

# Evaluation of environmentally friendly Ag-PTFE composite coating for use in threaded compression fittings

R. Sieh<sup>1</sup>, H.R. Le<sup>2</sup>

<sup>1</sup>School of Marine Science and Engineering, University of Plymouth, United Kingdom

<sup>2</sup>Department of Engineering, College of Engineering and Technology, University of Derby, United Kingdom

## Abstract

Threaded tubular fittings are used in a wide variety of industries for critical applications involving fluid transfer in a pressurised or vacuum system. These fittings are made out of corrosion resistant metals such as stainless steel which are desirable in corrosive operating conditions; however, stainless steel is prone to galling which can cause threads to seize, resulting in loss productivity. To prevent this, threads are electroplated using silver (Ag) coatings which prevent galling and serve as a solid lubricant during the make-up process. The Ag cyanide electroplating process currently used in industry is both hazardous to human health and its wastes are detrimental to the environment. The objective of this work is to evaluate environmentally friendly self-lubricating Ag and Ag-PTFE coatings using a non-cyanide electroplating process against the commercially available cyanide Ag coating through the analysis of torque-angle signatures and the torque-angle slope which characterises the make-up process. Results from the experiments suggest that the non-cyanide Ag-PTFE coating is a potentially viable replacement option. Investigation and analysis of the coating performance have also highlighted potential risks of failure through poor lubrication during the make-up process and suggestions for improving the make-up process.

## Key Words:

Non-cyanide; silver plating; Ag-PTFE; compression fittings; threaded connections; pulse electroplating;

## Nomenclature

$I$  = Current in Coulombs per second

$t$  = Time in seconds

$A$  = Atomic weight of the metal in grams per mole

$n$  = Valence of the dissolved metal in solution in equivalents per mole

$F^*$  = Faraday's constant (96,485.309 Coulombs/equivalent)

$T^*$  = Thickness of deposit in microns

$\rho$  = density in grams per cubic centimetre

$S$  = surface area of the part in square centimetres

$T$  = Torque applied or input torque

$F$  = Force generated during tightening of the nut

$C$  = Constant (friction factor)

$P$  = Thread pitch

$\mu_{thread}$  = Coefficient of friction in threads

$r_t$  = Effective radius of thread contact

$\cos \beta = 30^\circ$  for UN/ISO threads

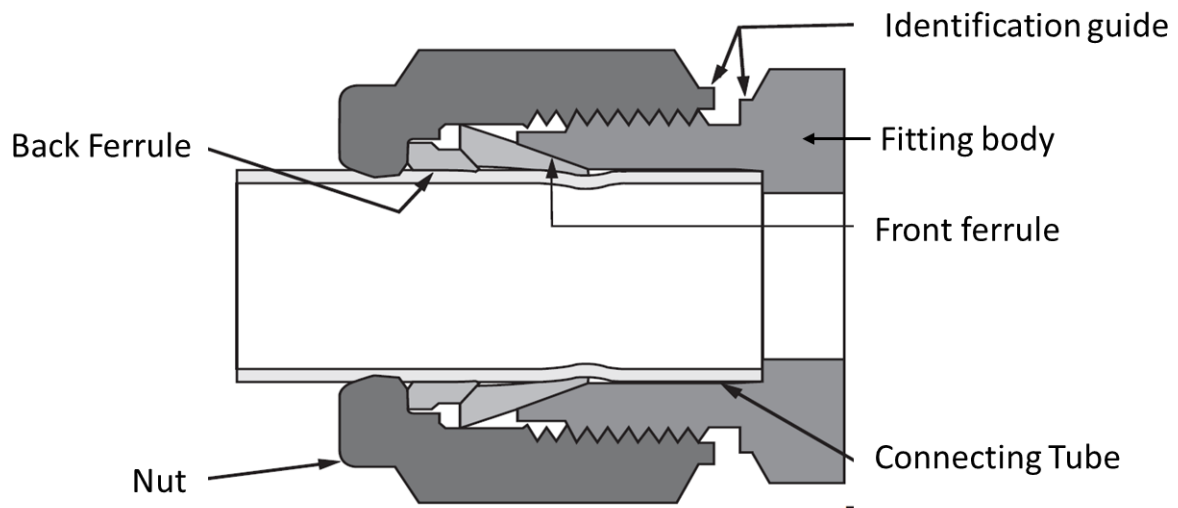
$\mu_b$  = Coefficient of friction under head

