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Changes in friction of zinc flake coated threaded fasteners due to humidity, temperature and storage duration

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ABSTRACT

The friction coefficient of a specific material combination is often assumed to be invariant in manufacturing processes such as during the tightening of threaded fastener joints. This paper considers the impact of storage conditions on threaded fastener friction. Fasteners were stored in hot humid and sub-zero temperature conditions to study friction in the thread and under-head contacts. Four Zn-flake coatings, commonly used in the automotive industry were considered. Consequent tightening of these fasteners at room temperature revealed that storage history had a significant impact on their friction coefficients, halving under-head friction in some cases. This varied behavior was considered to be a response to coating nano-hardness and structure and differences in adsorption/absorption of water and zinc-oxide formation during storage.

1. Introduction

Threaded fastener coatings protect against corrosion to maintain functionality of the joint and fastener. They also aim to provide stable and predictable friction behavior during tightening, essential to achieve the desired clamp force to safely keep the joint together when external loads are applied. There are many types of commercial coatings available including Zn-phosphate, electroplated zinc, Zn-Ni, and Zn-flake. Coating selection depends on the fastener application, level of corrosion protection required, target friction coefficient, cost requirements, etc. Zn-flake coatings are the most widely used coatings in automotive industry [1]. Zn-flake coatings can be water or solvent-based and applied by dipping or spraying. As the name suggests, the coatings are "flaky", which may make them susceptible to physical and chemical changes in adverse environmental conditions especially if stored for a long time.

Local environmental conditions, particularly temperature and humidity, can vary greatly depending on where a manufacturing plant is geographically located. Additionally, most components are transported and stored under a range of conditions prior to assembly. This may be inside vehicles or warehouses with no environmental control, in assembly plants or outdoors, especially when "just-in-time" practices are employed. In all cases, local humidity and temperature can affect the surface condition of the fasteners. Ambient humidity and temperature are well known to influence the friction of many materials, including metals, consequently if the same assembly tools and tightening strategies are used in different manufacturing plants at different locations, it follows that joint quality will be affected by the recent storage history. Since torque-controlled tightening is commonly used, higher friction will give loosened joint, while lower friction may lead to fastener breakage.

Humidity is an important environmental parameter causing a different degree of water adsorption on metal surfaces depending on temperature. Surprisingly, humidity values are rarely mentioned in papers reporting threaded fastener friction behavior even though it is well established that at low humidity, the intermetallic contact increases due to reduction in oxidation in the contact area [2]. Experiments, conducted with a steel-on-steel contact in a pin-on-disk machine, have revealed a two-fold reduction in friction at 80% humidity compared to 28% humidity [3]. A similar study [4] conducted in a reciprocating pin-on-disk machine with a steel sphere sliding on a disk, coated with Zn-14%Ni coating, at relative humidity levels of 0%, 20%, 40%, and 60%, showed higher humidity levels contributed to formation of hexagonal zinc oxide in the contact area resulting in stable friction and

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lower wear rate. The lack of lubricious zinc oxide at lower humidity levels contributed to higher wear in the contact area.

The presence or absence of differing types of lubricants on parts of threaded fasteners has also been studied in detail. A study of grease, mineral oils and solid lubricant influence on friction in the thread and under-head contacts showed that the lowest thread and under-head friction was obtained with the solid lubricant [5]. Friction with grease and mineral oils was at a similar level, but at a low tightening speed medium and high viscosity oils showed lower friction than grease. Lubrication of either the thread or under-head contacts using a ceramic paste gave the lowest friction when the under-head contact was lubricated with the thread contact left "dry" [6].

Studies of friction in unlubricated coated threaded fasteners have been fairly limited. However, in one study, when an electroplated zinc coated fastener was used without lubrication, the under-head friction was found to be 44% higher than the thread friction [7], indicating the frictional dominance of the under-head contact during the tightening process. A study of friction under repeated tightening of threaded fasteners with an electroplated zinc coating showed that the clamp force after the sixth tightening was almost half of the force after the first tightening [8]. Adhesive wear in the thread contacts was shown to have increased friction considerably. It was recommended to avoid reusing electroplated zinc coated fasteners without lubrication.

Other influences on threaded fastener friction have been found to include tightening speed and washer surface condition/type. Nassar et al [9] showed that increasing tightening speed from 10 rpm to 150 rpm reduced under-head friction by 20% while thread friction increased by 5%. In comparing the effect of washer material, the most significant drop, 26%, in under-head friction was seen for Zn-based washers. A study of coating thickness on friction in the coarse and fine threads showed a 18% reduction in thread friction for a Zn-based coating when the coating thickness of a fine threaded fastener was increased. For the coarse thread an increase in the coating thickness gave a reduction of 7% in thread friction while only a 2–3% reduction in friction was measured in the under-head contact [10]. Recent studies into threaded fastener friction have also considered effects such as the impact of joint asperity interaction [11], plasticity and thread damage [12] and joint materials [13].

To our knowledge, there is no published data on how humidity may influence friction behavior of the threaded fasteners. This paper addresses this issue by providing data on the effect of storage history and humidity on friction for Zn-flake coated fasteners.

2. Experimental details

Four Zn-flake coatings, commonly used in the automotive industry, were selected for experimental investigation. One set of fasteners was exposed to hot and humid environment for up to 30 days. A second set was stored at − 20°C for 1 day. The fasteners were then tightened at room temperature. Observed friction responses were correlated with storage conditions, nano-hardness data and focused ion beam images of the coating cross sections.

2.1. Materials

We used M10 standard thread hexagonal head fasteners [14–16]. The information related to the threaded fastener joint is summarized in Table 1. The fasteners were coated with different water-based and solvent-based Zn-flake coatings by third party suppliers and the exact composition of these coatings is commercially confidential [17]. However, all of these coatings are widely used in industry particularly for automotive applications, so this investigation derives results which are representative of component behavior in businesses worldwide.

The Zn-flake 1 and Zn-flake 2 coatings were solvent-based with different types of water-based top-coat. The top-coat acts as both a sealant and a friction modifier. Zn-flake 3 and Zn-flake 4 coatings were water-based coatings without any top-coat. Nano-hardness testing of the coatings was performed using a NanoTest Vantage from Micro Materials. The instrument was calibrated for load, displacement, frame compliance and indenter shape according to ISO 14577–4 [18]. A Berkovich diamond indenter was used for conducting the hardness measurements. A load of 50 mN was applied to the sample in a 20 multi-load cycles. There was a hold of 60 s at the 90% unloading point from the last cycle to collect compensation data and correct for any thermal drift in the measurement. The tests were repeated ten times on each sample at a temperature of 25°C and 50%(RH). Hardness and reduced elastic modulus were determined by power-law fitting of the unloading curves using an Oliver and Pharr approach [19]. The elastic modulus and Poisson ratio of the diamond indenter was assumed to be 1141 GPa and 0.07.

Cross-sectioning of the Zn-flake coatings was performed using a Focused Ion Beam instrument FEI Nova 600 FIB-SEM with a dual-beam system capable of taking ultra-high resolution SEM images. A thin gold layer was first deposited on the coating to provide electric conductivity. The coating thickness varied from 8 to 16 μm. The fasteners with Zn-flake 1 (KL120) and Zn-flake 2 (KL100) coatings were provided by Bulten Sweden AB. Bufab Sweden AB provided fasteners coated with Zn-flake 3 (GEOMET 500B) and Zn-flake 4 (DACROLIT). The fasteners had different lengths due to availability constraints (Fig. 1b).

Square plates (40 × 40 mm), of Domex 650 MC, were machined to a specific average surface roughness value (*Ra*) according to ISO 16047 and used to simulate joint surfaces representative for components in the automotive industry, see Fig. 1b. New plates were used for each tightening test, but not replaced during retightening of a given fastener.

The surface roughness measurements were performed using a Surftest SJ-400 (Mitutoyo). The plate surface topography was examined

Table 1
Threaded fastener joint information.

