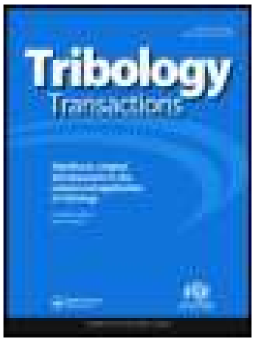

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An Experimental Study on Factors Affecting the Friction Coefficients in Electroplated Bolts

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ABSTRACT
Electroplated bolts have been widely used in the automotive industry for many decades. In the present work, for the first time, the role of each layer of the bolt coating (i.e., electroplating, passivation layer, and topcoat layer) on the coefficient of friction of a bolted joint were experimentally and systematically investigated. The coefficient of total friction (μ_{tot}), coefficient of friction between threads (μ_{th}), and coefficient of friction between bearing surfaces (μ_b) were calculated by a torque–tension testing system and compared under different experimental conditions. It was found that the μ_{th}, μ_b, and μ_{tot} on pure Zn plating was slightly different from that on Zn-Ni alloy plating; the black passivation layer had lower μ_{th}, μ_b, and μ_{tot} than the clear passivate when topcoats were applied; and the topcoat played a dominant role in reducing the μ_{tot}. Other factors including washer material, nut plating, and heat treatment were also studied. Results showed that the aluminum washer gave a dramatically higher μ_b than the steel washer. The tested plating layers of Zn, Ni, and Zn-Ni alloy on the nut did not significantly affect the μ_{th}. Tested bolts could withstand heat up to 182 °C for 1 h. Higher temperature or longer heating time increased μ_{th}, μ_b, and μ_{tot}. These findings will increase the understanding of the factors that affect the friction coefficients in electroplated bolts and will help to better design bolted joints.

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Coefficient of friction; electroplated bolts; coating

Introduction

The single largest fastener consumer is the automotive industry, accounting for approximately 42% of the fastener industry *(1)*. Bolts are the main type of automotive fasteners. Typically, bolts for the automotive industry are coated to provide corrosion resistance and to modify the torque–tension relationship, as well as to improve other properties like appearance, wear resistance, heat resistance, fluid compatibility, and so on. There are various methods for coating bolts, such as electroplating (electro-galvanizing), zinc flake coating, hot-dip galvanizing, mechanical plating, and electrocoating. Among these methods, electroplating and zinc flake coating are widely used for fasteners in the automotive industry. Although these two technologies are fundamentally different, electrolytically or nonelectrolytically applied, both technologies are based on the sacrificial properties of zinc against steel and can be used for similar applications. Generally, for zinc flake coating there is no risk of hydrogen embrittlement and the corrosion resistance is better than that of zinc electrodeposits, whereas electroplating more easily achieves a relatively homogenous coating thickness on complex-shaped parts and superior corrosion resistance can be achieved by Zn-Ni alloy electrodeposits. The present study will focus on electroplated bolts.

The coating applied to electroplated bolts typically has a triple-layer structure, as shown in Fig. 1. The first layer, the bottommost layer, includes the electrodeposits of zinc or zinc alloy, which provide good corrosion protection to underlying steel substrates both as a sacrificial layer and as a barrier *(2)*. This layer is the thickest layer among the three layers and usually has a thickness of 8–15 μm. Zinc plating has been used for many decades and is still the most common finish due to the low cost and high supplies of zinc *(3)*. Zinc alloys combine zinc and an "iron block" metal such as Zn-Ni, Zn-Fe, or Zn-Co. Among these alloys, Zn-Ni is the most successful. The commercial development and application of Zn-Ni alloy plating began during the 1980s *(2)*. Compared to pure Zn, Zn-Ni alloy offers much higher corrosion resistance *(2, 4–6)*, greater microhardness *(6–9)*, and better wear resistance *(6, 9)*. Therefore, Zn-Ni alloy has been extensively adopted by the automotive industry *(10)*. The nickel content in the alloy is usually in the range of 12–16%.

The second layer, the middle layer, is the passivation layer, which prevents the early onset of "white rust" by preventing moisture from reaching the Zn or Zn-Ni alloy surface as a barrier and by inhibiting dissolution of the metal of the substrate and the reduction of oxygen *(11, 12)*. Traditional Cr(VI)-based chromates, which can be traced back to 1936 (13), were successful for a long time but have been banned in the automotive industry by the REACH regulation in the European Union since 2017 and are severely restricted in the United States due to the fact that hexavalent chromium is

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Review led by M. Dube.

Nomenclature

D	Nominal diameter
D_b	Diameter of the bearing surface
d_2	Basic pitch diameter of the thread
F	Bolt tension or preload
K	Torque coefficient or nut factor
P	Pitch of the thread
T	Input torque
μ_b	Coefficient of friction between bearing surfaces
μ_{th}	Coefficient of friction between threads
μ_{tot}	Coefficient of total friction

toxic and carcinogenic. Today the less toxic Cr(III)-based passivate, first introduced in 1951 (14), is the preferred alternative to the Cr(VI)-based chromate. The passivation film consists basically of colloidal hydroxides of Zn(II) and Cr(III) (15), which evolve into oxides/hydroxides after drying and dehydrating (7). The layer might also contain Co or Ni oxide/hydroxide, fluoride, and possibly oxyfluoride species depending on the chemistry of the passivation solution (16, 17). The passivation layer also increases the surface adhesion for additional coatings. This layer is the thinnest in the triple-layer structure, usually less than 500 nm (e.g., 250–500 nm (12), 150–350 nm (15), and 40–340 nm (17)).

The third layer, the outmost layer, is the topcoat or sealer layer, which stabilizes the passivation layer, enhances the corrosion resistance, and modifies the surface properties such as friction, color, gloss, and wear resistance (1, 12, 18, 19). On black passivated surfaces and barrel parts (18, 19), trivalent passivation layers showed inferior corrosion performance compared to that of hexavalent chromates due to the lack of self-healing effect; thus, the use of topcoats is often mandatory to meet the corrosion demands (19). Together with the Cr(III)-based passivation layer, the topcoat layer acts as a highly efficient barrier that effectively decelerates zinc corrosion. A topcoat is also required when the torque–tension relationship needs to be controlled (19, 20). There are a wide variety of topcoats; for example, organic polymer, silicate, and silane (18). Not every topcoat is an organic matrix with other ingredients. Some could be inorganic. But in most cases, it's organic, a topcoat is an organic matrix mixed with wax, an inorganic corrosion inhibitor, and surface additives (21). After centrifuging and drying, the topcoat forms a dry film on top of the passivation layer. The thickness of this layer is usually 0.5–4 μm.