$r_n$  = Effective radius of nut under-head contact

CoF = Coefficient of friction

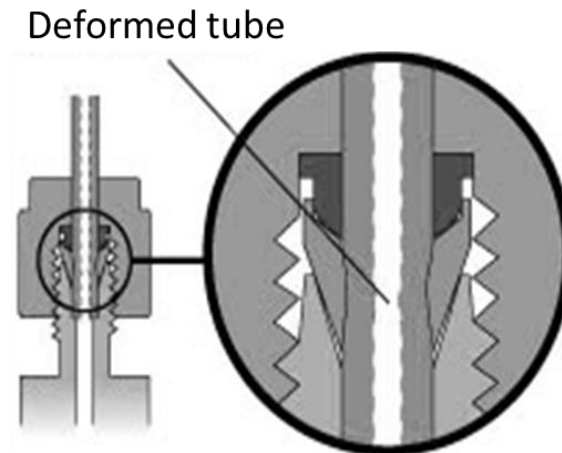
## 1. Introduction

Compression tubular fittings which can be in the form of one or two piece ferrules are used in various industries including oil and gas, chemical, semiconductor, bio-tech, and so on for securing fluid tubing connections. These ferruled compression tube fittings can be used in a wide range of critical applications due to its ability to provide leak-tight seals, including instrumentation connections, hydraulic, pneumatic, power, refrigeration and various other applications that involve the transportation of fluid using either metal or plastic tubing<sup>1</sup>. Manufacturers often offer this system where reliability in high pressure situations during the connection of metal tubing is required. This is achieved through the sealing surface of the system, where a leak-tight seal is crucial to prevent liquids or gases from escaping during its use. For a double ferrule compression fitting as shown in Figure 1, the sealing surface is where the front ferrule meets the fitting body. Based on the design of this compression fitting system, there is a reduced risk of the sealing surface rotating when tightening force is applied, thus preserving seal integrity during its installation.



*Figure 1: Cutaway section of a double ferrule compression fitting*

A double ferrule compression fitting system is illustrated through the use of a cutaway section in Figure 1, and comprises of several key elements, including the fitting body, front ferrule, back ferrule, connecting tube and the nut. The system works on the basic concept of crimping which deforms the tube in the process, highlighted in Figure 2. During the tightening process, the nut travels in the direction of the fitting body and exerts a compressive force on the back ferrule. The back ferrule in turn transfers a compressive force on the front ferrule to compress the connecting tube, guided by the angled fitting body. The system is designed to create a liquid tight seal, resist loosening of the nut due to vibration and prevent blow outs when in either vacuum or pressure systems during its operational life<sup>2</sup>.



*Figure 2: Compression fitting system - tube deformation of compression fittings*

Fittings suitable for use with metal tubing are typically made of the same metal as the tube. The inner diameter of the ferrules will be the same as the outer diameter of the tubing used. For high performance, non-cost prohibitive applications, such as instrumentation connections which require high corrosive metals such as stainless steel to be used, an added challenge to be considered is the susceptibility of stainless steel to galling between the contact surfaces. Galling is a phenomenon of surface damage as a result of sliding contact between solid surfaces under conditions of inadequate or no lubrication and under high contact pressure which is visible through macroscopic effects of surface roughening or protrusions<sup>3,4</sup>. Under high compressive forces, the adhesion of the materials will cause slipping and tearing of crystal structures beneath the surface, which often results in material transfer, plastic flow or friction welding between the sliding surfaces.

For stainless steel compression fittings, a coating of silver is typically applied to the inner faces of the nut, including its threads to prevent galling and thus seizures during the make-up process of the compression fitting. Silver is also known for its tribological advantage of reducing the friction coefficient between the sliding contact surfaces and has been in used within the aerospace industry for several decades<sup>5</sup>. The use of silver within the aerospace industry has been primarily in fasteners, where low friction silver coated self-locking nuts are used in the demanding conditions within aerospace engines<sup>6</sup>. Furthermore, as a relatively noble metal it is corrosion resistant, a desirable material property for applications in highly corrosive environments.

Silver coatings can be obtained through electroplating, which is a relatively low cost method of generating a surface coating which is scalable at the same time; however, the traditional silver plating methods used by industry increase the risk to health and safety as a result of the toxic cyanide component in the plating bath<sup>7,8</sup>. Furthermore, the disposal methods for silver cyanide electroplating wastes are both costly and are of detriment to the environment and successful silver electroplating using a non-cyanide silver plating bath has been reported<sup>9,10</sup>.

Apart from electroplating pure metals such as silver, there has been a relatively new trend of incorporating other materials such as polymers during the electroplating process to form a metal matrix composite (MMC)<sup>11</sup>. It is possible to improve the tribological properties of a silver MMC through the addition of the fluoropolymer PTFE which is already established in its use as a solid lubricant for various applications<sup>12</sup>. However, those familiar with the PTFE polymer are also aware of the issues surrounding its handling during the incorporation process as it is chemically inert and has a low surface tension. The issue of surface tension can be overcome through the addition of a suitable surfactant, which changes the surface tension of PTFE as it is suspended in the electroplating bath. Surfactants have been used for this and one that has been used successfully for PTFE is the FC-4 surfactant<sup>13</sup>.

The electroplating process is one that can be carried out through several methods, namely through the use of a direct current method, pulsed current method or a pulsed reverse current method. The direct current method is carried out by the application of a direct potential and current in an electroplating bath; on the other hand, pulsed current or pulse reversed current methods use the rapid alternating of current between different values in a series of pulses with the amplitude, polarity and duration separated by zero current<sup>14-16</sup>. Changes in the electroplating methods will result in changes to the microstructure of the coating. The advantages of pulsed plating over typical direct current plating apart from improved throwing power is that it is able to generate coatings that are harder with smaller grain sizes and of low porosity as well as being able to have increased foreign particle incorporation<sup>17-19</sup>.

There is a tribological benefit of reducing the friction coefficient when incorporating PTFE particles into the electroplated silver metal matrix composite<sup>10</sup>. The issue of friction in threaded connections is one that has been studied in detail for threaded fasteners such as bolts and nuts, where the conversion of torque input to the applied forces within the system is greatly affected by friction<sup>20</sup>. A practical technique for evaluating and verification of the force achieved for fasteners is the torque-angle curve. The torque-angle signature method is carried out through the examination of the tightening and loosening curves during the installation process, through plots of torque versus angle during its installation and removal<sup>21</sup>.

