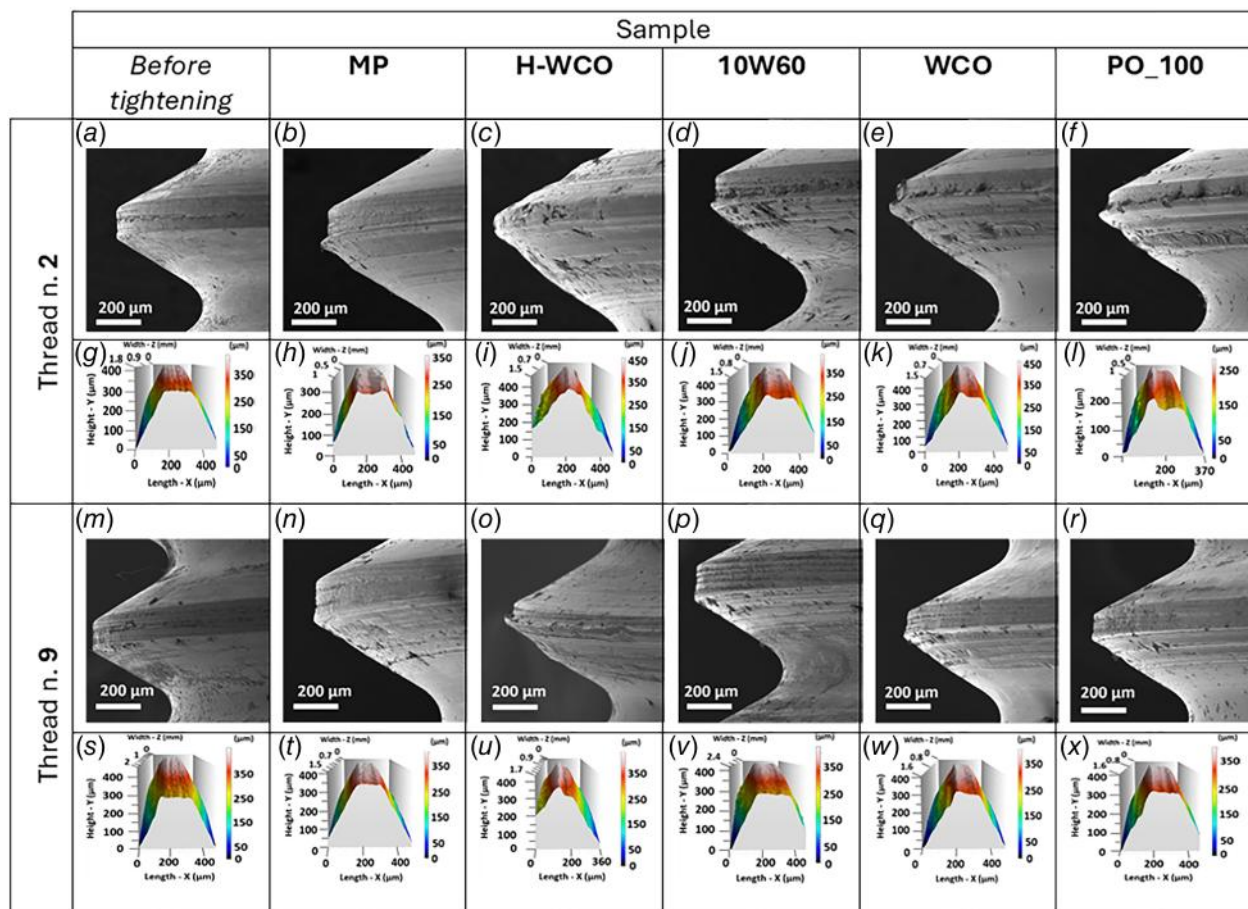


Fig. 10 Morphology of the first (n. 2) and last (n. 9) screw threads: SEM images before (a, m) and after tightening using: (b, n) MP; (c, o) H-WCO; (d, p) 10 W60; (e, q) WCO; (f, r) PO_100 (g–l, s–x). Color height scale topographies: before (g, s) and after tightening using: (h, t) MP; (i, u) H-WCO; (j, v) 10 W60; (k, w) WCO; (l, x) PO_100.