Fastener coating	Fastener geometry				Clamp length [mm]
	Size	Head type	Length [mm]	Standard	
Zn-flake 1	M10	Hexagonal	90	ISO 4017	56
Zn-flake 2					
Zn-flake 3			60	DIN 931	46
Zn-flake 4			80		56
		Nut geometry			
Type			Hexagonal head		
Strength class			8		
Standard			ISO 4032		
Nut coating			EPZ		
		Plate			
Roughness [μm]			*Ra* 1.35 (± 0.096/ ± SD)		
			Rz 7.48 (± 1.088/ ± SD)		
Hardness [HV1]			205-264		

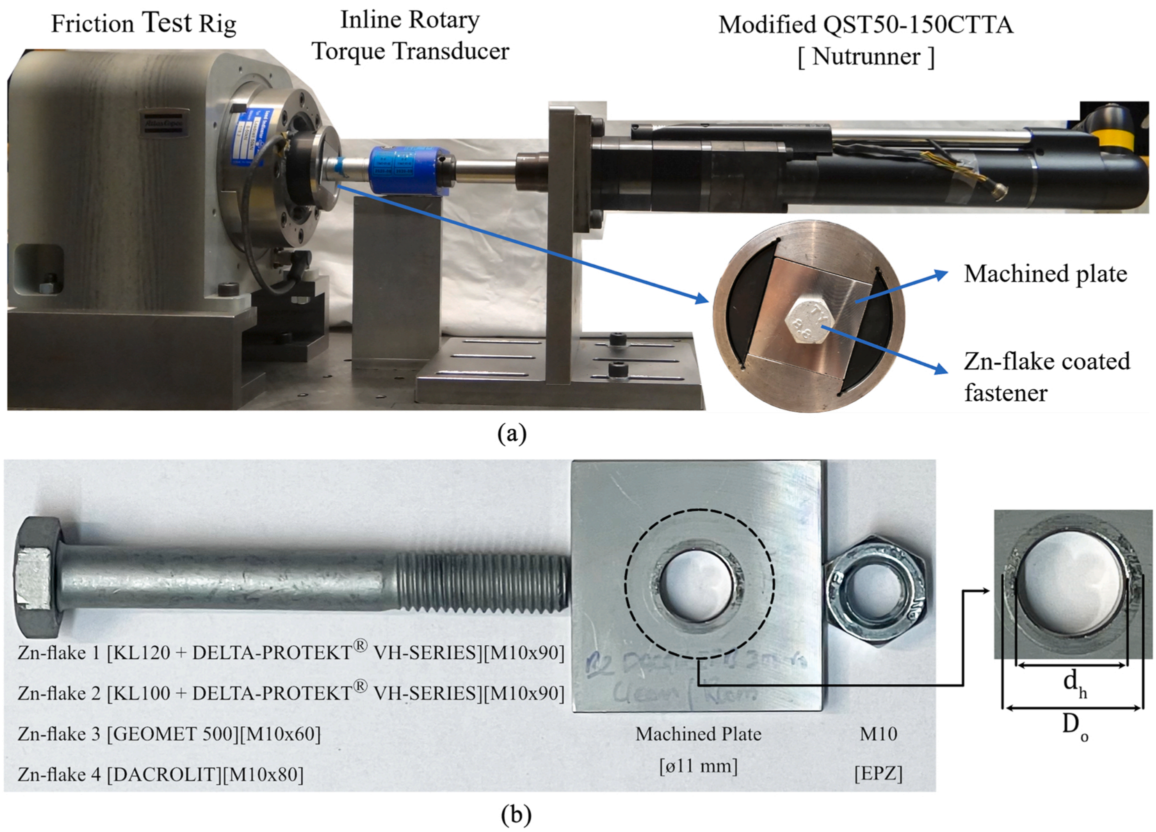


Fig. 1. Experimental set-up (a); Zn-flake coated fastener, nut and machined plate with a close-up of the wear track, diameter D_0 (b).

before and after the tests by using a white light interferometer, Zygo New View^{TM} 7300 with a vertical resolution below 1 nm. The topography was measured on the same plate outside the wear track, representing the surface prior to tightening, Fig. 2a, and inside the wear track, representing the surface after the fourth retightening, Fig. 2b. Inspection reveals that there was no significant change in the surface topography caused by the tightening process. Both the Bearing Area curve and the Surface Height Distribution were very similar and the height range was the same. The main visible effect is a slight roughing of the asperity tips on the counter-face, reflected in a slightly steeper Bearing Area curve in the corresponding range and which may be due, in part, to material transfer of the Zn-flake coating. Material transfer was also evident in Fig. 1b, especially where the plate lay is perpendicular to the motion of the rotation of the under-head surface of the fastener, which enhanced abrasion. This outcome is not surprising as all Zn-flake coatings were substantially softer than the plate.

The hardness of the plates was measured using an Emcotest M4U-025. The clearance hole in the plates was 11 mm in diameter, corresponding to a medium clearance hole for the M10 threaded fasteners according to ISO 273 [20]. The nuts deployed with the fasteners were coated with electroplated-Zn (EPZ) [21–23].

2.2. Experimental set-up

The experimental apparatus used in the study is shown in Fig. 1a. The setup included a friction test rig (FTR), an inline rotary torque transducer (IRTT, Crane Electronics Ltd.), an assembly tool (a "nutrunner"), a controller, and a data acquisition system. During tightening the clamp force was measured by a load transducer and the thread torque was measured by a torque transducer, both were installed in the FTR. The FTR was developed at Atlas Copco Industrial Technique AB [24,25].

The IRTT measured the tightening torque and tightening angle. The assembly tool was a modified QST 50–150CTTA nutrunner, controlled by the Power MACS 4000 controller. ToolsTALK 10.9.10 software from Atlas Copco Industrial Technique AB was used to control tightening. Signals from the FTR and IRTT were recorded using a Dewe-43A data acquisition system, manufactured by DEWESoft®. All signals were filtered with a third order Butterworth filter with a cut-off frequency of 500 Hz. Further post-processing of the data was done using MATLAB®.

2.3. Test procedure and analysis

To simulate the impact of storage conditions in the different parts of the world, hot humid weather and sub-zero temperatures, fasteners were placed in a Vötsch VC^3 4034 climate chamber. The threaded fasteners were kept, from a minimum of 2 days to a maximum of 30 days, at 40°C, at more than 95%(RH) to simulate storage in hot humid climate. Another set of fasteners was kept at − 20°C for 1 day to simulate the impact of sub-zero temperatures. In all the cases, tightening was performed within 30 mins of the fasteners being taken out of the climate chamber.

The square plates received from the supplier (Martin Fredin Precision AB) had residues of metal working fluids on the surfaces. Such residues could influence under-head friction and thus clamp force, as demonstrated by Kumar et al. [26]. To remove residues of metal working fluids, the plates were washed with technical grade, min 98.5% purity heptane in an ultrasonic bath for an hour. The plates were then air-dried and stored at 21–23°C, 40–55%(RH). The threaded fasteners and nuts were visually inspected to remove components that had burrs and mechanical deformity in the threads.

Fasteners were tightened using a 2-step tightening strategy outlined in Table 2. The selected speeds are used in the automotive industry to tighten threaded fastener joints. Each fastener was tightened to 25 kN, this being about 75% of the proof load as recommended by ISO 16047:2005 [27]. Tightening tests were repeated at least five times. In each test, a new threaded fastener, plate and nut were used. The fastener