## 2. Experimental method

The performance of non-cyanide Ag and Ag-PTFE coatings were tested along with the commercially available cyanide silver coated nuts through a torque experiment. Based on the assembly instruction for the compression fitting provided by the manufacturer, the whole compression fitting assembly should be made up and the nut tightened to finger tight prior to applying torque using a torque wrench. An angle target of 450 degrees is provided by the manufacturer for making up the specified assembly without any specific torque specification. To ensure a consistent finger tight value is achieved across the experiment, all the nuts are pre-tightened to a value of 2Nm. For the reassembly process, although certain manufacturers state that lubricants may be applied if required, none will be applied for all the experiments.

Each of the nut coatings were tested for 5 attempts using the equipment setup as shown in Figure 3. Prior to testing, the test parts (nut, front ferrule, back ferrule, body and tube) were thoroughly cleaned using acetone and alkaline to remove any residual contamination from the manufacturing and electroplating process. The torque and angle from the attempts were recorded using the data acquisition and recording TL Box supplied by Norbar. To ensure consistent velocity across the tightening attempts, each tightening attempt was completed in 4 seconds.

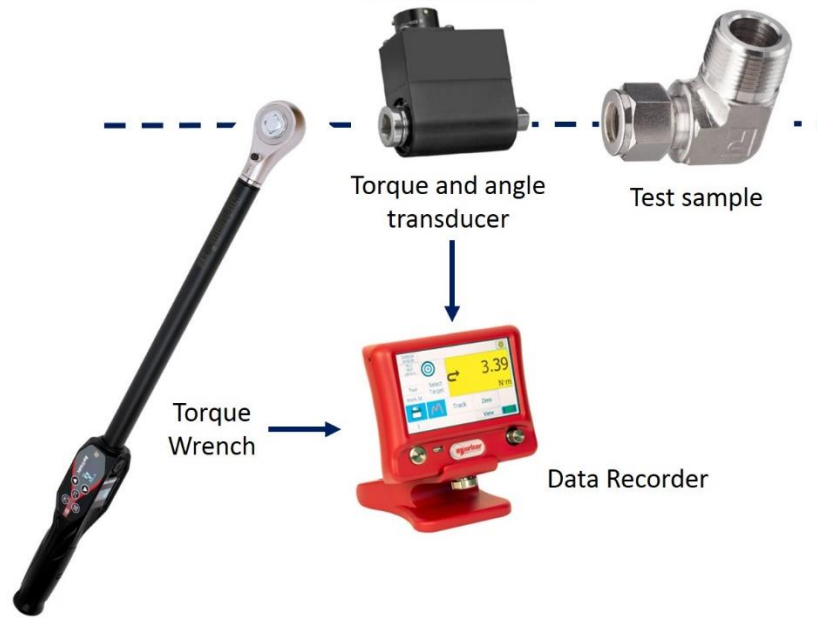


Figure 3: Torque experiment equipment setup

For the electroplating of the non-cyanide Ag and Ag-PTFE coatings, a two-electrode anode-cathode system coupled with a programmable power supply model BK9174 supplied by BK Precision Electronics was used in conjunction with the non-cyanide silver plating cell to carry out the electroplating of the 7/16" UNF thread class 2 compression fitting nuts. The silver-PTFE composite coatings were obtained from a succinimide silver nitrate bath, with composition and operating conditions shown in Table 1. The only difference between the silver and silver composite plating bath is the addition of PTFE along with a surfactant. For the Ag-PTFE coating, an FC-4 surfactant was used to lower the surface tension of PTFE particles prior to its addition into the bath at a PTFE to FC-4 ratio 1:1. To ensure that the PTFE particles are homogeneously mixed and continually suspended in the bath, the bath was magnetically agitated at a speed of 500 RPM. The pH value was adjusted to 9.5 using potassium hydroxide.

Composition	Conditions
Silver Nitrate (g/L)	34.7
Succinimide (g/L)	80.0

Solution pH	9.5
Temperature (°C)	28.0

Table 1. Non-cyanide silver plating electroplating bath composition and parameters

As with any surface finishing operations, sample preparation and pre-treatment prior to electroplating is a vital part of the process to ensure a repeatable, consistent finishing quality is achieved for all the nuts summarised in Table 2. Prior to the pre-treatment process, all the uncoated nut threads were thoroughly cleaned using a non-abrasive nylon brush to remove any large foreign debris. After a thorough rinse of the nuts using DI water, the pre-treatment process started off with the immersion of the nuts in a high purity acetone solution which was then put into an ultrasonic bath for approximately 5 minutes. Another rinse using DI water followed and the nuts were then dried and immersed in an alkaline cleaning solution containing sodium hydroxide, sodium carbonate, tribasic sodium phosphate and sodium metasilicate in ultrasonic bath for further 5 minutes. After a final DI water rinse, the nuts were dried using hot air at approximately 60°C. After drying, the external surfaces of the nut were masked using a non-conductive primer which was left to dry in ambient air for a minimum of 24 hours. After priming and before electroplating, pickling of the cathode surface was carried out using a 5% hydrochloric acid solution. The electroplating process for Ag-PTFE was carried out using pulse plating with a frequency set at  $f = 10\text{Hz}$  and constant current with a density of  $0.2\text{ A/dm}^2$ . On the other hand, the pure non-cyanide silver electroplating was carried out using a constant current method of the same current density. A pure silver (99.9% purity) acting as the anode was used in both instances. Pulse plating was chosen for the Ag-PTFE to aid incorporation of the PTFE particles into the MMC, where coupled with cationic FC-4 surfactant, high PTFE incorporation (in excess of 10 wt.%) was noted using EDS on test plates.