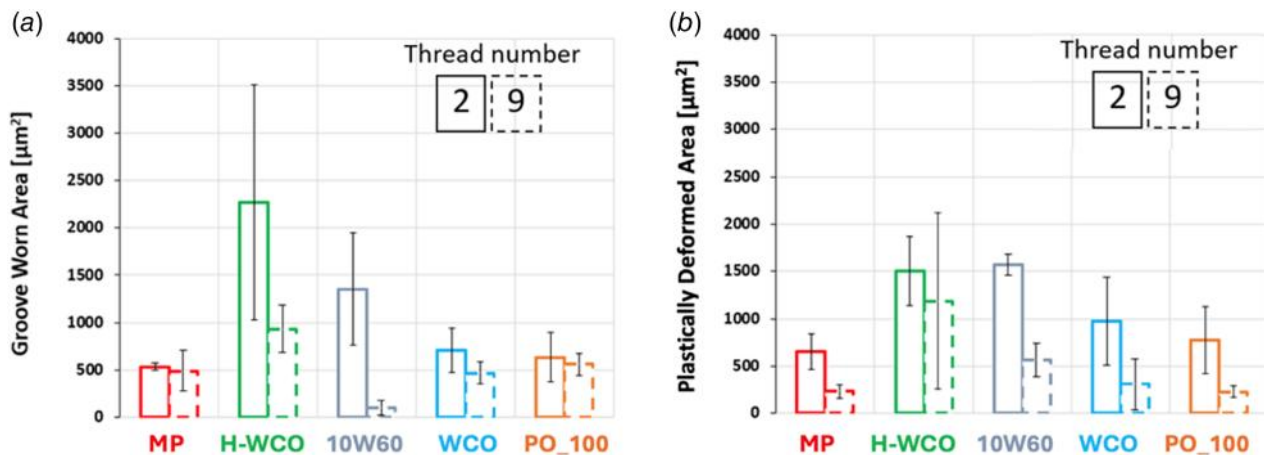


Fig. 11 Histograms showing (a) groove worn area (GWA) and (b) plastically deformed area at the screw thread edge (PDA) for each lubricant formulation, considering thread n. 2 (solid line) and n. 9 (dashed line)

unable to protect the threads from extensive wear damage. The quantitative data in these histograms also confirm the prevalence of damage in the first threads (e.g., n. 2) rather than in the last ones (e.g., n. 9), as previously discussed.

Regarding the underhead (UH) regions of screws and nuts, Fig. 12 compares the root mean square surface-roughness (S_q) variation, measured in different regions of interest (ROIs, see Fig. 6

for the location of each ROI) before and after tightening for each tested lubricant, whilst Fig. 13 displays the morphology of the whole surfaces of bearing-plate UHs as a function of lubricant type.

Profilometric data from nut UHs (Figs. 12(a)–12(c)) revealed an increasing S_q trend (directly related to wear damage) from internal (b) toward external (c) ROIs, leading in both cases to values higher than in the area not involved in the contact (a). A comparable