After coating, when bolts are used in mechanical and structural applications, they must be tightened to gain a certain amount of preload to hold two or more pieces together. The most widely used method to tighten a bolted joint is the torque controlling method, where a torque is applied to the head or the nut by using a torque wrench. When a bolt is tightened, it gets stretched, generating a tension on the bolt and thus a clamping force that compresses connected pieces. Eighty to 90% of the input torque will be consumed in overcoming friction, including both the underhead and the thread friction, leaving only 10–20% of the torque to generate useful clamping force (22). The less torque consumed to overcome the friction, the more clamping force can be produced by the bolt.

Friction plays a critical role in controlling the torque–tension relationship of a bolted joint. Without friction, connected parts cannot be held together because there is no locking mechanism involved in the joint (23). Too low friction will lead to an underestimation of the actual bolt tension and thus make the fastener yield or even fracture due to overstressing (24). Too high friction, on the other hand, will lead to an overestimation of the bolt tension and cause fastener loosening, joint separation, leakage, and rattle. Roughly 85% of vehicle issues are caused by assembly problems (25). Therefore, it is important to control the friction in a certain range. Most automotive companies specify an acceptable range of coefficient of friction (CoF). GM and Tesla, for example, specify that the CoF of fasteners be within the range of 0.10–0.16 (26, 27). German manufacturers typically specify a lower value; for example, Volkswagen specifies 0.09–0.15 (28).

Many researchers have studied factors affecting the frictional properties and the torque–tension relationship of a bolted joint. Among these factors, joint lubrication is highly significant. A number of previous studies have shown that the use of lubricants could significantly reduce the CoFs of a bolted joint (24, 29–33). Coating is another significant factor and can reduce the CoF (32, 34), weaken the effects of other factors (29), or change the joint's sensitivity to clamping force and repeated tightening–loosening (29, 34, 35). An increase in coating thickness moderately or slightly decreased the nut factor and the CoF (36). The washer material also significantly influences the CoF (29, 32, 34, 37). An aluminum plate had a higher bearing CoF than a steel plate (38).

Mixed results were obtained on the effect of repeated tightening–loosening. The CoF could decrease, increase, or not change with repeated tightening–loosening cycles (23, 24, 29, 34, 35, 38). In most cases, however, repeated tightening–loosening increased the friction of a bolted joint. The surface treatment, type of nut, type of washer, coating of the joint, and lubricants all could potentially affect the joint's sensitivity to repeated tightening–loosening.

In most cases, the tightening speed (24, 29, 34, 35) and clamping force (34, 37) had a marginal influence on the CoF of bolted joints. The contact area and position of the washer also had a marginal effect on the bearing friction (34). Fine threads caused a slight increase in the nut factor compared to coarse threads (35, 36). The surface roughness had a slight effect on the nut factor (33, 39). One study showed that increasing surface roughness slightly reduced the nut factor (39), whereas another study found the opposite (33).

Despite these aforementioned previous studies, there is a lack of published work that focuses on electroplated bolts and systematically studies the effects of each layer of the coating on the frictional properties of a bolted joint, even though electroplated bolts are widely used and each layer in the triple-layer coating structure could potentially play a role. Therefore, the present article is the first study that experimentally investigated the effects of each layer of the

Table 1. Dimensional and mechanical details of bolts used in this study.

Bolt parameters	M10 × 1.50 × 60mm grade 10.9	M10 × 1.50 × 40mm grade 9.8
Pitch diameter of the thread	8.86–8.99 mm	8.83–8.96 mm
Major diameter	9.73–9.97 mm	9.73–9.97 mm
Maximum grip length	34.0 mm	14.0 mm max.
Minimum thread length	26.5 mm	6.5 mm min.
Length	59.0–61.0 mm	39.2–40.8 mm
Head height	6.17–6.63 mm	6.17–6.63 mm
Across flats	15.73–16.00 mm	14.73–15 mm
Across corners	17.77 mm min.	16.76–17.32 mm
Core hardness	33–39 RC	27–36 RC
Surface hardness	77–78 R15N	75.3–78.7 R15N
Proof stress	830 MPa	650 MPa
Proof load	48,200 N	37,700 N
Tensile strength	60,400 N	52,200 N

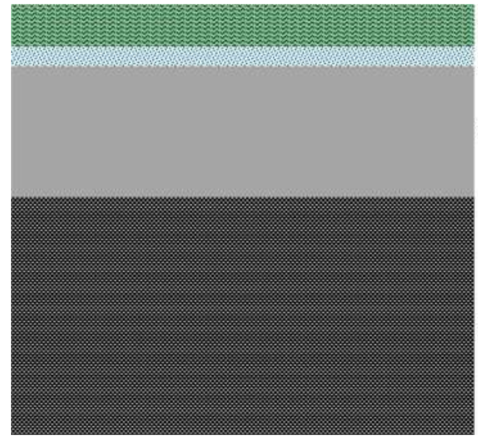

Figure 1. Schematic of a typical triple-layer coating structure of electroplated bolts in the automotive industry. The triple-layer structure consists of a layer of Zn or Zn alloy plating layer, covered by a passivation layer, with a topcoat on top.

bolt coating (i.e., plating, passivation layer, and topcoat layer) on the CoF of a bolted joint. The coefficient of total friction (μ_{tot}), coefficient of friction between threads (μ_{th}), and coefficient of friction between bearing surfaces (μ_b) were calculated by a torque–tension testing system and compared under different experimental conditions. Other factors including washer material, nut plating, and heat treatment were also studied. This study will increase the understanding about the factors that affect the CoFs in electroplated bolts.

Theoretical foundation

In the torque controlling method, the relationship between the torque applied and the bolt tension generated is often simplified as presented in Eq. [1]:

$$T = KDF, \qquad [1]$$

where T is the input tightening torque applied to the fastener head or nut, K is the torque coefficient or nut factor, D is the nominal diameter of the bolt, and F is the bolt tension or preload. When there is no external load applied, the system works like a series of mechanical springs during the tightening process, so the bolt tension F equals the clamping force in magnitude. The nut factor, K, sums up the effects of all variables affecting the torque–tension relationship, such as friction, the pitch or angle of bolt threads, the contact area, and the contact pressure distribution. It is assumed that the torque–tension relationship is linear in most cases as predicted by theory and as implied by the ISO 16047 standard *(40)*. It should be pointed out, however, that when the nut factor K is dependent on the clamping force, a nonlinear torque–tension relationship could occur, as observed in a number of previous studies *(33, 34, 38, 41)*.

According to the DIN EN ISO 16047 standard *(40)*, the coefficient of total friction is approximately determined using Eq. [2]:

$$\mu_{tot} = \frac{\frac{T}{F} - \frac{P}{2\pi}}{0.577d_2 + 0.5D_b}, \qquad [2]$$

where μ_{tot} is the coefficient of total friction, d_2 is the basic pitch diameter of thread, D_b is the diameter of the bearing surface under the bolt head or nut, and P is the pitch of the thread.