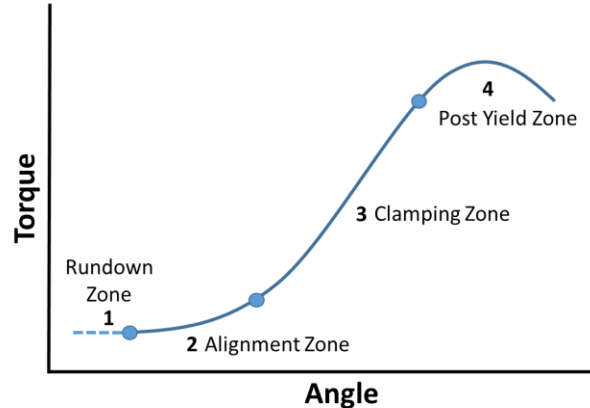
Procedures	Conditions
Acetone cleaning	u/s bath 300s at ~20°C
Alkaline cleaning	u/s bath 300s at ~20°C
Rinsing Temperature	~20°C
Acid etching, 5% w/v HCL	240s at ~20°C
Rinsing Temperature	~20°C
Ag-PTFE Electroplating	28°C
Rinsing	~20°C
Drying	60°C

Table 2. Sample pre-treatment prior to electroplating

### 3.0 Results, discussion and Analysis

#### Torque-angle signature curve

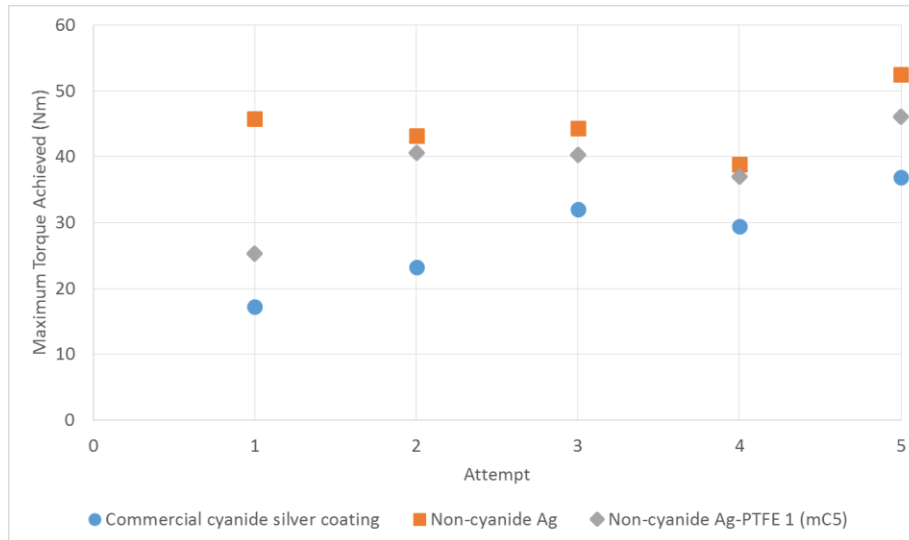
Hand torque experiments were carried out based on the setup discussed in the previous section in an attempt to understand the performance of each coating during the make-up process. Data was continually recorded across the 450 degree rotation range and used for further analysis based on the torque-angle signature method. This method is commonly used to characterise the fastener installation process and can thus be used as a reference to characterise the compression fitting installation process. As shown in Figure 4, threaded fasteners require a rundown zone which is also called the prevailing torque zone. The assembly process of threaded compression fittings achieves this through finger tightening of the nut. The make-up of compression fittings will also experience snugging alignment where the components and contact surfaces are either drawn into alignment or into a stable situation. This nonlinear area of the curve is a complex function of the drawing together the mating parts and ensuring a snug fit of the system. The third zone is where elastic clamping occurs for fasteners and where crimping occurs for compression fittings. Under normal situations, the components in the compression fitting system will not exceed the yield and as such the fourth zone will be primarily applicable to threaded fasteners is where the yield point of the joint, gasket, threads or clamped components has been exceeded.



*Figure 4: Generic torque-angle signature curve for fasteners showing the four distinct zones used to characterise the installation process*

As expected, the torque-angle signature curve for the threaded compression fitting make-up experiments followed the exhibited pattern of the fastener installation process, with the exception of zone 4 which is post yield. The maximum magnitudes of torque for each coating has been presented in a graphical format in Figure 5. Comparing the coatings based on each attempt, the commercial cyanide silver plated coating exhibited consistently lower maximum tightening torque values when compared to the non-cyanide Ag and Ag-PTFE coatings.