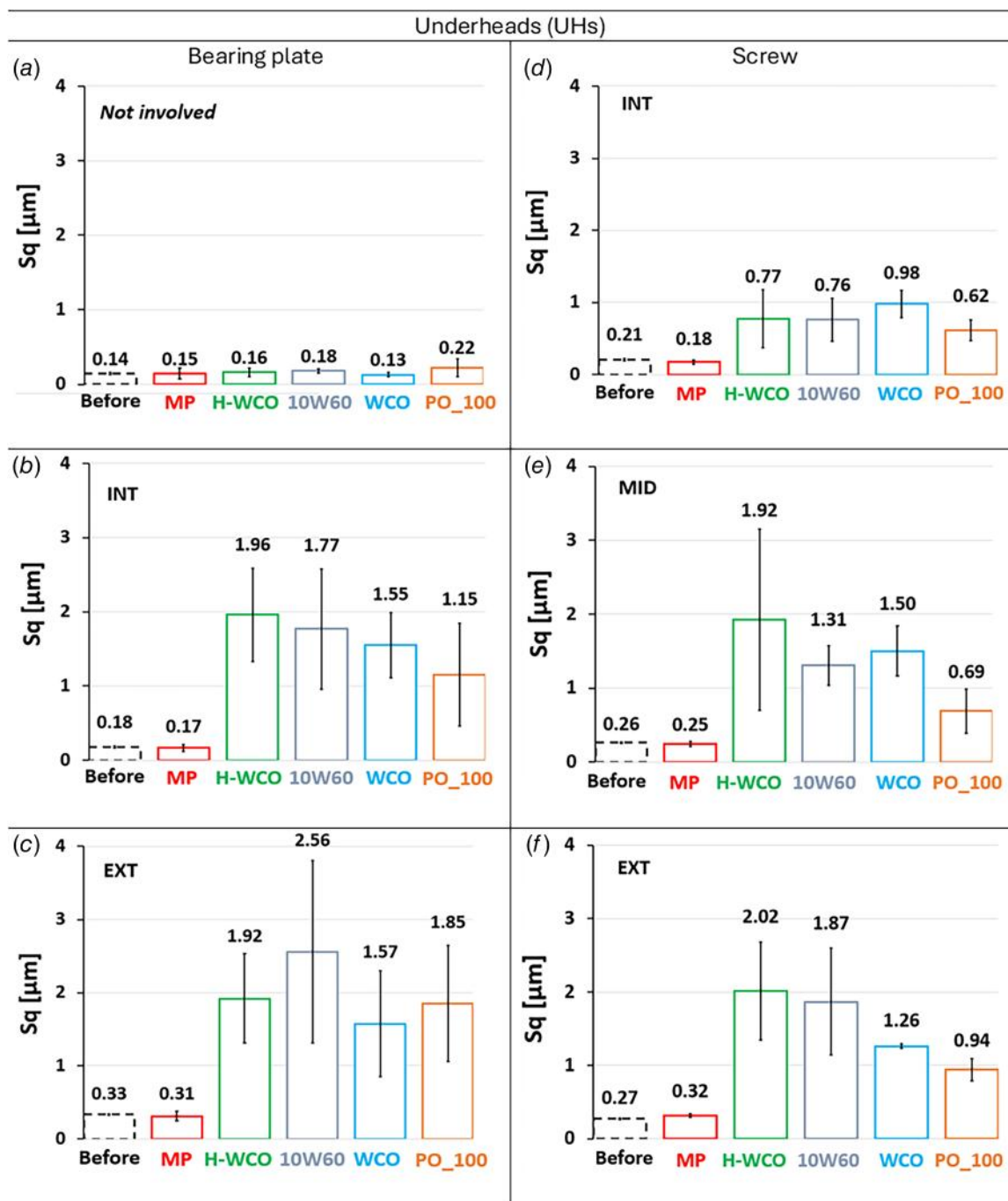


Fig. 12 Screw and nut underheads (UHs): root mean square surface-roughness (S_q) comparison in each Region of Interest (ROI); see Fig. 6 for indications about the location of ROIs. Bearing-plate UHs: (a) not involved in the contact, (b) INT, (c) EXT. Screw: (d) INT, (e) MID, and (f) EXT

pattern was observed for the screw UHs (Figs. 12(d)–12(f)), where the highest S_q values were measured in the middle (e) and external (f) ROIs. This trend is expected due to the inclination of the screw UH surface (see Sec. 2.1), which produces a higher pressure in the external region of the contact surface. The extension to the middle region is due to the progressive formation of localized intrusions and protrusions (i.e., grooving), which originate at the initial contact surface and extend to the subsequent regions as tightening progresses, as observed also by [48].

The benchmark grease MP induced minimal surface modifications after tightening, both in the nut and screw UHs (Fig. 12). In fact, the typical grinding marks from the manufacturing

process remain unaltered in the bearing plates after tightening (Figs. 13(a) and 13(b)).

Among the WCO-based alternative lubricants, WCO and PO_100 performed better in terms of wear reduction (lower increase of the S_q value) compared to H-WCO. The differences among alternative lubricants became particularly evident in the internal region of the connecting rod UHs (Fig. 12(b)), and the middle and external regions of the screw UHs (Figs. 12(e) and 12(f)). More specifically, PO_100 exhibited the best anti-wear performance among all alternative lubricants, even outperforming 10 W60, and resulted in reduced damage on the UH surfaces from the initial contact region through to the internal areas.