Substituting Eq. [2] into Eq. [1], one can obtain the following equation:

$$K = \frac{T}{DF} = \frac{1}{D}(0.577d_2 + 0.5D_b)\mu_{tot} + \frac{P}{2\pi D}. \qquad [3]$$

One can see from Eq. [3] that the nut factor basically depends on two things: μ_{tot} and bolt geometry parameters. Generally, for the same type of bolts, their geometric parameters are constant; thus, there is a linear relationship between the nut factor K and μ_{tot}. The higher the total friction, the higher the nut factor, indicating the importance of friction in the torque–tension relationship.

Experiment

Materials

Grade 10.9 (M10 × 1.50 × 60mm) and 9.8 bare steel bolts (M10 × 1.50 × 40mm) were used in this study. See Table 1 for dimensions and mechanical details. The sample preparation and treatment is shown in a flow diagram in Fig. 2. Purchased bolts were tumbled in a tumbler first in an alkaline cleaner for at least 2 h, then in an ultrasonic bath for 30 min, and finally in an electro cleaner for 5 min with an immediate water rinse after each step. Following cleaning, bolts were activated in an acid pickle of diluted hydrochloric acid for 1 min and then rinsed with water. Then bolts were electroplated in a Zn or Zn-Ni alloy bath, dipped in a trivalent chromium passivate for 30 s or 1 min, and finally dipped in a topcoat for 1 min and dried in a spin dryer at 1,075 rpm for 5 min. Water rinses were applied after each step except after topcoat dipping. For studies on the effects of heating, finished bolts were baked in an oven and then cooled to room temperature. For other tests, finished bolts were not baked. The cleaner, electroplating bath, passivate, and topcoat are all products of a private research company. Unless otherwise noted, the following products were used in this study: an acid Zn-Ni water bath that consists of $ZnCl_2$, $NiCl_2$, H_3BO_3, and other additives; a Cr(III)-based clear passivate with the components of $Cr(NO_3)_3$, water, and other proprietary additives; and a water-based organo-mineral topcoat,

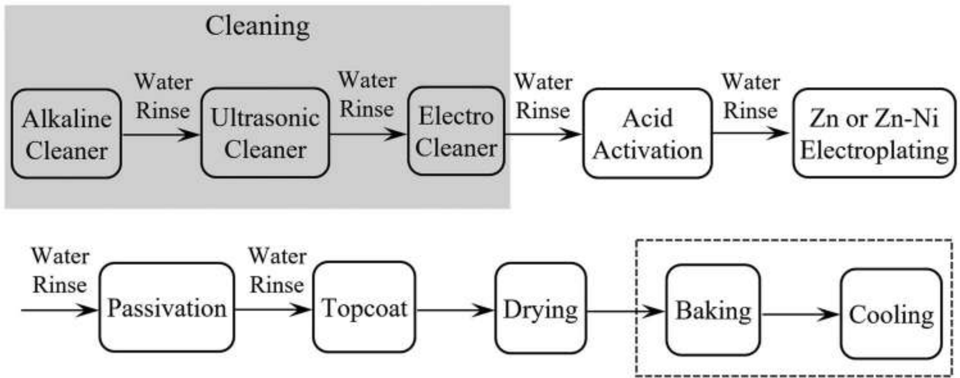

Figure 2. A flow diagram for the sample preparation and treatment of bolts. The steps in the grey box are for cleaning. The steps in the dashed-line box are only applied to study the effect of heating on the CoF of a bolted joint.

Table 2. Details of washers used in this study.

Washer parameters	M10 square plain	M10 square Zn-plated	M10 square aluminum
Square dimension	29.5–30.0 mm	22.7–23.3 mm	22.7–23.1 mm
Hole diameter	11.0–11.2 mm	10.3–10.5 mm	10.3–10.5 mm
Thickness	2.3–2.4 mm	1.8–2.0 mm	1.8–3.1 mm

Table 3. Details of nuts used in this study.

Nut parameters	M10 × 1.5 plain	M10 × 1.5 Zn-plated
Minor diameter	8.4–8.7 mm	8.4–8.6 mm
Thickness	8.0–8.4 mm	8.7–9.1 mm
Across flats	15.7–16.0 mm	14.7–15.0 mm
Proof load	60,300 N	60,296 N

which is composed of water, an organic polymer binder, a wax emulsion, and other proprietary additives. Prepared bolts were cured for 48 h at room temperature and then were subjected to torque–tension measurements.

The thickness of the electroplating layer studied in this article ranged from 6 to 12 μm, measured by a nondestructive X-ray diffraction method; the thicknesses of the passivation layer and topcoat layer were in the range of 70–114 nm and 260–583 nm, respectively, measured by a focused ion beam scanning electron microscope (FIB-SEM) technique.

M10 square test washers and M10 × 1.5 hex nuts were used in this study; their dimensions are shown in Tables 2 and 3, respectively. Zn-plated steel washers and nuts were used as received. Plain steel washers and nuts were tumbled in a tumbler first in an alkaline cleaner for at least 2 h and then in an ultrasonic bath for 30 min and finally dried in a spin drier at 1,075 rpm for 5 min immediately prior to torque–tension measurements. Zn-Ni- and Ni-plated washers and nuts were obtained by electroplating plain steel washers and nuts in the Zn-Ni and Ni plating bath following the cleaning and electroplating processes shown in Fig. 2. No passivation layers and topcoats were applied on washers and nuts. Unless otherwise specified, Zn-plated washers and nuts were used for torque–tension measurements.

Torque–tension measurements

The torque–tension measurements were carried out on a commercial fastener testing system, as shown in Fig. 3, to determine the CoFs, including μ_{tot}, μ_{th}, and μ_b. The experimental setup was described in previous studies *(35, 36)*. The measuring procedure was the same for all bolts, which were treated under different experimental conditions to have different plating, passivation, and topcoat layers. For each test, after a bolt, a washer, and a nut were properly assembled in the apparatus by hand, the DC electric motor applied an input torque to the bolt head until a shut-off target was reached. Then the bolt was loosened to its starting position. The shut-off parameter was a predetermined tension point; that is, 36,100 N for grade 10.9 bolts and 28,300 N for grade 9.8 bolts, representing 75% of the proof load of each bolt grade. Real-time test data including input applied torque (T), thread torque (T_{th}), and clamp load (F) were collected and used to calculate CoFs μ_{tot}, μ_{th}, and μ_b based on Eqs. [2], [4], and [5], respectively *(40)*. A constant turning speed of 30 rpm was used in each test. A linear torque–tension relationship was observed in all tests conducted in this study, with an example shown in Fig. 4, which is generated automatically by the apparatus. All tests were performed under normal room temperature conditions. To study the effect of heating, bolts were baked in an oven and then cooled in air to room temperature prior to torque–tension measurements. For each measurement, at least 10 bolts were tested and the average value was used.