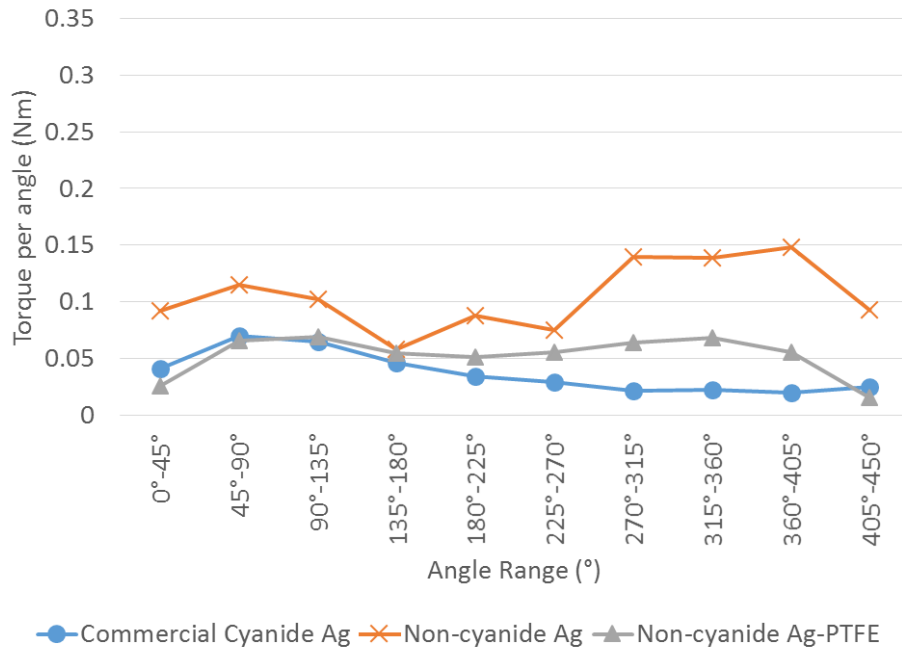


*Figure 5: Maximum magnitudes of torque in Nm recorded during the make-up process for each coating*

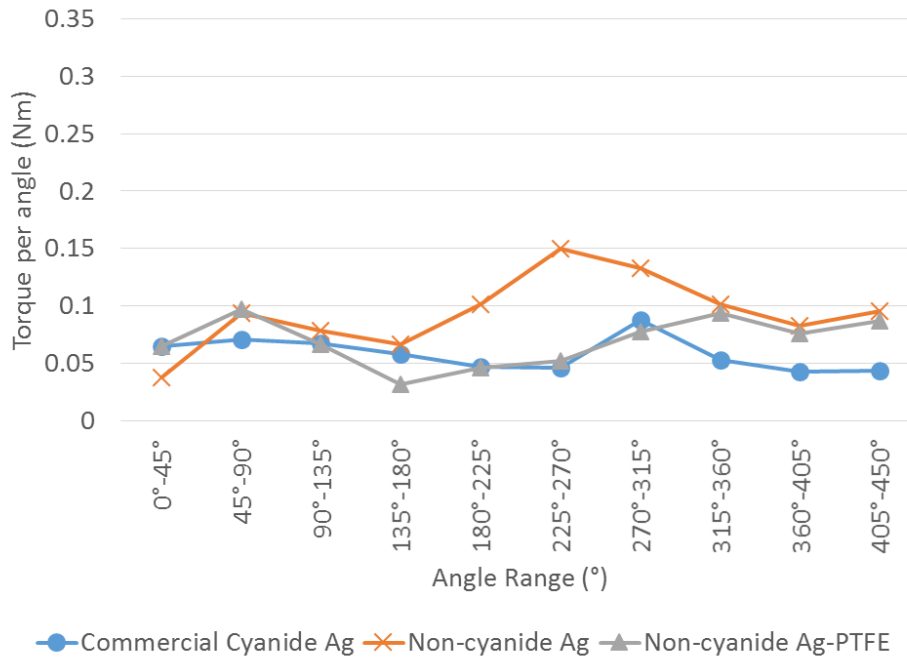
Although being useful in providing the maximum torque achieved during make-up, this technique does not provide adequate information about each coating performed for an informed comparison and analysis to be made.

### Torque-angle slope

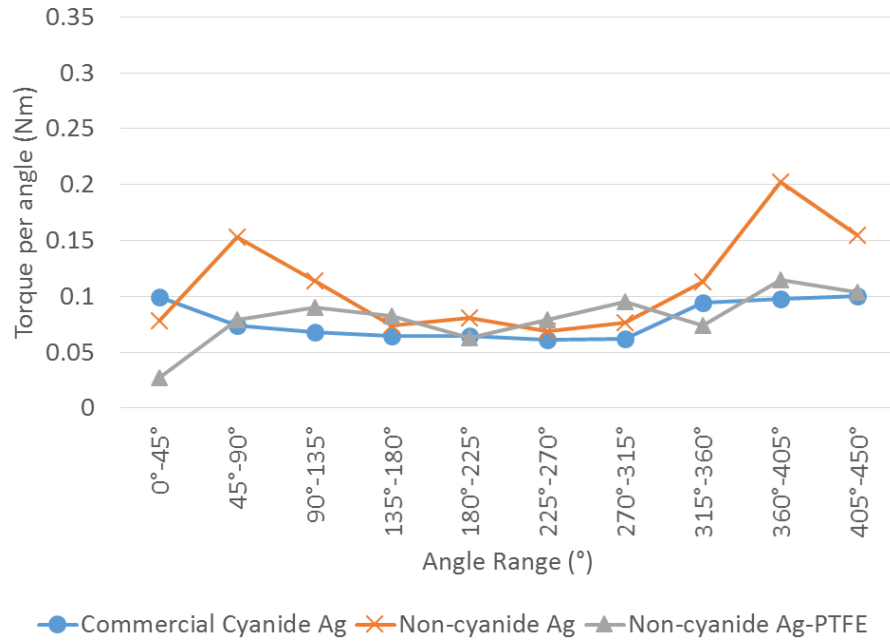
The torque-angle slope from the crimping process was considered to better understand the performance characteristics of the coatings. This slope is calculated using the torque over angle and is broken down into 10 rotation ranges from the continuous torque and angle measurements. Each range covers 45 degrees of rotation for the slope to be calculated. This in turn provides the average force in Nm per angle for each rotation range. The corresponding graphs plotted provides the coating performance characteristics for the coating as shown in Figure 6.