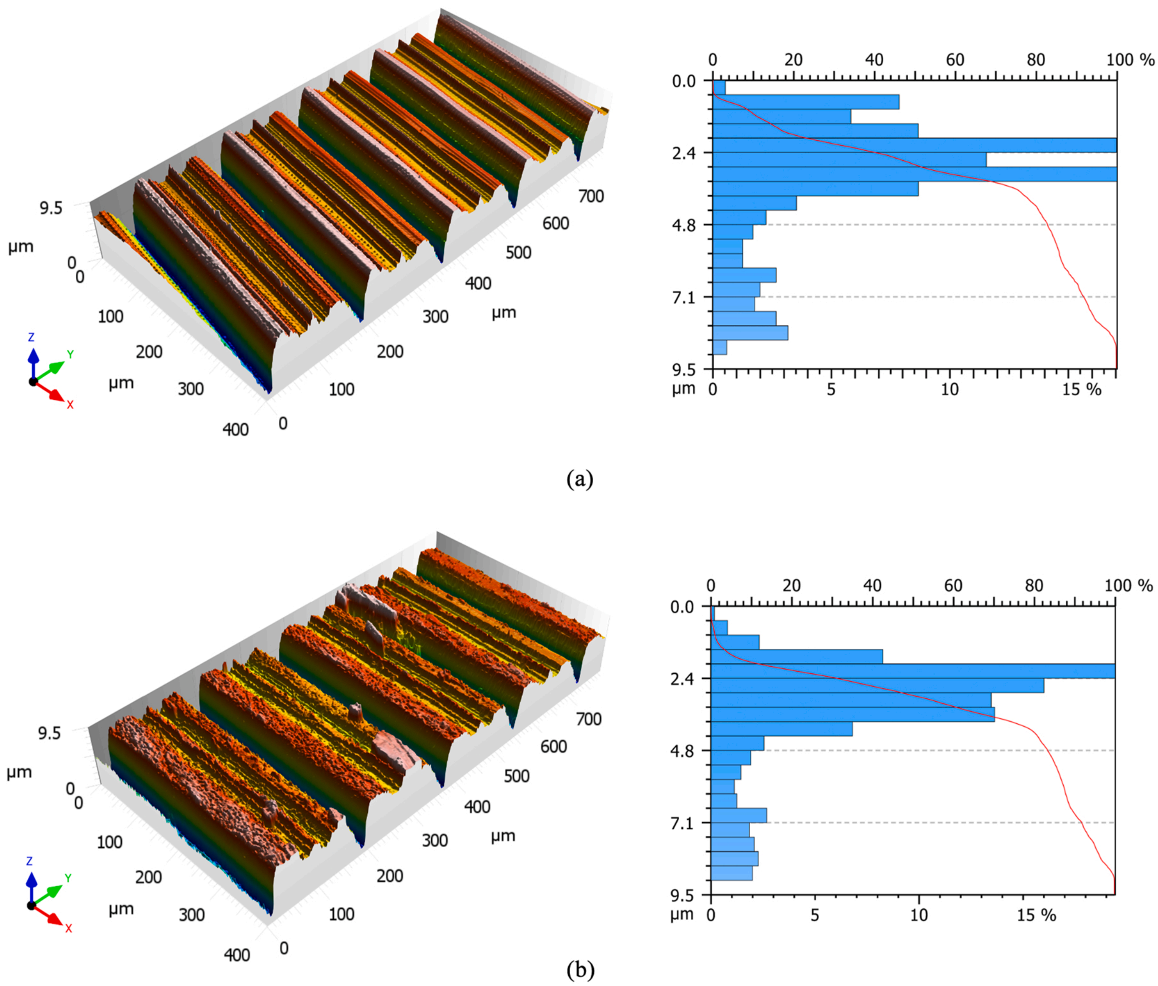


Fig. 2. 3D images, bearing curves and height distributions illustrating the surface texture of the plate before (a) and after (b) the fourth retightening done at room conditions for Zn-flake 1.

Table 2
Experimental conditions.

Fastener coating	Load controlled tightening		Environmental conditions		
	Speed in Step 1	Speed in Step 2	Room condition	Hot / Humid	Cold
Zn-flake 1, 2, 3, 4	300 rpm	60 rpm	21–23°C, 40–55% (RH)	40°C, ≥ 95% (RH) [2, 7, 14, 30 days]	-20°C [1 day]

was tightened and untightened five times without disassembling the joint. There was a 5 s pause between each retightening.

Two distinct contact interfaces contribute to friction in threaded fasteners. These are in the thread and under the fastener head. It is possible to distinguish between these two contributions theoretically [27,28]. The under-head torque T_{un} [Nm]is the difference between the tightening torque T_{tot} [Nm] and thread torque T_{th} [Nm]:

$$T_{un} = T_{tot} - T_{th} \tag{1}$$

The thread friction coefficient μ_{th} is obtained by

$$\mu_{th} = \frac{\frac{T_{th}}{F} - \frac{P}{2\cdot\pi}}{0.577\cdot d_2}, \tag{2}$$

where F is the clamp force [kN], P is the pitch [mm] and d_2 is the pitch diameter [mm]. The elastic stretching of the threaded fasteners $P/(2\cdot\pi)$ is deduced from the thread torque, T_{th}, in the numerator. The under-head friction coefficient, μ_{un}, is calculated as follows

$$\mu_{un} = \frac{T_{un}}{0.5\cdot D_b\cdot F}, \tag{3}$$

where friction diameter D_b [mm], defined as

$$D_b = \frac{D_o + d_h}{2}, \tag{4}$$

Using these relationships the thread friction and under-head friction can be compared in the different storage conditions.

3. Results

All fasteners were tightened to the same clamp force of 25 kN. An example of the filtered signals from the sensors in the FTR and IRTT are shown in Fig. 3. The signals are for the Zn-flake 1 coated fasteners, but are representative of data for the other fasteners. The tightening torque, thread torque and clamp force signals are shown after the snug point[2] [29] as a function of time into tightening. It can be noticed that the tightening process continued slightly beyond the 25 kN clamp force. The overshoot was caused by the shut-off mechanism of the tightening tool.

For each coating the tightening torque and friction coefficients in the

[2] Snug point is a point at which all parts of the joint have "mated" after rundown.

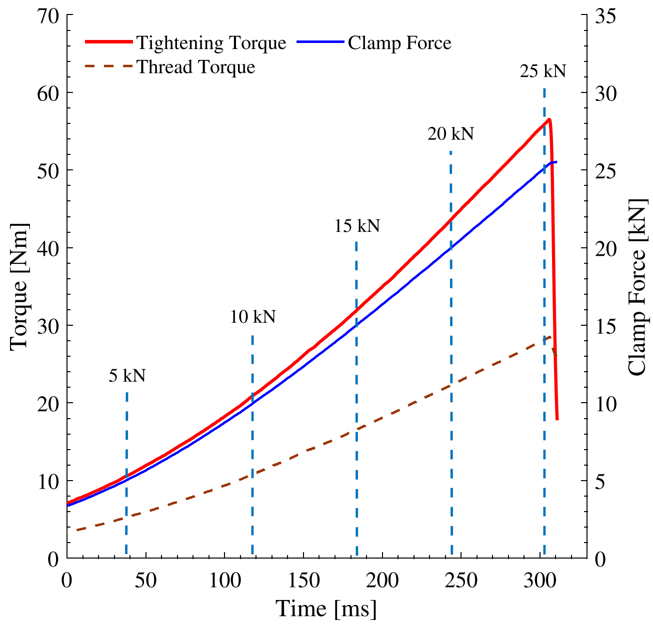


Fig. 3. Evolution of the torque and clamp force during tightening of the Zn-flake 1 coated fasteners at room temperature.

thread and under-head contacts are discussed below. The tightening torque and thread torque values at five clamp force levels (5, 10, 15, 20, 25 kN, dashed lines in Fig. 3) were extracted to calculate friction coefficients using the Kellerman-Klein equations 2 and 3.

3.1. Performance of Zn-flake 1 coating

Fig. 4 shows the tightening torque required to achieve clamp force of 25 kN with the Zn-flake 1 coated fasteners stored in different environments: room conditions, hot humid conditions and at − 20°C. Each fastener was retightened four times. After 2 days exposure to hot humid conditioning no change in friction was observed, but after 7 days exposure there was a 10% drop in T-1 tightening torque, compared to the fasteners stored at room conditions. After 14 and 30 days of exposure, further reduction in the T-1 tightening torque was minor. The tightening torque increased during the first retightening (T-2), but then

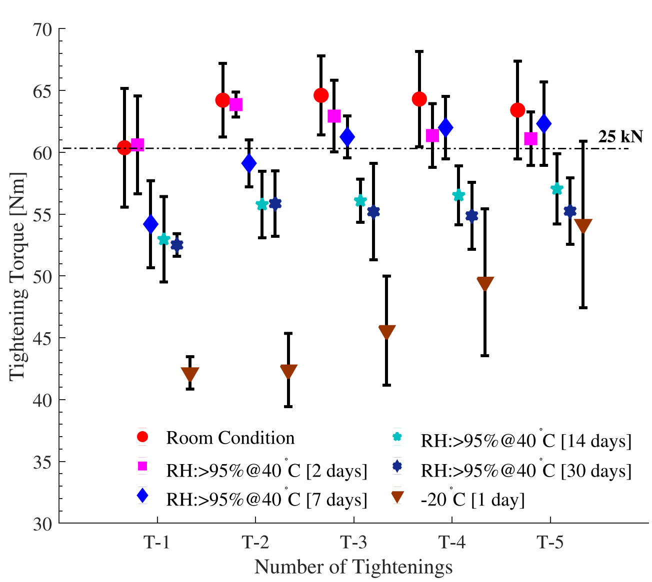


Fig. 4. Tightening torque required to achieve 25 kN clamp force for the Zn-flake 1 coated fasteners.

showed a tendency to decrease or plateau with each consecutive retightening. The scatter variation did not follow a clear trend, except for the fasteners with the longest exposure time. They showed the lowest scatter during the T-1 tightening and the lowest increase in torque towards the last retightening.

Fasteners stored at − 20°C showed a dramatic drop of 30% in T-1 tightening compared to the fasteners stored in room conditions. When the fasteners were removed from the environmental chamber and exposed to room conditions, 20–21°C, 40–45%(RH) (water content of 5.85–7 g in one kilogram of air), water condensed on the cold fastener surfaces, perhaps even forming icy patches, probably contributing to relatively low tightening torque and scatter values during the T-1 tightening. The torque level remained the same for the first retightening (T-2), but scatter increased. Consecutive retightening increased both the torque and scatter values.