$$\mu_{th} = \frac{\frac{T_{th}}{F} - \frac{P}{2\pi}}{0.577 d_2} \quad [4]$$

$$\mu_b = \frac{T - T_{th}}{0.5 D_b F}. \quad [5]$$

Results and discussion

Effect of plating

Electroplated Zn or Zn-Ni alloy is the first layer applied to the substrate in the triple-layer coating structure. Automotive manufacturers normally specify the thickness of this layer to be in the range of 8–15 μm. Variation in the thickness is common in electroplating due to the difference in current density in different areas depending on the fastener geometry, the barrel's ability to uniformly tumble fasteners, and possible overloading of fasteners. Figure 5 shows

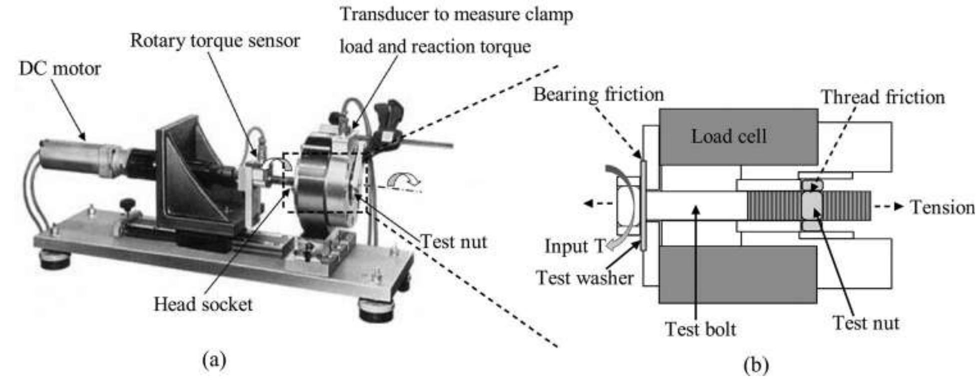

Figure 3. Experimental setup for measuring the coefficient of friction: (a) Fastener testing assembly and (b) schematic of the torque–tension head.

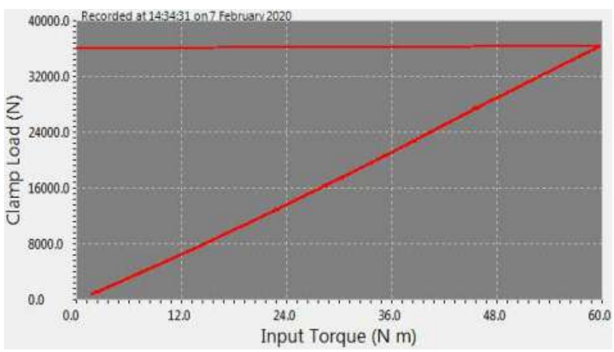

Figure 4. Representative clamp load vs. input torque curve.

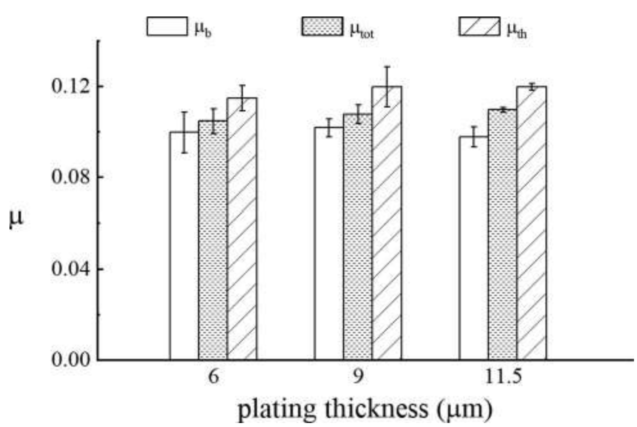

Figure 5. Effect of the thickness of the plating layer on the CoF of a bolted joint.

the effect of plating layer thickness on the μ_{th}, μ_b, and μ_{tot} of a bolted joint. As shown, within the studied range (6.0–11.5 μm), plating thickness has a marginal effect on μ_{tot}, μ_{th}, and μ_b. In a previous study, Nassar and Zaki *(36)* also studied the effect of coating thickness on the μ_{th} and μ_b of bolted joints. Two bolt coating thicknesses, 16.4 and 22.6 μm, were compared. Their results also showed that coating thickness did not play a significant role in the CoFs in bolts.

From Fig. 5, one can also note that μ_{th} was always higher than μ_b. This finding was in agreement with previous studies *(23, 29)*. Possible reasons include the following: *(1)* the pressure present in the threaded area is higher than that between the bearing surface (see Fig. 3b for a sketch of the bolt: the threaded area is inside the nut and the bearing surface is between the bolt head and the washer), leading to increased wear particles and ploughing; *(2)* the deformation and distortion of threads increase the friction; *(3)* the male threads go into the female threads like a wedge, increasing the resistance to bolt rotation; and *(4)* the cut surface of the female thread is often rougher than the surface of the washer. Nevertheless, Grabon et al. *(41)* reported that the mean μ_b was higher by about 44% than the mean μ_{th} in their experiments. It should be mentioned that it is necessary to have the same condition to compare μ_{th} and μ_b, such as the same material and the same surface treatment. In Grabon et al.'s experiments, the washers and nuts were not the same material. Figure 5 also shows that the value of μ_{tot} is between μ_{th} and μ_b because μ_{tot} is the coefficient of the overall friction in the joint combining friction from both the threaded and bearing surfaces.

Commercially available Zn and Zn-Ni plating baths can be broadly divided into either acidic or alkaline based. Figure 6a compares different plating systems, including chloride Zn, alkaline Zn, acid Zn-Ni, and alkaline Zn-Ni. Zn-plated steel washers and nuts were used in the measurements. As shown, for the Zn plating systems, chloride Zn and alkaline Zn coatings showed similar μ_{th}, μ_b, and μ_{tot}. For the Zn-Ni plating systems, the alkaline Zn-Ni bath gave slightly higher μ_{th}, μ_b, and μ_{tot} than the acid Zn-Ni bath.