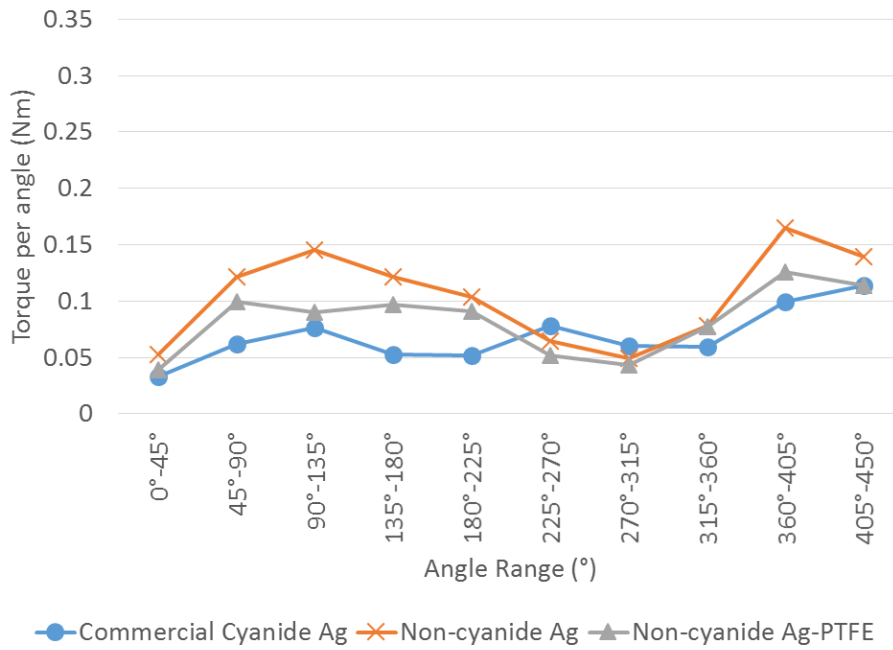
(a)



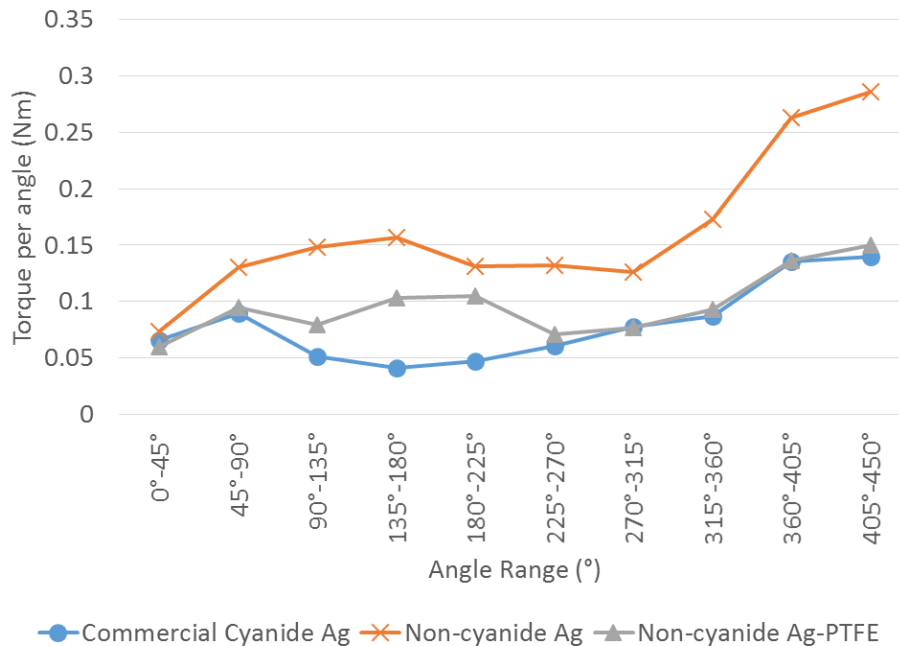
(b)



(c)



(d)



(e)

Figure 6: Coating performance characteristics for the 5 make-up attempts using the torque-angle slope analysis method for commercial cyanide Ag, non-cyanide Ag and non-cyanide Ag-PTFE. Each point represents the torque required to turn the nut 1°, grouped in 45° degree increments (a) Make-up attempt 1, (b) Make-up attempt 2, (c) Make-up attempt 3, (d) Make-up attempt 4, (e) Make-up attempt 5

### Commercial cyanide Ag coating performance

The commercial cyanide Ag coating thickness was 15 microns. The performance exhibited during the first make-up attempt showed that the average torque peaked between 45-90 degrees of rotation before exhibiting a downward trend in torque per angle. This downward trend was present on the first and second make-up attempts, but the magnitude of the downward trend gradually decreased. The following 3 make-up attempts then exhibited an upward trend that is increasing in magnitude.