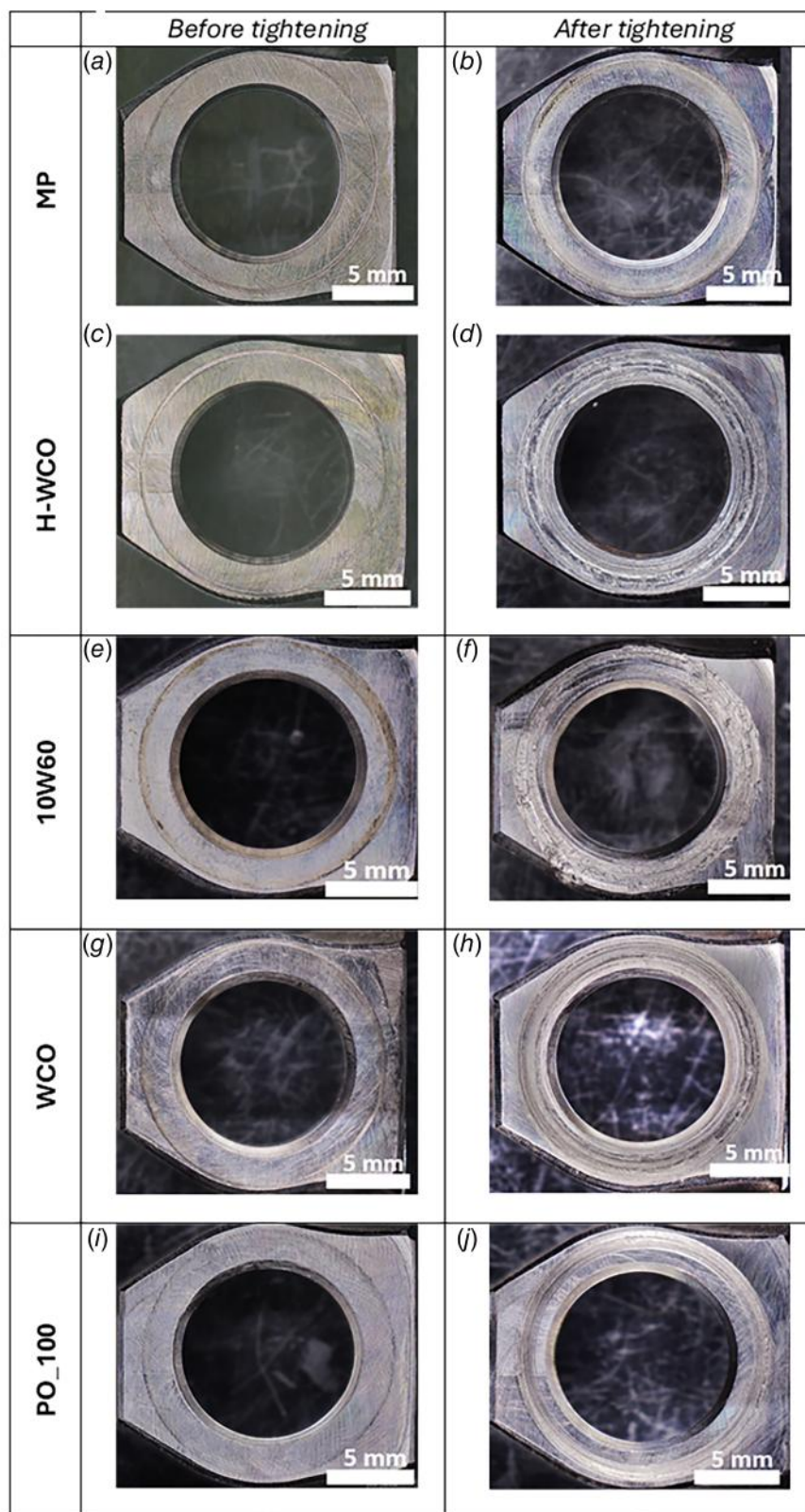


Fig. 13 Optical macrographs of the bearing-plate (connecting rod) UHs before and after tightening, using (a, b) MP; (c, d) H-WCO; (e, f) 10 W60; (g, h) WCO; and (i, j) PO_100

WCO was more effective than PO_100 in wear protection only in the external ROIs of the connecting rods (Fig. 12(c)); however, the comparable standard deviations between the two oils indicate that no clear distinction can be made, likely because the contact

pressure in this region was too high to differentiate the performance of the alternative lubricants. The worst-performing lubricants (i.e., H-WCO and 10 W60) exhibited the highest standard deviations, indicating uneven lubricant distribution on the mating surfaces