The comparison between pure Zn and Zn-Ni alloy coatings showed that if Zn-plated steel washers and nuts were used (Fig. 6a), Zn-Ni alloy coatings showed higher μ_{th}, μ_b, and μ_{tot} than pure Zn coatings. If Zn-Ni-plated steel washers and nuts were used (Fig. 6b), opposite results were obtained. This phenomenon is probably due to the fact that Zn-Ni alloy coating is harder than pure Zn coating *(6–9)*. When pure Zn and Zn-Ni alloy surfaces are in contact and relative motion, the asperities on the Zn-Ni surface plough the Zn surface, increasing the real contact area and friction. Different surface morphologies of pure Zn and Zn-Ni alloy coatings might also play a role *(6, 9)*. Pure Zn film has a pyramidal morphology, whereas Zn-Ni alloy shows a cauliflower-like morphology. Future investigation of the microstructure and hardness of the coating is needed to better understand the mechanism.

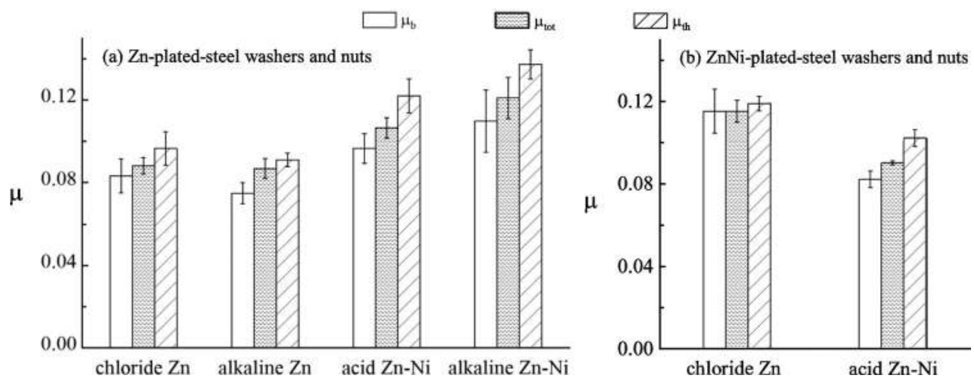

Figure 6. Effect of different plating systems on the CoF of a bolted joint: (a) Zn-plated steel washers and nuts were used in the measurements and (b) Zn-Ni-plated steel washers and nuts were used in the measurements.

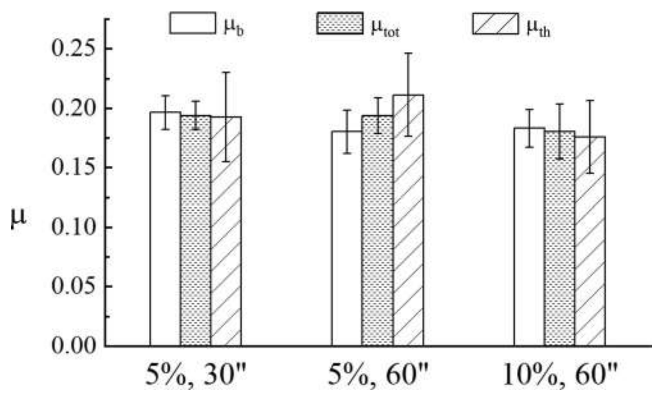

Figure 7. Effect of dipping time and concentration of the clear passivation solution on the CoF of a bolted joint. Bolts were dipped in the (left) 5% clear passivation solution for 30 s, (middle) 5% clear passivation solution for 60 s, and (right) 10% clear passivation solution for 60 s.

Effect of passivation layer

The passivation layer is the second layer applied to the substrate in the triple-layer coating structure. Figure 7 shows the effect of dipping time and concentration of the passivation solution on the μ_{th}, μ_b, and μ_{tot} of a bolted joint. The tested passivation solution was a clear solution. Two dipping times (i.e., 30 and 60 s) and two dipping concentrations (i.e., 5 and 10%) were compared. As shown in Fig. 7, the dipping time and concentration of the passivation solution did not change the μ_{th}, μ_b, and μ_{tot} much. This may be because the passivation film reaches a limiting thickness when nitrate (oxidant) in the passivation solution no longer contacts the substrate and thus the film thickening is ceased *(16)*. When the immersion time was further increased, a part of the deposited $Cr(OH)_3$ was dissolved back into the solution as $[Cr(OH)_4]^-$ due to an increased interfacial pH *(16)*.

The use of black passivation layer coatings is often favored by the automotive industry. Figure 8a compares the clear and black passivation layers, showing that μ_{th}, μ_b, and μ_{tot} were all lower on the black passivation layer than on the clear passivation layer. The tested black passivation layer consists of $Cr(NO_3)_3$, $CoSO_4$, and other proprietary chemicals. Bolts were sealed by topcoats in this test. The reason behind the result is still unknown. To further study this phenomenon, another experiment was conducted without applying topcoats after passivation, with the result shown in Fig. 8b. When no topcoat was applied, clear and black passivation layers had similar μ_{th}, μ_b, and μ_{tot}, suggesting that the black passivation layer and the topcoat may play a synergistic role in reducing the friction.

In order to better understand the involved mechanism, it is helpful to look at the processes of the formation of the trivalent chromium passivate layer. When a clear passivation solution is used, the formation of the passivation film can be simplified into the following three steps *(15, 16, 19)*.

Step 1: When pure Zn or Zn-Ni alloy coating is immersed in the passivation bath, zinc dissolution, reduction of the oxidant, and hydrogen evolution occur at the interface between the Zn or Zn-Ni alloy surface and the passivation bath. In the Cr(III)-based passivation process, the oxidant is mainly nitrate *(19, 42)*.

Step 2: Due to the consumption of protons in step 1 and thus a local pH increase near the interface, the soluble Cr^{3+} and Zn^{2+} ions hydrolyze and precipitate on the interface, building up a very thin passivation layer. Depending on the chemistry of the passivation bath, precipitates of $Co(OH)_2$ and CrF_3 may also form *(16)*. The chemical state of Cr(III) in the film was mainly revealed as hydroxide *(16, 43, 44)*, oxide *(43, 45)*, and fluoride *(16, 44)*.

Step 3: If the immersion time is too long, a part of the precipitates re-dissolves into the bulk solution *(16)*.