Fig. 5 shows how storage conditions influenced friction coefficients at the different interfaces in the case of Zn-flake 1 coated fasteners. The results are shown for the fasteners stored at room conditions (R-M), in hot humid conditions for 7 days and at − 20°C for 1 day.

For thread friction, there was a small decrease in friction for hot/humid conditions compared to R-M after the T-1 tightening. The gap remains the same even after the fourth retightening (T-5). The overall trend for the thread friction shows a small increase in friction for R-M and hot/humid conditioned bolts. When the fasteners were subjected to cold climate and tightened for the first time, there was a reduction of 16% in thread friction. The gap was further widened to 31% after the fourth retightening (T-5) for cold climatic comparing to R-M.

Under repeated tightening, under-head friction was found to increase for the T-1 tightening in R-M and hot/humid conditions. This friction coefficient also increased as higher clamp forces were applied. However, an opposite trend was seen for fasteners in cold conditions. A decrease of 47% in under-head friction was observed in the T-1 tightening compared to R-M conditions. For all the conditions under-head friction was found to increase with every consecutive tightening, unlike thread friction. After the fourth retightening (T-5), the under-head friction had converged to the same level as that for R-M conditions. This behavior contrasts with friction behavior observed for the thread friction which increased with retightening. Fig. 6b shows a cross sectional cut of the Zn-flake 1 coating on the under-head surface of the fastener obtained using focused ion beam sectioning. The darker substrate is located at the bottom. The basecoat (central) zone of the coating is paler about 5 μm thick and appears to be relatively homogeneous. The thinner top-coat is darker in color and is approximately 1 μm thick. The hardness of the coating structure was measured using a nano-indenter and the results are shown in Fig. 6c. The top-coat hardness appears to be low compared to the base coat. Within the base coat, the hardness did not change significantly with depth. Fig. 6a shows the cross sectional cut of the electroplated zinc coating on the nut. The cross section indicates that the coating is homogeneous. There was only a minor reduction in hardness and increased scatter with depth, as shown in Fig. 6c.

3.2. Performance of Zn-flake 2 coating

All coatings were investigated in a similar fashion to Zn-flake 1. Fig. 7 shows data collected for Zn-flake 2. It can be seen there is a T-1 tightening torque decrease of 13% after the two-day conditioning in hot/humid conditions compared to storage in room conditions. This drop in torque could be partly due to the method used to apply the coating to the surface of the fastener during the manufacturing process. The coating section, shown in Fig. 9a, reveals significant porosity between different layers, which could serve to trap moisture between the layers resulting in lower torque in a shorter storage time than Zn-flake 1. A further reduction in T-1 tightening torque did not happen with storage time for Zn-flake 2. On the contrary, a slight upward trend is visible in the T-1 tightening torque with more storage time in hot/humid climatic conditions (Fig. 7). Scatter in torque varies between each retightening. A

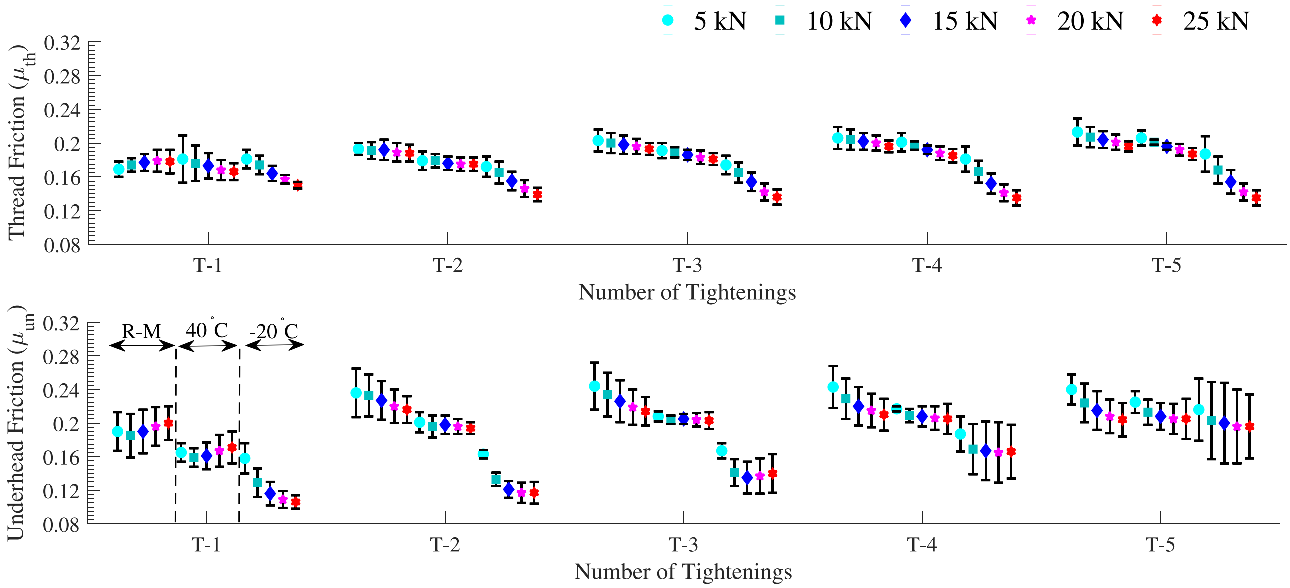


Fig. 5. Coefficient of friction in the thread and under-head contacts for the Zn-flake 1 coated fasteners at 5, 10, 15, 20 and 25 kN clamp force. Storage conditions: room temperature **(R-M)**, 7 days at 40°C and 1 day at − 20°C.

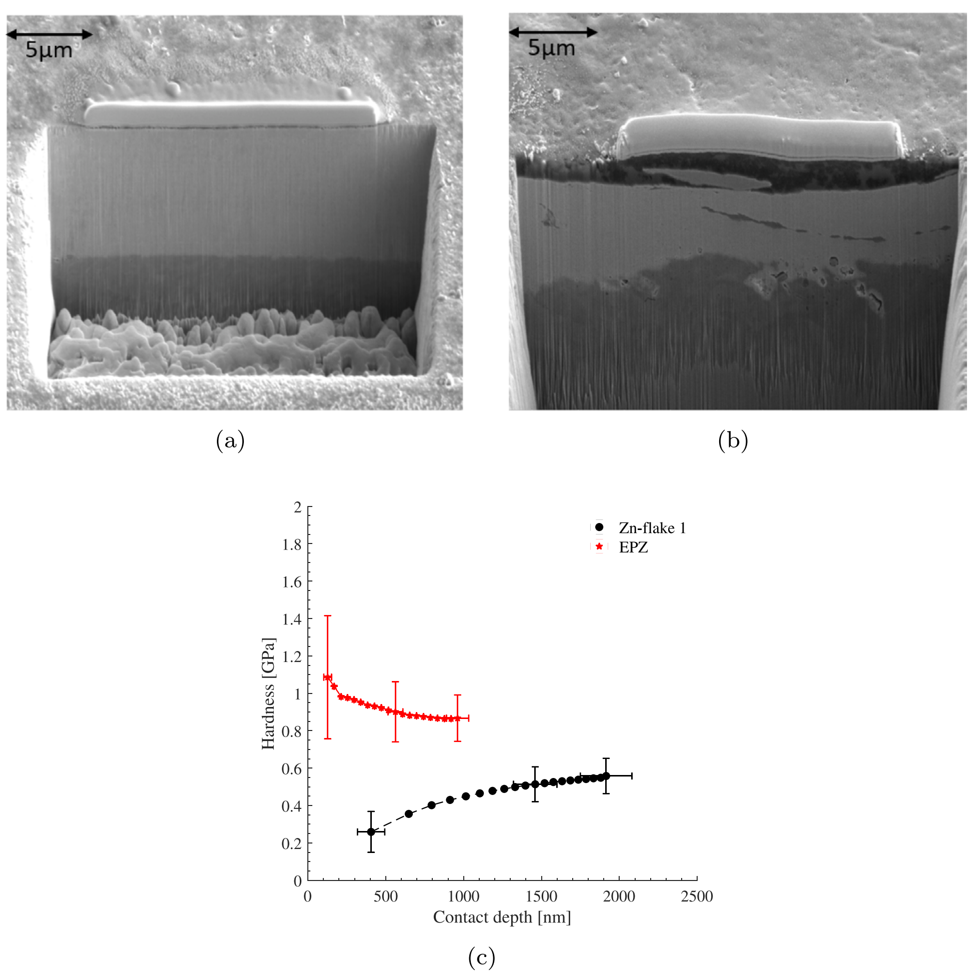


Fig. 6. Cross-sectional view of the EPZ coating on the nut surface (a) Zn-flake 1 coating on the under-head surface of the fastener (b) and variation in coating nano-hardness (c), shown with ± SD, 50 *mN* load for Zn-flake 1 and 20 *mN* for EPZ.