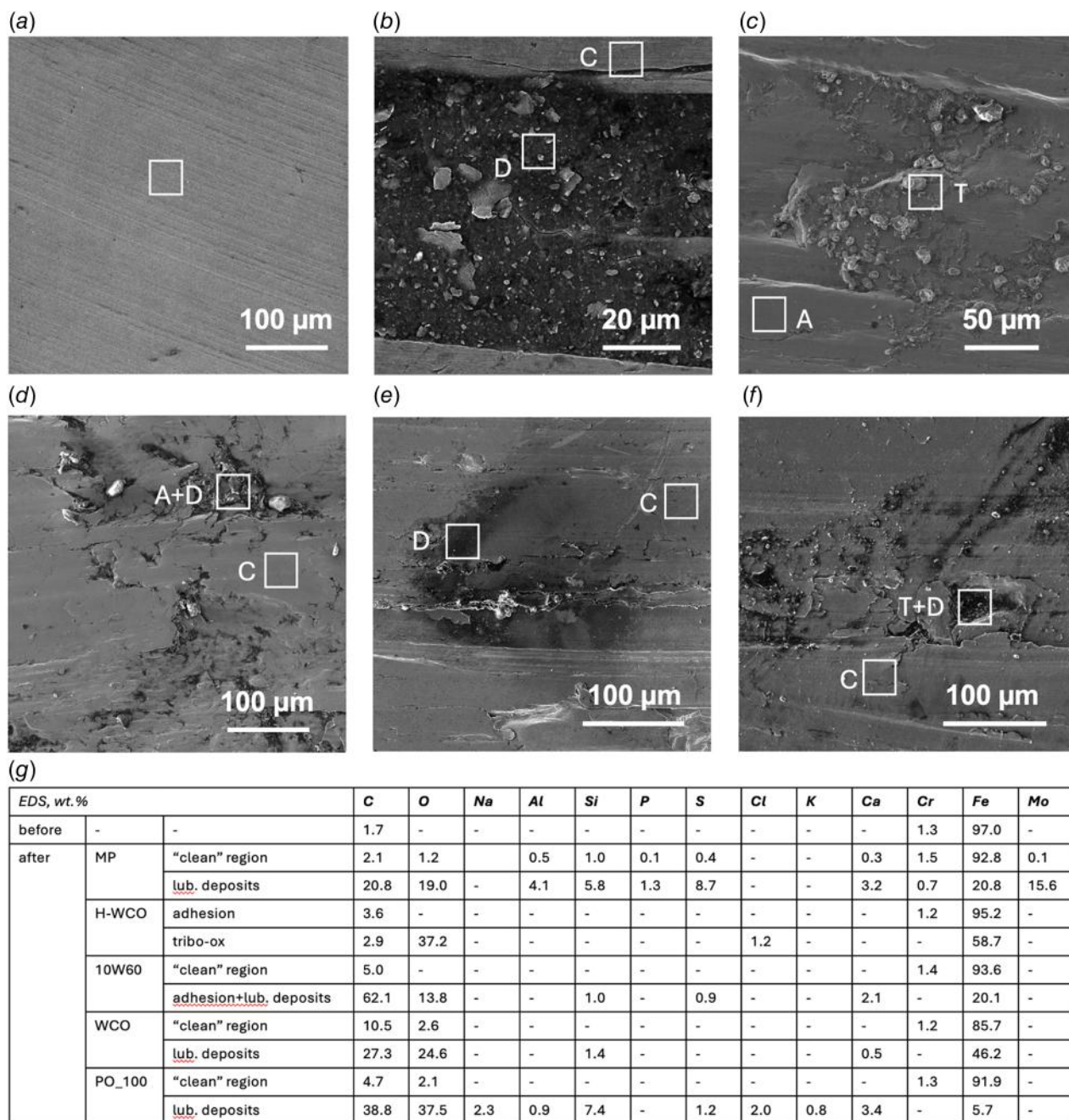


Fig. 14 SEM images (a–f) and localized elemental composition (g: EDS, wt%) of nut UHs: (a) before tightening, (b) MP, (c) H-WCO, (d) 10 W60, (e) WCO, (f) PO_100. The white squares indicate the location of EDS analyses, with the corresponding wt% reported in (g). A: region where severe adhesion prevails; T: region where mild tribo-oxidation prevails; C: "clean" region; and D: region with lubricant deposits.

and, consequently, large variability in surface roughness among repeated tests.