When a black passivation solution is used, the formation of black finishes using Cr(III)-based passivation solution is generally similar to that of black hexavalent chromates *(19)*. For the black chromate, it was proposed that the chromate conversion film was formed of two superimposed layers *(46, 47)*. The inner layer was a very thin black pigment layer formed at the zinc or zinc alloy surface. It consisted of blackish oxides of chromium, nickel, or other transition metals such as iron or cobalt *(19, 46)*. The outer layer (the major part of the passivation layer) was formed from the precipitation of the trivalent chromium and consisted of Cr_2O_3, $Cr(OH)_3$, $Cr(OH)CrO_4$, $Zn_2(OH)_2CrO_4$, and a small amount of absorbed H_2O *(46)*. The formation processes for the outer layer are similar to those described for the clear passivation layer. Due to the formation of black pigments in the inner layer, the growth of the outer layer is commonly

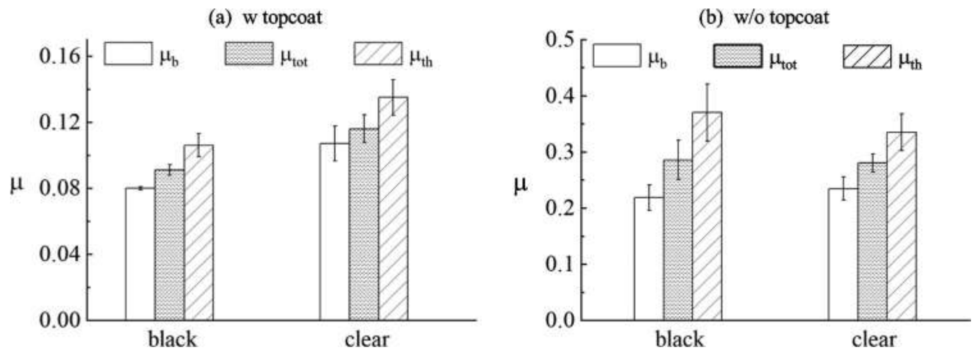

Figure 8. Effect of the type of passivation layer (black vs. clear) on the CoF of a bolted joint: (a) Bolts were sealed by a topcoat and (b) bolts were not sealed by a topcoat.

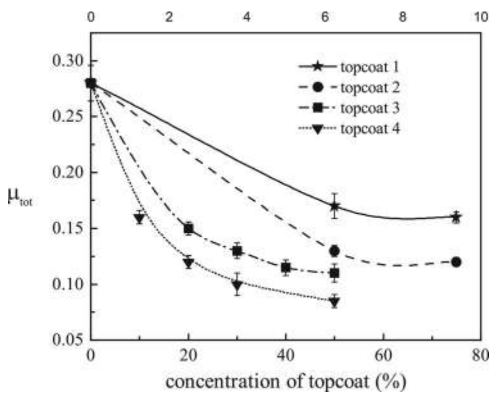

Figure 9. Effect of topcoat on the CoF of a bolted joint.

found to be limited *(19)*. It is possible that the hardness of the black passivation layer is slightly lower than that of the clear passivation layer due to the formation of the inner layer, causing a lower CoF. Future in-depth research using methods like SEM, X-ray photoelectron spectroscopy, and FIB-SEM is needed to completely understand the mechanism.

Effect of topcoat

Trivalent passivation layers do not improve the torque–tension relationship and thus a subsequent topcoat is usually required. Topcoats can control the friction of bolts and can be tailored to satisfy different customer requirements. Four different topcoats were investigated in this study and the results are shown in Fig. 9. It is apparent that topcoats played a significant role in modifying the frictional properties of the bolts. When there was no topcoat applied (i.e., the concentration of the topcoat is 0), μ_{tot} was ~0.28, which is in agreement with previous studies *(19, 20)* that showed that without a topcoat or sealer, the CoFs of a bolted joint were high (0.27–0.41); these values are not acceptable per most automotive specifications. When a topcoat was applied, μ_{tot} decreased significantly to as low as 0.08, representing a 71% reduction to the coating without topcoats. It was also shown that topcoats can reduce the scatter of the μ_{tot}. Jiang and co-workers *(34)* also reported that organic coatings reduced the CoF and its scatter. Different topcoats have different strengths in reducing μ_{tot} depending on the type of waxes used in the topcoat.

After the topcoat is applied and topcoat dried in a centrifuge, water in the topcoat evaporates and the topcoat layer becomes a dry self-lubricating film, which is composed of nanoparticles of organic polymer binder and wax and some additives. The mechanisms of wax behavior in topcoats are complex *(48)*. One mechanism is that the wax migrates to the surface of the coating and forms a continuous film, providing a protective layer with properties of lubrication and abrasion resistance. In another mechanism, the waxes in the coating exist as discreet and independent particles on the surface of the coating, with the wax particles protruding from the surface of the coating. Thus, the wax particles become the first contact point with the contacting surface and act as spacers between two surfaces. In most cases, a combination of both mechanisms occurs.

Effect of washer material

Friction is a system property; therefore, all surfaces involved in the joint should be considered. Figure 10 shows the effect of washer materials on the μ_b of a bolted joint. Two types of bolts (grade 10.9 and 9.8 bolts) were tested in this experiment and similar results were obtained. As can be seen from Fig. 10, plain steel, zinc-plated steel, and Ni-plated steel washers show similar μ_b (~0.11), whereas the Zn-Ni-plated steel washer exhibits a slightly lower μ_b (~0.10). For the aluminum washer the μ_b was ~0.29, which was dramatically higher than that of steel washers. This finding is in agreement with a previous study *(38)* that reported that an aluminum plate had a higher bearing friction than a steel plate. The hardness of the tested bolts, steel washers, and aluminum washers was around 350, 500, and 100 HV, respectively. When an aluminum washer was used, the difference in hardness caused increased ploughing and/or microcutting of asperities of the hard bolt surface into the soft aluminum washer surface, increasing the real contact area and resistance to relative motion *(49, 50)*. A previous study *(49)* showed that hard metals had lower frictional resistance than softer metals because the atomic bonds were stronger in hard metals and hence the resistance to adhesion from the contacting surface was increased.

The main building material used in the automotive industry is still relatively cheap steel; nevertheless, the use of aluminum has grown continuously during the last 4 decades

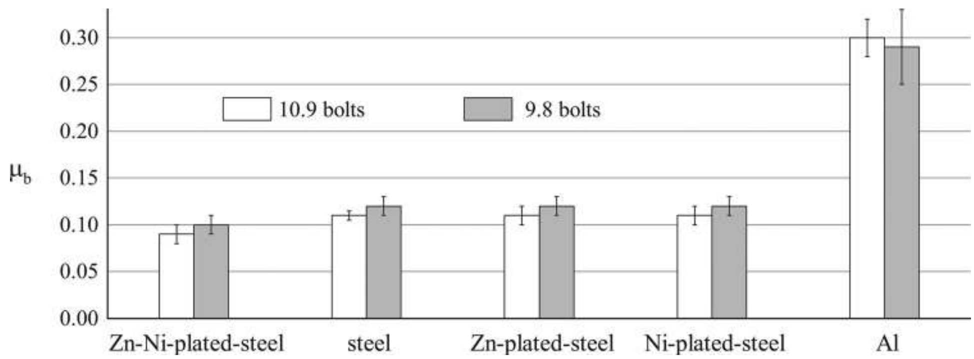

Figure 10. Effect of washer material on the CoF of a bolted joint.