The post testing inspection revealed traces of a transfer film between contact areas which would have aided in the lubrication during the make-up process. Visual inspection of the coating also indicated that the first few threads of the coated nut experienced heavy wear which is typical of threaded parts. The underside of the nut which is in contact with the back ferrule also experienced heavy wear. Wear induced during the first make-up attempt had resulted in coating performance reduction for the following make-up attempts. There were several latter peaks which were observed in Figure 6(b) and 6(d) which suggests that the wear had broken down the transfer film which was providing lubrication between the contact surfaces. Analysis of the spread of the torque measurements as well as its

distribution suggests the increase in wear of the coating under the high operational stresses which have resulting in its impairment in providing adequate solid lubrication after consequent make-up processes; however, it had still managed to maintain a relatively stable performance.

### Non-cyanide Ag coating performance

This study is focused towards the performance of the non-cyanide Ag-PTFE coating compared against the current commercial silver cyanide coating, and an assumption was made that a thinner non-cyanide Ag-PTFE coating could have equivalent performance when compared with a thicker commercial silver cyanide coating. The non-cyanide pure Ag coating in this instance serves as a control and was deposited using similar parameters to that of the non-cyanide Ag-PTFE coating.

The theoretical coating thickness based on the electroplating parameters for the non-cyanide Ag coating was 3.83 microns, whereas measured thickness placed this at approximately 3.3 microns. Theoretical coating thickness calculations in microns were based on Faraday's law of electrolysis:

$$T^* = \frac{I * t * A * 10,000}{n * F * \rho * S} \text{ --- Eq (1)}$$

Current efficiencies for direct current electroplating are typically around 90%; however, electroplating a complex geometry will result in uneven deposit thicknesses due to uneven current distribution. In terms of the torque-angle slope, a consistent higher overall slope average suggests higher friction is inherent in the system. Although post testing visual inspection revealed traces of transfer film on the contact surfaces, it was less apparent as opposed to the commercial cyanide Ag coating. Examination of the maximum torque values achieved during each make-up process reveals that the coating had consistently achieved the highest torque. This is further supported by the torque-angle slope analysis, where the non-cyanide Ag coating required the highest average torque per angle to make-up. Furthermore, the coating had behaved erratically and relatively unpredictably during make-up. In relative terms, this is a clear indication of poor coating wear characteristics which can be attributed to a thinner overall coating.

### Non-cyanide Ag-PTFE coating performance

The non-cyanide Ag-PTFE coating managed to exhibit a more consistent performance over its non-cyanide Ag counterpart; however, its performance was still generally below that of the commercial cyanide Ag coating. Based on the maximum torque observed during the make-up process, the non-cyanide Ag-PTFE coating required on average approximately 8Nm more input torque. One major factor could be the coating thickness achieved for the current coating. A theoretical thickness of 2.3 microns was calculated based on a typical pulse plating current efficiency value of 60%. The maximum measured thickness was 2.1 microns in this instance. Post make-up visual inspection showed moderate amounts of transfer film residue in relation to the other coatings considered. It is believed that the PTFE embedded in electroplated silver contributes to providing a more stable friction and overall increased tribological performance of the coating.

## Excess torque and wear

An assumption can be made that approximately 17Nm of tightening torque is sufficient for successful make-up of the system based on ideal conditions. This value represents the lowest torque value from the cyanide silver coating after a 450 degree cycle of rotation. However, significantly higher readings of maximum torque in the torque-angle curve were registered for all the coatings tested during the make-up attempts which require an adequate explanation as to where this excess torque has gone in the system. Furthermore, a lot of work has gone into the study of the relationship between input torque and applied force for threaded fasteners (nut and bolt) which is potentially transferrable to explain this during the study of threaded compression fittings. The relationship can be expressed through the short form equation as:

$$T = F * C \text{ --- Eq (2)}$$

Although the constant “C” is an experimentally determined factor and is subject to variations, the straight line relationship between torque applied and force achieved will always be assumed to be accurate<sup>22</sup>. The application of this relationship requires the long form equation, where there are 3 forms of long form equations that are generally used which yield the same results. These include the ISO 16047 equation which has been attributed to Kellerman and Klein, DIN946/VDI2230 equation and Motosh’s equation<sup>23</sup>.

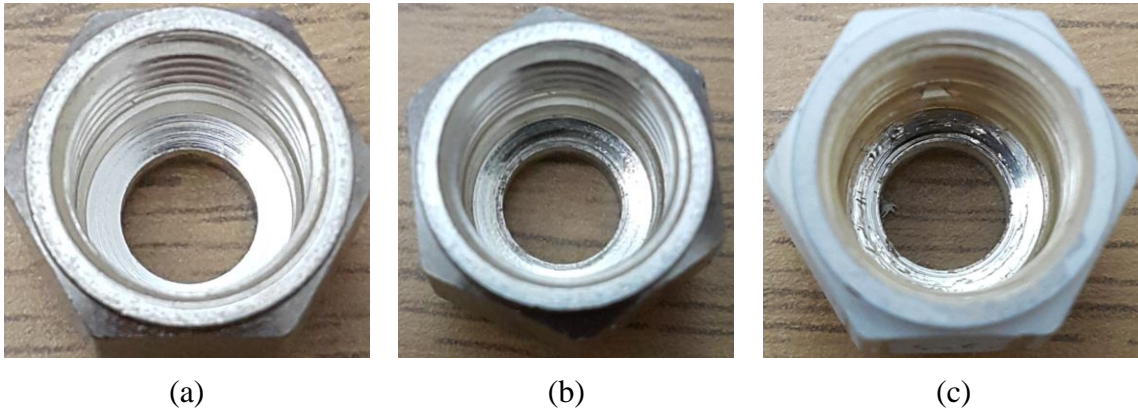
Motosh’s equation below is in a format that is easiest to understand, with constituent terms that make up the equation for the constant, namely thread pitch incline plane, thread friction and nut under-head friction resistance.