Macroscopic optical examination (Fig. 13) revealed that all nut UH surfaces showed varying degrees of edge wear. This localized degradation was primarily attributed to the limited extension of the initial contact region between the mating surfaces, which is expected to undergo the most rapid damage during testing [48]. However, Fig. 13 shows that for WCO (Fig. 13(h)) and PO_100 (Fig. 13(j)) only mild tribo-oxidation/abrasion was observed, whilst severe adhesive wear marks were detected on the surfaces lubricated with H-WCO (Fig. 13(d)) and, even more evidently, with the 10 W60 mineral oil (Fig. 13(f)).

SEM/EDS Analyses were also performed to assess the morphology and localized composition of the nut UH surfaces (Fig. 14).

Generally, in all these images, the darkest areas correspond to the presence of light elements, such as C, showing the distribution of lubricant deposits. These results further confirmed that both WCO (Fig. 14(e)) and PO_100 (Fig. 14(f)) maintained the system within a mild wear regime, with some areas still covered by deposits containing the lubricant marker elements (C and O, Fig. 14(g)). In contrast, the surface lubricated with 10 W60 (Fig. 14(d)) exhibited pronounced plastic deformation associated with severe adhesive phenomena. H-WCO (Fig. 14(c)) also failed to prevent adhesion and material transfer, although a few regions showing evidence of mild tribo-oxidation were still observed. Finally, the surfaces lubricated with the benchmark MP grease (Fig. 14(b)) remained extensively covered by lubricant deposits, also containing the dichalcogenide additive marker elements Mo and S (Fig. 14(g)), which contributed

to the superior performance of this grease compared with the other investigated lubricants.

To assess the ability of lubricants to protect the system from undesired geometrical variations (i.e., potential changes resulting from stresses induced by the tightening torque), the angle between the shank and the screw underheads was measured before and after tightening (Fig. 15).

Both WCO and PO_100 prevented changes in the shank–underhead angle after tightening, maintaining values comparable to both

the pre-tightening condition and those obtained with the benchmark MP grease.

Cross sections of the screw UHs were investigated both beneath the wear track and in the bulk, to compare the microstructural modifications induced by tightening with the tested lubricants.

Figure 16 shows the screw head before tightening: typical flow lines resulting from the forming process are visible in the macrograph of Fig. 16(a), whilst the bulk microstructure is characterized by tempered martensite (Figs. 16(b) and 16(c)).

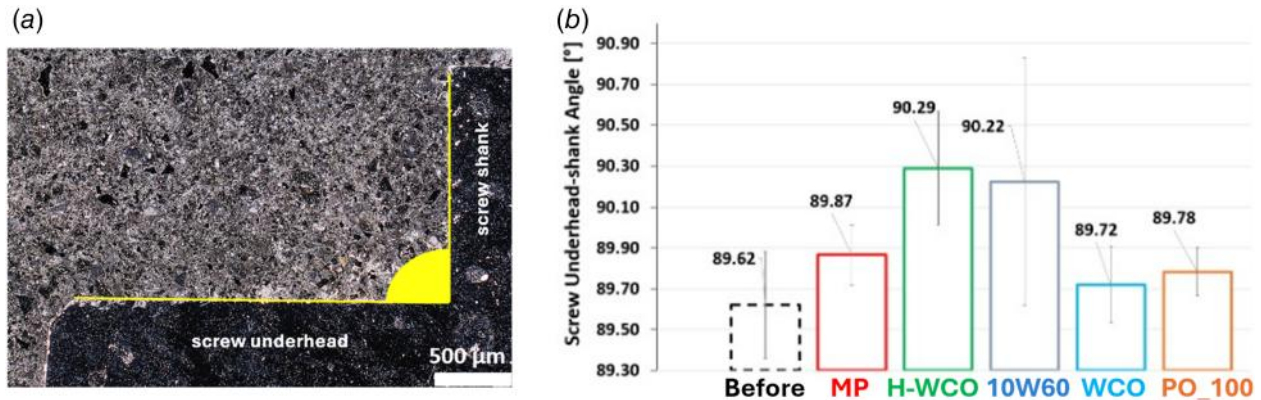


Fig. 15 (a) Representative example of the underhead angle measurement on a polished metallographic cross-section (illustrative only), (b) comparison between the angles measured before and after tightening using different lubricants (average values $\pm$ standard deviations calculated from 4 measurements per specimen (2 cross sections $\times$ 2 sides) for each lubricant