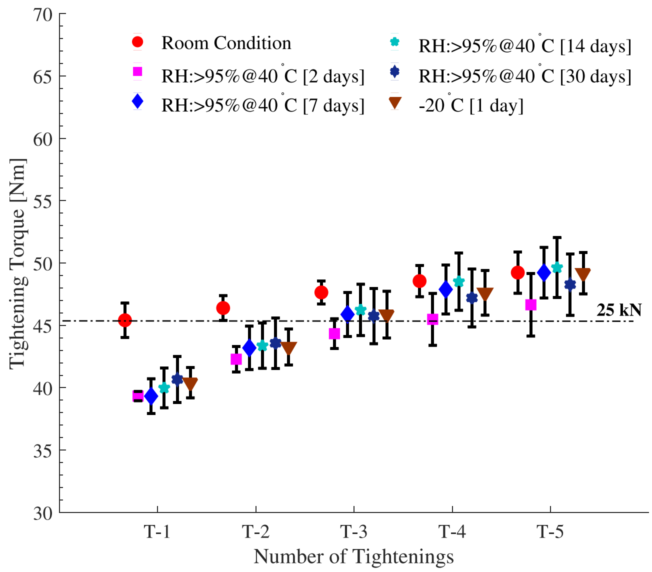


Fig. 7. Tightening torque (tightening 1–5) for the fastener with Zn-flake 2 subjected to room conditions, humidity conditioning at 40°C in the climate chamber for different time intervals (2, 7, 14 and 30 days) and one day conditioning at − 20°C in the climate chamber.

T-1 tightening torque reduction of 11% was observed for cold climatic compared to the room conditions, which, interestingly, was lower than being kept in hot/humid conditions for 2 days. The mean T-1 tightening torque showed an upward trend when fasteners were conditioned in hot/humid for 7 days and further.

The thread and under-head friction values were evaluated for the fastener subjected to room conditions (R-M) and were compared with fastener conditioned at hot/humid climatic (40°C) for 2 days and cold climatic condition (−20°C) for 1 day. Results are shown in Fig. 8. In contrast to Zn-flake 1 thread friction was more affected for hot/humid conditions than it was by cold conditions. For the T-1 tightening, a reduction of 20% in the thread friction was observed compared to room conditions whereas a decrease of only 3% was recorded for cold conditions compared to R-M. An increase in friction was seen for the cold condition with consecutive tightenings, but some other values remain stable. After the T-5 tightening, the thread friction was 17% higher in cold conditions compared to R-M. For the T-1 tightening in under-head friction, a reduction of 12% for hot/humid and 19% for cold climatic conditions compared to R-M was reported. The under-head friction increases with every retightening. The friction was similar for all the conditions after being retightened four times. Irrespective of the tightening condition, thread and under-head friction increased with increasing clamp force.

Fig. 9 a shows the layered deposition of the coating on the fastener and a significantly higher flake coating thickness which appears to involve two forms (shades) of material. The Zn-flake 2 coating seems to be harder near the surface, but as the nano-indenter penetrates the coating, hardness reduces as shown in Fig. 9b. The scatter in the depth increases as the indenter penetrates the coating. In contrast, the scatter in hardness decreases with increase in depth. The reason for this could be that the layered deposition of the coating on the fastener surface creates air gaps between the layers that collapse during indentation. The fasteners with the Zn-flake 2 coating were conditioned in the climate chamber 40°C@ 50%(RH) for 2 days condition and tightened in room conditions to rule out any effect of temperature. The results show that tightening torque was not affected by temperature following the same trend as other tests when tightened a similar number of times.

3.3. Performance of Zn-flake 3 coating

For fasteners coated with Zn-flake 3 and subjected to 2 days of conditioning in hot/humid conditions a reduction in T-1 tightening torque of 13% was observed as shown in Fig. 10. The T-1 tightening torque reduced further after storage for seven days, but an increase in torque was observed after 14 days of storage, followed by a reduction after 30 days (The slight increase in T-1 tightening torque after 14 days is probably an outlier). For cold conditions, the reduction in T-1 torque was 27% compared to room conditions. The scatter in torque generally remained consistent or increased between retightening.

The thread and under-head friction values were evaluated at different clamp forces during the tightening following different storage conditions, as shown in Fig. 11. For the T-1 tightening, the fastener conditioned in cold climatic showed the lowest thread friction compared to the rest. Friction increased on subsequent retightening after storage in R-M and hot/humid conditions. This increment was roughly 15–17%.

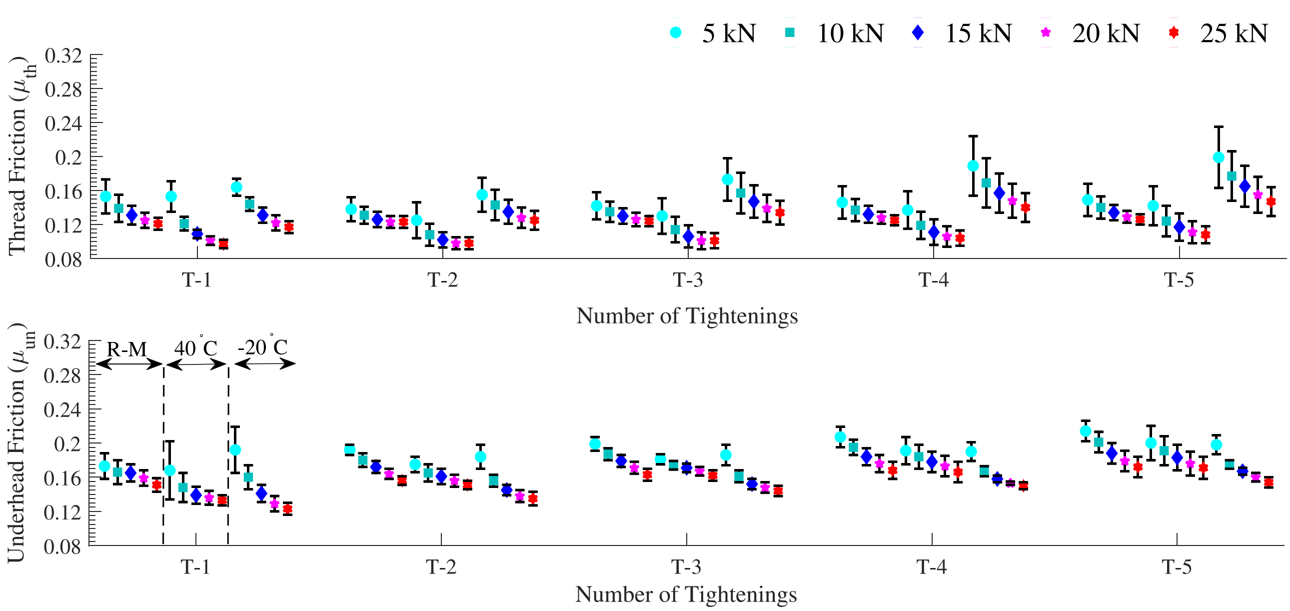
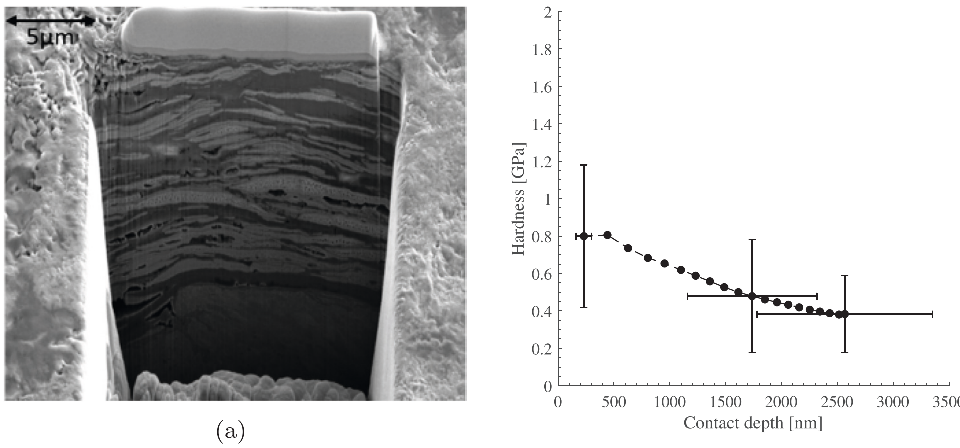


Fig. 8. Coefficient of friction in the thread and under-head contacts for the Zn-flake 2 coated fasteners at 5, 10, 15, 20 and 25 kN clamp force during tightenings of the joint under three different environmental conditions: at room temperature **(R-M)**, 2 days at 40°C and 1 day at − 20°C.