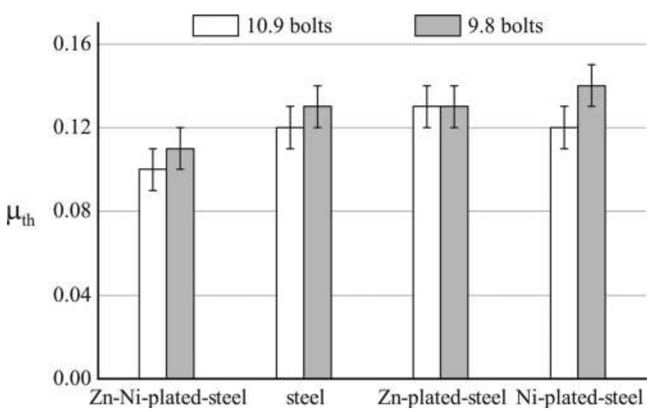

Figure 11. Effect of nut plating on the CoF of a bolted joint.

due to its capability to enhance fuel efficiency, vehicle performance, safety and corrosion resistance and to reduce CO_2 emissions *(51, 52)*. In addition, at the end of a vehicle's life, nearly 90% of the aluminum, on average, is recycled. Despite these advantages, aluminum exhibits tribological problems, including poor resistance to seizure and galling *(52)*. Tightening grade 10.9 (or 9.8) bolts onto aluminum will result in the acceptable bearing pressure being exceeded and plastic collapse. Prasad and Mecklenburg *(53)* showed that the friction coefficient between aluminum and steel couples was high ($\sim$0.5–0.6). The present work showed a lower friction coefficient ($\sim$0.29) due to the coatings on the bolts. This value is still too high for automotive companies. This problem can be solved either by improving the lubricity of coatings on bolts or by reinforcing aluminum through dispersing solid lubricants, hard ceramic particles, short fibers, and whiskers in aluminum *(52)*. Based on the model developed by Xie and Williams *(50)*, increasing the hardness of the softer surface raises the limit at which plasticity intervenes and reduces the overall friction. Al-based particulate composites, chiefly SiC/Al and Al/Al_2O_3, have been successfully applied in the automotive industry in pistons, engine blocks, disc rotor brakes, drums, calipers, connecting rods, drive shafts, and other parts *(52)*. On the other hand, improvements in the lubricity of the bolt coating will further increase the applications of aluminum in the automotive industry. In the present study, nanoparticles were not added in the Zn-Ni electroplating layer. The work by Shourgeshty et al. *(54)* showed that the incorporation of Al_2O_3 nanoparticles into the Zn-Ni electrodeposits reduced the CoF, indicating that Al_2O_3 nanoparticles acted as a lubricant in the coating. Mahidashti et al. *(55)* estimated that incorporation of lubricant particles such as graphite, MoS_2, and polytetrafluoroethylene (PTFE), into electrodeposits would reduce the CoF.

It should also be mentioned that tightening of bolts onto aluminum can be overcome by the use of a SEMS (https://trademarkalertz.com/trademark/75830619-SEMS) unit (a washer being held captive on the bolt shank) or a KEPS (https://trademarkalertz.com/trademark/76398273-KEPS) unit (a washer being held captive on a nut). Full tightening of a plain hexagon headed bolt/nut joint of property grade 10.9 onto aluminum will lead to surface yielding and a subsequent loss of preload due to relaxation effects. In such situations a car manufacturer would use a flange headed fastener or a hardened washer.

Effect of nut plating

Figure 11 compares μ_{th} on different plating layers of the nuts. Two types of bolts (grade 10.9 and 9.8 bolts) were tested and similar results were obtained. All nuts are steel based but have different plating metals, including pure Zn, pure Ni, and Zn-Ni alloy. As shown, the tested plating metals did not play a significant role in changing μ_{th}. Steel and Zn- and Ni-plated steel nuts show similar μ_{th}, whereas Zn-Ni-plated steel nuts show a slightly lower μ_{th}. The thickness of the plating layers on the nut ranged from 6 to 11 μm, which might be too thin to change the hardness of the coating and thus the μ_{th} of the bolted joint.

Effect of heating

The automotive industry requires fasteners that can resist high temperature. For example, fasteners used in the engine compartment are expected to have a temperature load of up to 150 °C. The exhaust manifold, catalytic converter, and turbocharger areas all require high-temperature-resistant fasteners to ensure optimum performance. Higher efficiency engines today and beyond will place stronger demands on the heat resistance of bolts.

Figure 12 shows the effect of heating temperature on the μ_{th}, μ_b, and μ_{tot} of a bolted joint. Finished bolts were baked in an oven at different temperatures for 1 h and then cooled in air to room temperature. As shown in Fig. 12, μ_{th}, μ_b, and μ_{tot} all remained similar up to a baking temperature of

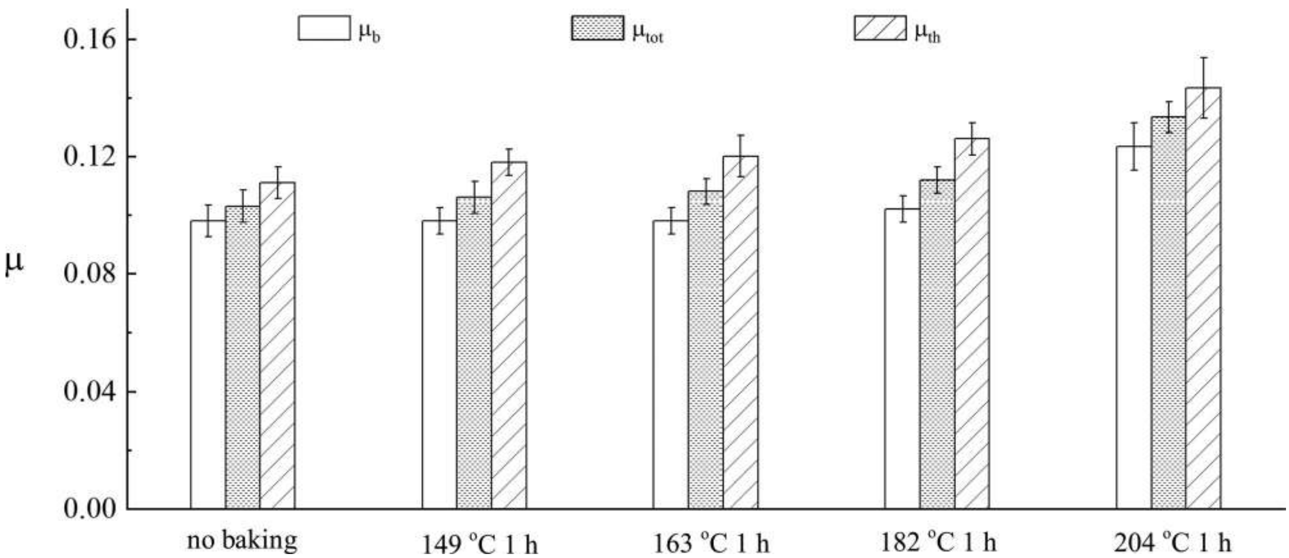

Figure 12. Effect of heating temperature on the CoF of a bolted joint.