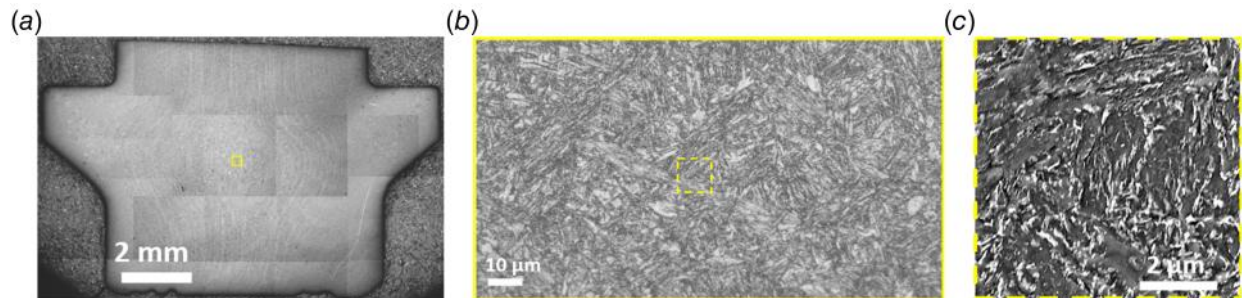


Fig. 16 (a, b) Optical and (c) SEM cross-section micrographs of the screw head before tightening

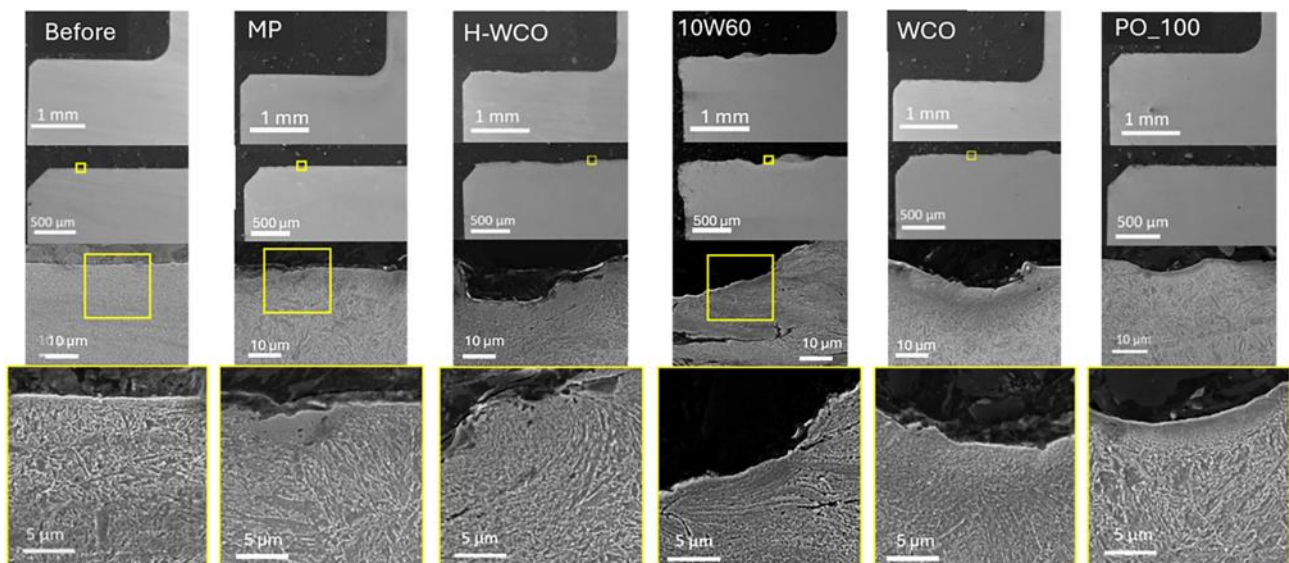


Fig. 17 SEM Micrographs of the screw cross sections after tightening, showing sub-surface plastic deformation in the mating region