(a) (b)

Fig. 9. The cross-sectional cut of the Zn-flake 2 coating on the under-head surface of the fastener through FIB (a) and nano-hardness data, shown with ± SD, of the Zn-flake 2 coating at a load of 50*mN* (b).

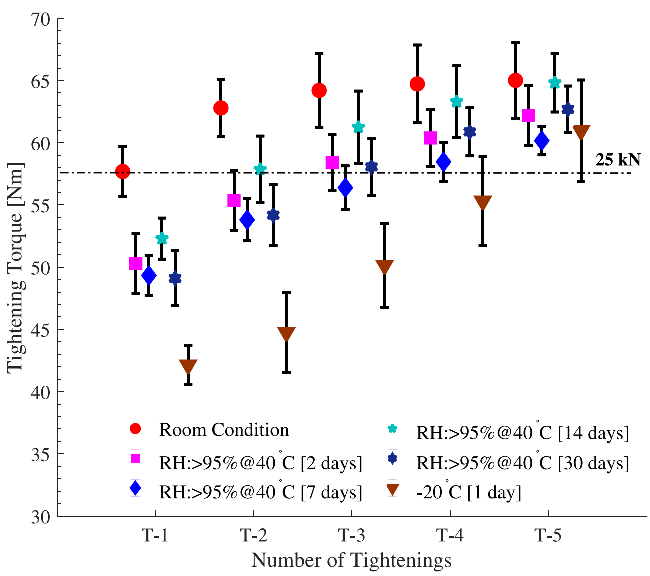


Fig. 10. Tightening torque (tightening 1–5) for the Zn-flake 3 coated fasteners subjected to room conditions, humidity conditioning at 40°C in the climate chamber for different time intervals (2, 7, 14 and 30 days) and one day conditioning at − 20°C in the climate chamber.

However, no increase in friction was seen for consecutive retightening following cold storage.

A higher impact of different climatic conditions on friction was evident for the under-head contact compared to thread contact. The fastener conditioned in cold climate showed the least friction compared to R-M and hot/humid conditions. A rapid increase in under-head friction was observed with repeated tightening for all environmental conditions. From T-1 to T-5, the under-head friction increased by 13% for R-M, 37% for hot/humid, and 101% for cold climatic conditions. Tightening at R-M showed a slight increase in friction with an increase in clamp force for the T-1 tightening for under-head contact surfaces. From T-2 and further, a decreasing trend in friction with an increase in clamp force was reported for all environmental conditions.

Fig. 12 a shows a cross-section images of the Zn-flake 3 coating. The coating was deposited in layers on the fastener surface. The coating was very porous in nature and air gaps can be seen between each layer. This probably explains the larger scatter in the nano-indentation measurements, presented in Fig. 12b. The average hardness curve shows that the hardness does not change with an increase in depth.

3.4. *Performance of Zn-flake 4 coating*

Zn-flake 4 coatings subjected to 2 days conditioning in hot/humid conditions showed a reduction of 10% in T-1 tightening torque compared to those stored in room conditions, as shown in Fig. 13. Increasing the conditioning time from 2 days to 30 days in hot/humid conditions did not lead to a significant change in the T-1 tightening torque (4%). Additionally, there is a probable outlier in the measurement where the T-1 tightening torque increases after day 7 of conditioning, but again decreases after day 14. This variation of tightening torque disappears from the T-2 tightening irrespective of the number of days stored. This could be due to variations in the fastener production even though they were from the same batch. For cold storage conditions, a reduction of 18% in T-1 was seen compared to room conditions. The tightening torque always increased with retightening irrespective of the climatic conditions. An exceptionally high torque scatter was observed after the fourth retightening (T-5) following cold storage.

Data comparing friction in the thread and under-head for Zn-flake 4 following storage in room conditions (R-M), hot/humid conditions (40°C) and cold conditions (−20°C) are presented in Fig. 14. For the T-1 tightening, the thread friction for R-M and hot/humid conditions were practically the same, but a reduction of 15% was seen following cold storage. The thread friction increased with consecutive retightening for both R-M and hot/humid storage. For the cold conditions, there was a 9% decrease in friction between the T-1 and T-5 tightenings.

The increment in under-head friction was similar for room and hot/humid conditions and finished at the approximately the same level after the T-5 tightening. After the T-1 tightening the under-head friction following cold storage was 25% lower than for R-M storage. For cold conditions, the increase in friction was around 96% from the T-1 to T-5 tightening, which was significantly greater than for all the other tests. The high scatter in under-head friction at the fourth retightening (T-5)

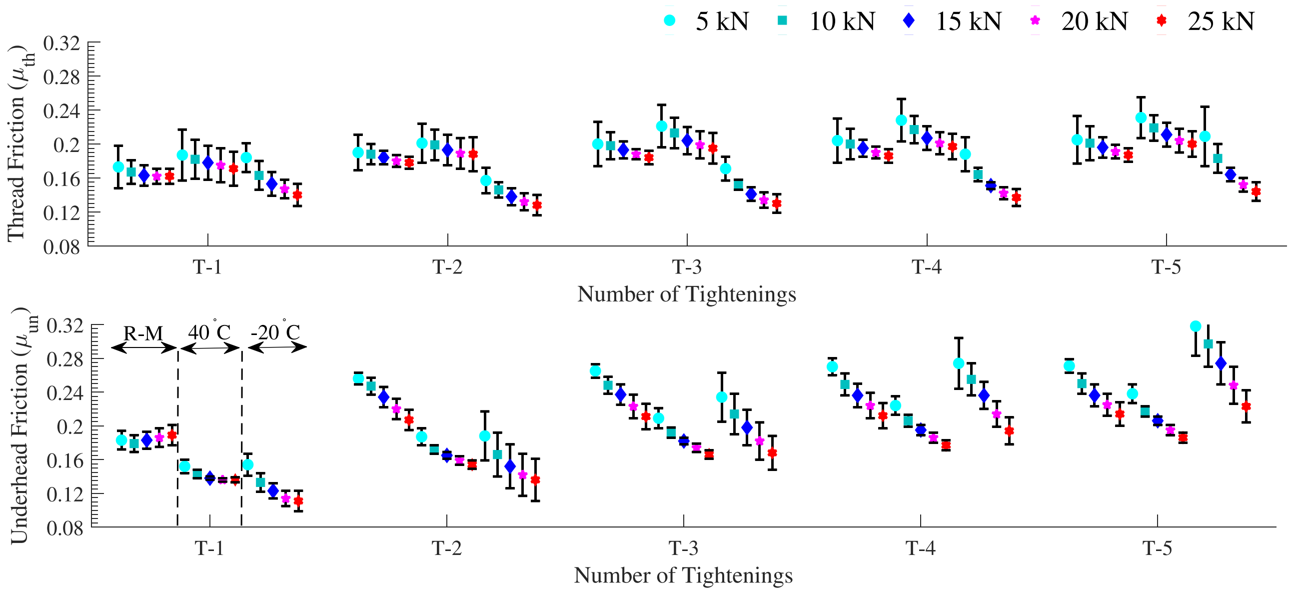
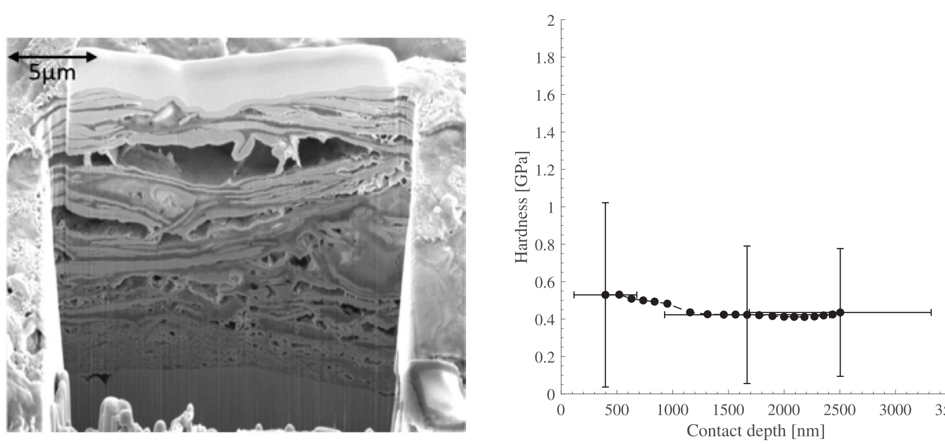


Fig. 11. Coefficient of friction in the thread and under-head contacts for the Zn-flake 3 coated fasteners at 5, 10, 15, 20 and 25 kN of clamp force for five repeated tightenings of the joint and three different environmental condition: at room temperature **(R-M)**, 2 days at 40°C and 1 day at − 20°C.