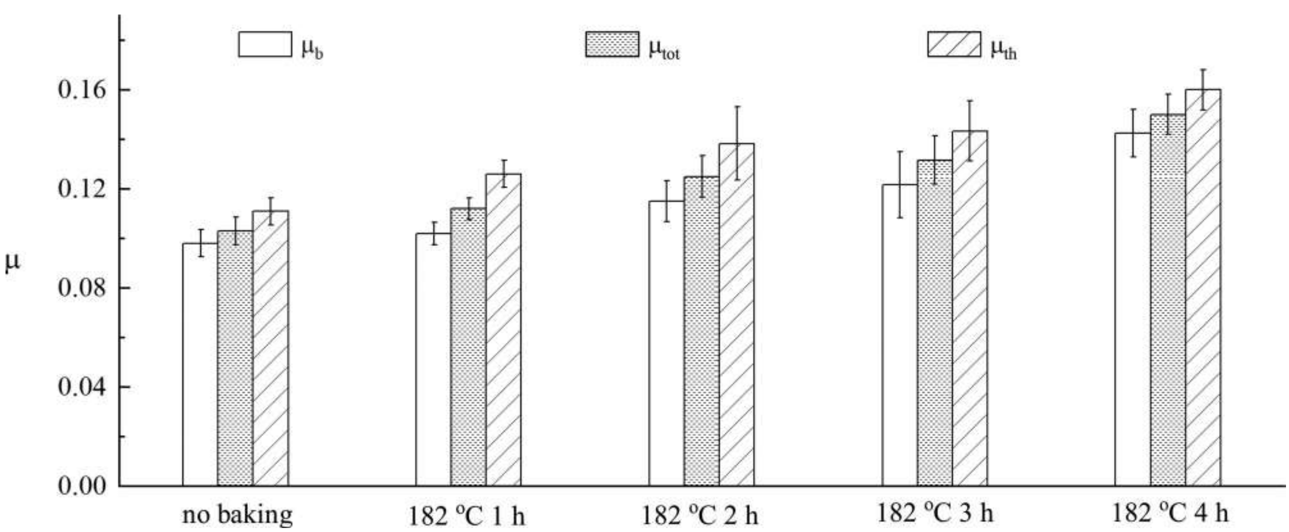

Figure 13. Effect of heating time on the CoF of a bolted joint.

163 °C; as the temperature increased to 182 °C, μ_{th}, μ_b, and μ_{tot} increased slightly; as the temperature further increased to 204 °C, the CoFs further increased. Figure 13 shows the effect of heating time on μ_{th}, μ_b, and μ_{tot}. Finished bolts were baked in an oven at 182 °C for different time durations and then cooled in air to room temperature. As shown, μ_{th}, μ_b, and μ_{tot} all increased with increasing heating time. The increase in these CoFs caused by heating at high temperature or long exposure time might be attributed to the melting or decomposition of the wax used in the topcoat, which changes the surface structure and morphology. The size of the wax nanoparticles on the surface might decrease and the surface might become much smoother after heat treatment, leaving less wax particles protruding from the surface. This issue needs further in-depth investigation.

Waxes used in the tested topcoat have limited heat resistance. Other lubricants such as MoS_2, graphite, and PTFE can be incorporated into the film to improve the heat resistance of the coating. PTFE and MoS_2 would be suitable for temperatures up to 260 and 400 °C, respectively *(56)*. Graphite will lubricate effectively above 400 °C. Waxes can only withstand heat up to 182 °C. Mahidashti et al. *(55)* reviewed some nickel-based electrodeposited tribo-coatings and showed that the CoF at high temperatures (up to 300 °C) decreased in the coatings containing graphite and MoS_2 and the graphite-containing coating had the best wear behavior at all temperatures ranging from 25 to 300 °C. Compared to other lubricants, however, wax is widely used due to its low cost. In addition, the densities of MoS_2, graphite, and PTFE are all much higher than that of water and thus tend to settle down at the bottom, creating a challenge to develop a well-dispersed stable topcoat.

The studied coating of Zn-Ni electrodeposits followed by a passivation layer and topcoat is frequently used in industry, and the use is still growing due to its excellent corrosion resistance. Compared to other electrodeposited tribofilms, such a film is relatively thin (8–16 μm) and thus is used to reduce the thickness of zinc coatings, avoiding the problems of low ductility and weldability caused by thick zinc coatings (>25 μm). It is also used to coat parts with a complex shape at a relatively more uniform thickness than zinc flake coating. In addition, this coating protects aluminum from galvanic corrosion in contact with steel and therefore is widely used in industries that deal with aluminum bodies *(10)*. Nevertheless, Zn-Ni alloys are considered expensive in view of the high nickel price.

Conclusion

The present article is the first published study that systematically investigated the role of each layer of the bolt coating (i.e., plating, passivation layer, and topcoat layer) on the μ_{th}, μ_b, and μ_{tot} of a bolted joint. The following conclusions can be drawn: *(1)* Topcoats played a more dominant role in controlling the μ_{tot} of a bolted joint. Applying topcoats can significantly decrease μ_{tot}. *(2)* The black passivation layer had lower μ_{th}, μ_b, and μ_{tot} than the clear passivation layer when the topcoat was applied. The dipping time and concentration of the passivation solution did not change the μ_{th}, μ_b, or μ_{tot} much. *(3)* The plating layer thickness had a marginal influence on μ_{th}, μ_b, and μ_{tot}. Different plating systems slightly affected μ_{th}, μ_b, and μ_{tot}.

This study also investigated other factors including washer material, nut plating, and heat treatment. It was found that the aluminum washer had a dramatically higher μ_b than the steel washer. The tested plating layers on the nut surface did not significantly affect μ_{th}. The heat resistance of tested bolts was good up to the temperature of 182 °C for 1 h. Higher temperature or longer heating time increased μ_b, μ_{th}, and μ_{tot}.

The information obtained here will increase the understanding of the factors that affect the friction coefficients in electroplated bolts and will assist the design and applications of industrial processes in the fastener, electroplating, and automotive industry.

Recommendations for future work

Future in-depth research on the microhardness, microstructure, and morphology of bolts with different variables is recommended to better understand the mechanisms related to each studied factor, such as why the black passivation layer has a lower CoF than the clear passivation layer. Use of more advanced equipment such as SEM, X-ray photoelectron spectroscopy, and FIB-SEM could be helpful.

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