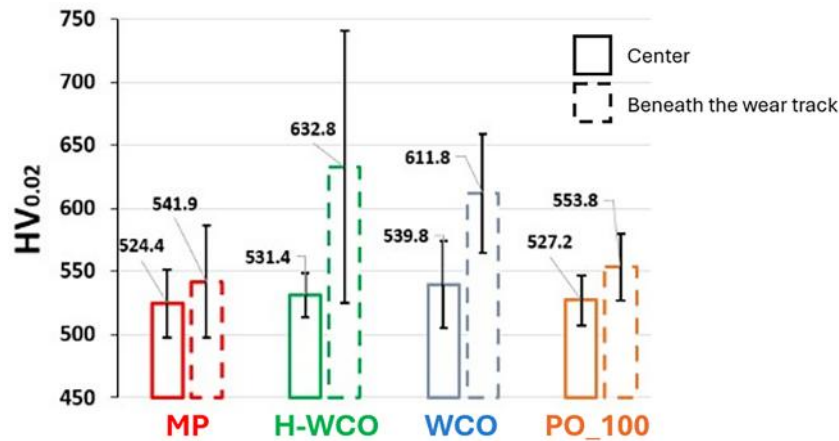


Fig. 18 HV_{0.02} approximately 10 μ m beneath the contact surfaces of screw UHs (best- and worst-performing samples from each lubricant category, i.e., oil and grease)

Figure 17 presents SEM micrographs of the screw cross sections after tightening with each lubricant.

In agreement with previous wear assessment data, WCO and PO_100 exhibit a more uniform and homogeneous cross-sectional microstructure (as for the MP benchmark grease) than H-WCO and 10 W60. This finding further supports the presence of a more uniform lubricant distribution, ensuring effective coverage of all areas exposed to elevated contact pressures throughout the tightening process. Conversely, H-WCO and 10 W60 showed signs of more severe plastic deformation, with deep grooves and built-up edges. At higher magnification, the screws lubricated with H-WCO and 10 W60 revealed significant plastic flow, along with a featureless, heavily deformed surface layer, analogous to white etching layers typically generated in steels due to severe contact conditions [53]. HV_{0.02} microhardness measurements were performed at approximately the same distance ($\approx 10 \mu\text{m}$) beneath the wear track on the best- and worst-performing samples from each lubricant category (i.e., oil and grease), to assess the localized micro-mechanical properties underneath the contact surface, taking into account the presence of the previously discussed micro-structural modifications (Fig. 18).

The highest HV_{0.02} increases were observed for H-WCO and 10 W60, indicating a more intensive plastic deformation than for MP and PO_100, in agreement with previous results.

4 Conclusions

This study provided an in-depth comparison between conventional mineral-based lubricants and innovative solutions derived from WCO for repeated tightening of high-strength threaded joints. Overall, the WCO-derived formulations outperformed the commercial mineral engine oil (10 W60), which showed the least robust behavior upon repeated tightenings, and they achieved friction levels and torque responses close to the benchmark mineral paste (MP grease).

A key outcome is that the best-performing WCO-derived lubricant reached this performance without solid lubricating additives such as MoS₂, indicating that its effectiveness stems primarily from its intrinsic molecular structure. In particular, the functionalized WCO route leading to PO_100 proved most promising: its polar functionality promotes strong affinity to oxidized steel surfaces and supports the formation and retention of a stable lubricating film during successive tightenings. This behavior is consistent with the observed improvement in repeatability and stability versus both minimally processed WCO and the hydrogenated WCO derivative.

Importantly, the tightening results were complemented by in-depth wear analyses, which helped link friction evolution to the actual damage mechanisms at the thread and bearing interfaces. These

observations confirmed that lubricant chemistry and film persistence strongly influence not only torque and friction stability, but also the extent and nature of surface degradation during repeated assembly.

Overall, the results point to two WCO-based routes with practical potential for this application: dehydrated/filtered WCO, which represents the simplest route and provided acceptable friction and wear behavior, and the functionalized route leading to PO_100 through epoxidation and ring opening. Among them, PO_100 showed the most balanced performance, combining stable friction and torque response over repeated tightening cycles with the best wear protection among the WCO-derived lubricants. These solutions support waste valorization and may reduce reliance on fossil-derived lubricant bases. Based on the literature on renewable and waste-derived lubricants, this could potentially translate into life-cycle environmental benefits. However, since no life-cycle assessment was performed for the specific WCO, H-WCO, and PO_100 production routes, and since H-WCO and PO_100 require chemical processing, no quantitative conclusion can be drawn on greenhouse-gas emissions or overall life-cycle impacts. These potential benefits should therefore be verified in future work through a dedicated LCA.

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Conflict of Interest

There are no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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