(a) (b)

Fig. 12. The cross-sectional cut of the Zn-flake 3 coating on the under-head of the fastener through FIB (a) and nano-hardness data, shown with ± SD, of the Zn-flake coating at a load of 50 *mN* (b).

for the cold storage explains why such a high scatter in the final torque was observed.

Fig. 15a presents a cross-section of the Zn-flake 4 coating which has been deposited in layers on the fastener surface in a fashion similar to that of the Zn-flake 2 and 3 coatings. The variable thickness of the coating was, in part, due to the unevenness of the surface of the fastener. Some small air gaps are visible in the cross section, but they are significantly less pronounced than those in the Zn-flake 3. The hardness of the coating increased with the depth of indentation, as shown in Fig. 15b. The scatter in hardness increased with an increase in nano-indentation depth.

A number of observations can be made by inspecting the data presented above.

Effect of storage conditions: The preliminary aim of this project was to investigate the impact of storage conditions on friction co-efficients. Table 3 summarizes the main observations.

Effect of coating type: Expecting that differing types of coating may have different frictional response to storage, both main classes of Zn coating, water based and solvent based, have been studied. A certain level of inhomogeneity and porosity was evident between the layers of all the Zn flake types and this was reflected in the hardness responses under nano-indentation testing. All the coatings showed changes in hardness with increasing depth of indentation and unsurprisingly the different coating types showed differing behavior in response to

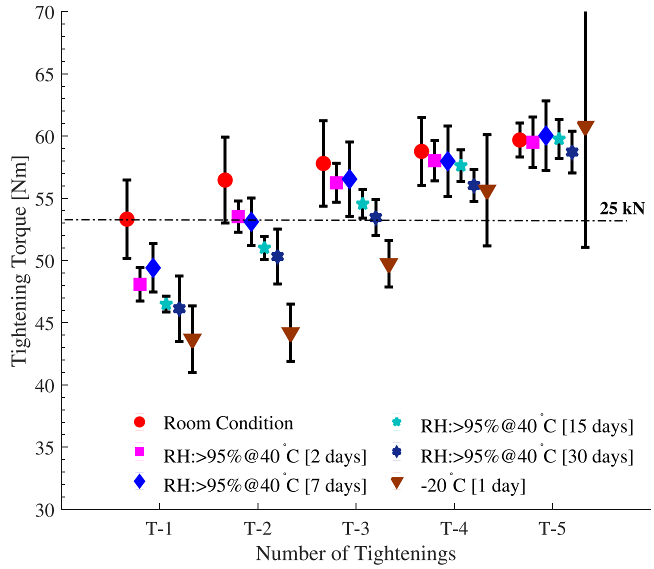


Fig. 13. Tightening torque (tightening 1–5) for the Zn-flake 4 coated fasteners subjected to room conditions, humidity conditioning at 40°C in the climate chamber for different time intervals (2, 7, 14 and 30 days) and one day conditioning at − 20°C in the climate chamber.

tightening, partly as a consequence of variation the hardness profiles. All the coatings also had a top-coat which influenced the impact of exposure to humidity. Details of friction responses to coating structure are summarized in Table 4.

Effects related to tightening: Changes in friction during tightening demonstrated a dependency on the tightening strategy. Friction in the threads and under-head reduced with increasing clamp force for almost all the combinations where friction increased during the first tightening. Details related to the effects of retightening are summarized in Table 5.

In manufacturing only the changes in friction on the first tightening are relevant. However, it is apparent in the data for all the coatings that friction coefficients for the under-head contact tend to increase under retightening, showing that the effect of storage condition is tenacious, continuing to persist, although with lowered impact, remaining lower than values for room storage up to the fifth tightening. Friction coefficients for thread friction show a mixed response, with two tending to reduce (Zn-flake 3, Zn-flake 4) and two staying mostly the similar (Zn-flake 1, Zn-flake 2).

The increases in friction coefficient probably reflect several factors including the loss of lubricating compounds, as well as damage to the surface of the interface components, which develops with retightening.

4. Discussion

This investigation has addressed the impact of a range of storage conditions involving humidity and temperature on the friction behavior of threaded fasteners coated with four commonly used water-based and solvent-based Zn-flake formulations. The purpose of the research has been to identify the likely friction behavior of these materials when they are used in “real-life” manufacturing situations which involve transport, storage and use of these components in a range of environmental conditions.

The reduction of friction is most dramatic for components stored in cold conditions. All coatings had a similar response when stored in cold conditions, possibly because their behavior is dominated by free water (in the form of condensation) following cold storage. Water is generally a poor boundary lubricant. However, the fastening process in all tests was performed at relatively high speed using the nut-runner. Consequently, free water and ice on the surfaces of cold components (see ESI for details) may have the opportunity to support limited micro-pockets of pressurized fluid contributing to a degree of mixed lubrication reducing friction for those components conditioned in cold storage. The scale of this effect will be moderated by several factors, including the surface roughness and the hydrophobic properties of the coating which will impact how effectively free water will maintain droplet form or spread out across the fastener surface. In the case of cold storage, the

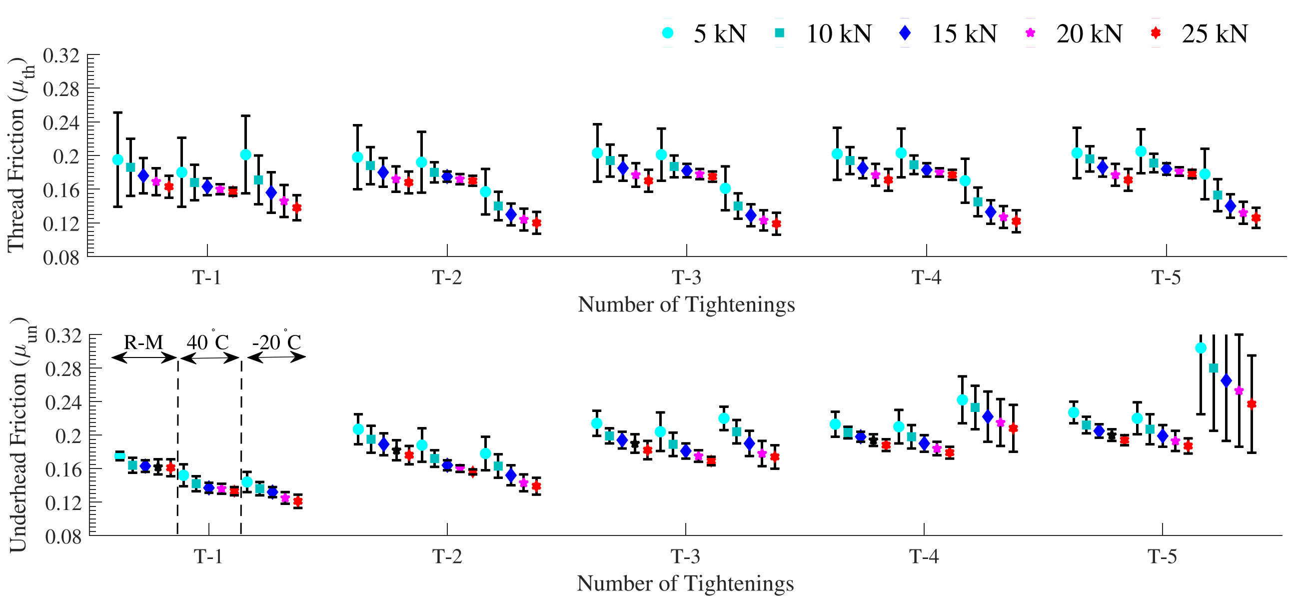


Fig. 14. Coefficient of friction in the thread and under-head contacts for the Zn-flake 4 coated fasteners at 5, 10, 15, 20 and 25 kN clamp force. Storage conditions: room temperature **(R-M)**, 2 days at 40°C and 1 day at − 20°C.

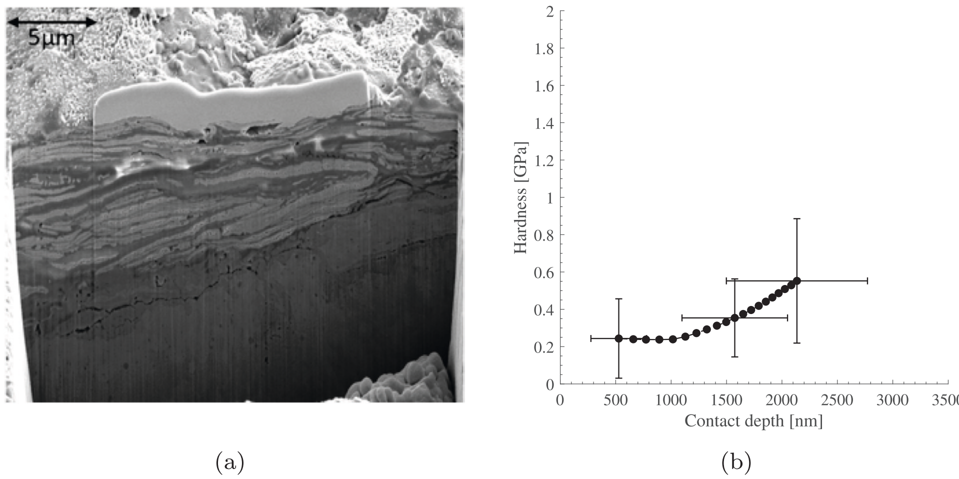


Fig. 15. The cross-sectional cut of the Zn-flake 4 coating on the under-head of the fastener through FIB (a) and nano-hardness data, shown with ± SD, of the Zn-flake 4 coating at a load of 50 *mN* (b).

Table 3
Effect of storage condition.

Cold conditions	Following cold storage, T-1 tightening torque was reduced by 30–11% compared to room storage conditions. Almost no difference in T-1 tightening torque was observed between different Zn-flake coating types.
Hot/humid conditions	Humid conditions influence changes in interfacial shear differently and appear to be linked to the type of Zn-flake coating. There appears to be a "saturation point" for exposure time in humid conditions beyond which no significant reduction in torque is observed for any formulation of Zn-flake coating.

Table 4
Effect of coating type.

Solvent-based	Zn-flake 1 (the densest coating with the least morphological variation) showed a T-1 torque reduction of 10% after 7 days of conditioning in the humid climate chamber. A saturation was observed after 7 days. After 30 days of conditioning, the torque reduction was an additional 3%. Zn-flake 2 showed 13% reduction in T-1 torque after 2 days of humid conditioning and demonstrated no further significant torque change after 30 days.
Water-based	Zn-flake 3 showed 13% and Zn-flake 4 a 10% reduction in T-1 torque after 2 days of conditioning. After 30 days of conditioning, the reduction compared to 2 days was additional 2% for Zn-flake 3 and additional 4% for Zn-flake 4.
Impact of top-coat	Even though the top-coat was water-based for both of the solvent-based coatings, it took 7 days of conditioning in hot/humid conditions for Zn-flake 1 to show the drop in tightening torque. However, for Zn- flake 2 it took 2 days. The reason for this could be linked to the porosity between the layers of the coating.

Table 5
Effects related to tightening.

Under-head friction	For Zn-flake 1, 3 & 4, the under-head friction was affected more for the T-1 tightening comparing R-M to hot/humid conditions. The under-head friction increases with a steeper gradient than thread friction with each retightening as the plate was rougher than the thread surface. Under-head friction was affected more by the retightening than thread friction for water-based coating, especially in a cold climate.
Retightening	Tightening torque increased with the number of retightenings irrespective of coating type. For both hot/humid and cold conditioning, the increase in tightening torque with the number of tightenings was much steeper for water-based coatings than for solvent-based coatings. Torque scatter after the fourth retightening (T-5) for Zn-flake 1 & Zn-flake 4 was adversely affected by condensed water in the contact surface. The mean torque for all the conditions converges to the same level for Zn-flake 2 & Zn-flake 4 after the fourth retightening (T-5).

distribution of water on the fastener will also have some dependence on the local heat capacity. Consequently, the fastener threads will develop a relatively small amount of condensation in comparison to the head, which has a large heat capacity. This may explain why the variation of under-head friction in cold conditions is much larger than the variation of thread friction.

Reductions in friction are also observed for components stored in warm humid conditions. Zinc in humidified air forms zinc oxide [30], so storage, especially in warm conditions, probably leads to the formation of zinc oxide. The mostly probable explanation for the reduced friction for these components is an increase in the amount of zinc oxide due to the presence of water. Although zinc flake has limited permeability, water can enter the upper layer of the flake coating by diffusion in both warm and cool conditions to form zinc oxide. Zinc oxide in particulate form is known to have a low friction coefficient when in contact with steel counter-faces [31] so longer exposure to water increases the proportion of lubricating zinc oxide.

4.1. Effect of coating hardness profile

The differing Zn-flake types have hardness-depth profiles which probably contribute to differences in their friction characteristics. Zn-flake 2 is the only coating in which hardness falls dramatically with depth and it can be seen from Fig. 8 that generally this coating has the most stable retightening characteristics of the four coatings tested. The reason for this is not entirely clear. However, it may be the case that the hardness profile reflects a reducing shear strength in the lower layers of the coating leading to an increased capability for internal sliding at higher contact pressures, thus tending to maintain a constant friction coefficient. Compression of the other coatings, where hardness increases with depth, potentially leads to shear strength increasing with contact pressure tending to further resist lateral shear. Evidence for this is present in data for room temperature stored fasteners with friction coefficients which uniformly reduce with load for T-1 under-head friction for Zn-flake 2, but tend to increase for Zn-flake 1,3 and 4. The effect is not evident for subsequent tightenings which suggests that debris generated in T-1 may serve to reduce friction following the first tightening.

4.2. Implications for users

It is clear that friction coefficients for zinc flake coated threaded fasteners are not stable when they are stored in differing environmental conditions or change position quickly from one environment to another before use. An important outcome of this investigation is therefore to have identified the scale of this effect. The changes in friction coefficients, of up to 47%, could have a significant impact on fastener tension and component clamp force when torque tightening is adopted. This can lead to either overtightening or under-tightening. Under-tightening potentially leads to clamp forces which are lower than specified, which may lead to loosening and loss of clamping by vibration induced loosening [32]. Over-tightening can lead to failure of a fastener when lateral loads are applied to the joint. These effects can span all applications where threaded fasteners are used, outdoors in applications such as railway installations and steel frame assembly for buildings through to factory manufacture including assembly (and maintenance) of aircraft, trucks, trains, wave energy converters, etc., potentially compromising joint security and user safety. Intriguingly, the impact of storage conditions on the tribological performance of threaded fasteners reported in the paper leads to the possibility that this effect may be the root cause of some unexpected and unexplained failures of threaded fasteners which are the subject of both anecdotal discussion and formal reports in industry.

At a time when the world is focusing increasingly on ways to address climate change, it looks likely that the “maintainability” of products will become increasingly mandatory. Such expectations require products to be dismantled. Often this is achieved using threaded fasteners, so it is reasonable to anticipate increased use of such components in the future. In this instance it is critical that we enhance our understanding of the behavior of these components and improve the way that they are used. Addressing the potential challenges that storage conditions have on friction coefficients in industrial practice is not entirely straightforward. In most cases it is not practical to rigorously condition bolts, e.g., by drying, in a production process. It may be possible to use some form of position-based tightening as a practical way of maintaining consistent fastening conditions in safety critical applications where a very reliable clamp force is essential, and higher costs in manufacture can be justified, but this will not be a solution in many mass production industries where torque tightening is widely employed.

Potentially, the most valuable outcome of the research reported here may simply be an increased awareness of the possible impact of storage conditions on friction to machine designers and industrial users of threaded fasteners. One way to acknowledge this may be to adopt “spot-testing” of assembly line components, involving testing of component clamp force and friction coefficients of threaded fasteners on a regular basis. Such an approach could have two outcomes: .

1. Assembly line tightening conditions could be regularly modified on the basis of statistical testing of manufactured products to “fine-tune” a torque tightening process.
2. Weather conditions, time of year, etc., could be correlated with their impact on the characteristics of torque tightened products for future reference to guide “shop-floor” operations.

5. Conclusions

This investigation has identified profound changes in the friction behavior of Zn-flake coated threaded fasteners following storage in both cold and hot/humid conditions. .

1. The mechanisms of friction reduction appear to differ between cold storage and storage in hot/humid conditions. Friction reduction due to cold storage results mainly from free water providing fluid lubrication on the fastener surface. Friction reduction following storage in hot/humid conditions probably results from the formation of zinc oxide due to water diffusion into the coating.
2. Storage in cold conditions seems to lead to the most dramatic effects, reducing tightening torque by up to about one third on the first tightening.
3. The majority of friction reduction occurs following cold storage is due to reduction of under-head friction with reductions of 47% (compared to room storage) in some cases at the highest clamp force. This could have a significant impact on fastener tension and component clamp force when torque tightening is adopted, influencing joint safety and security as well as fastener failure in production in a commercial environment and should be addressed by appropriate measures.

Declaration of Competing Interest

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

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.triboint.2022.107498.

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