$$T = F \left( \frac{P}{2\pi} + \frac{\mu_{thread} r_t}{\cos \beta} + \mu_b r_n \right) \text{ --- Eq (3)}$$

Assuming that Motosh’s equation is valid for threaded compression fittings as the considerations are the same, it can be rearranged to consider a global CoF value as follows:

$$\mu_{global} = \frac{\cos \beta \left( \frac{T_{input}}{F_{opposing}} \frac{P}{2\pi} \right)}{r_t + r_n \cos \beta} \text{ ----- Eq (4)}$$

The global CoF in Eq (4) then is directly affected by the input torque and instead of the force generated during tightening for fasteners, the opposing force should be considered. This opposing force component is comprised of the material resistance from the crimping process. An increase in the input torque directly results in the proportionate increase in global CoF. This only explains what is actually happening in an ideal situation though, where issues such as coating wear are not taken into consideration. The global CoF is therefore useful as a simplified equation of understanding the theoretical friction within the system during initial design. The actual application of this during the make-up process is challenging as there are first and foremost inherent variations in the component or assembly which is further compounded by the changes in contact pressures during coating wear.



*Figure 7: Underside of the nut (a) Untested commercial Ag cyanide coating (b) Commercial Ag cyanide coating after 5 make up attempts (c) Non-cyanide Ag-PTFE coating after 5 make up attempts*

In reality, during the compression fitting make-up process, there are several unlubricated contact surfaces, namely the interfaces between the front ferrule with the body as well as the interface between the back and front ferrule. If the CoF between these unlubricated contact surfaces are greater than the CoF between the back ferrule and the coated (lubricated) nut, the torque application will cause the lubricated surfaces to slip and rotate. The rotation of surfaces results in wear on the underside of the nut as can be seen in Figure 7. Upon continuous make-up using new ferrules with no previous transfer film residue to aid in lubrication, the CoF of the worn nut coating will exceed the CoF between the unlubricated surfaces. This will result in the front ferrule rotating against the body during make-up. As these surfaces are unlubricated, galling damage will occur along with adhesion between the unlubricated contact surfaces. As the opposing forces within the system increase, the application of a higher input torque is required to overcome this resulting in the front ferrule pushing downwards and outwards. This then creates extra resistance in the system when the front ferrule expands and comes into contact with the nut to create an extra contact surface. This very phenomena was observed as shown in Figure 8 and can be used to explain high peaks on the torque-angle slope for the coatings tested. When this occurs, several outcomes are possible. The worst case is when there is insufficient crimp force exerted onto to the tube and increased risk of system failure under high operational pressures. This is quite hard to detect during installation unless the whole system is disassembled and checked. Apart from that, there is also the possibility of the catastrophic screw thread failure due to internal stress build-up when galling has occurred to a point of internal component seizure. This is detectable but will cause unwanted downtime and cost.

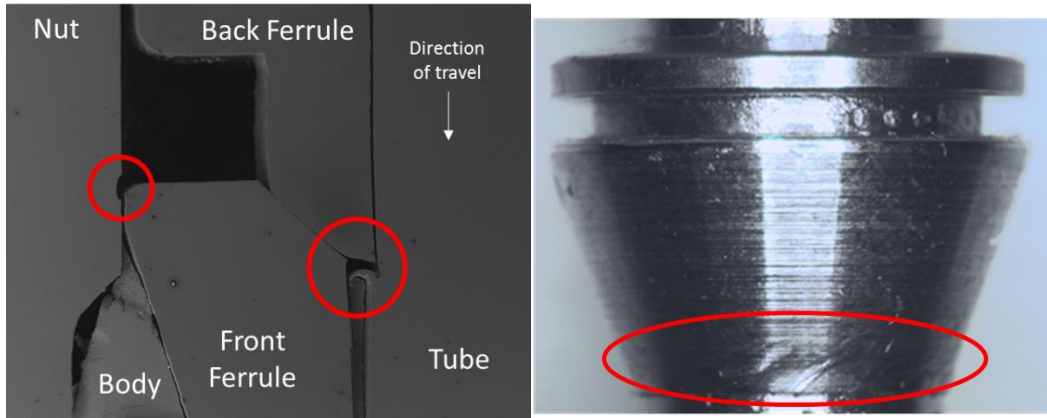


Figure 8: Observed damages from 5<sup>th</sup> attempt of non-cyanide Ag coating (Left) to the tube and nut as circled (Right) Galling damage on front ferrule

On the other hand, minor fluctuations in the average torque per angle value can also be explained through the phenomenon of adhesive wear. Adhesive wear causes a phenomena of stick-slip to occur at unlubricated surfaces or where the coating has been excessively worn during the sliding process. In the case of adhesive wear, a higher amount of force is required to overcome the sticking between sliding surfaces. Once this has been overcome, both surfaces will be able to slide with a lower force requirement. The torque per angle of turn manages to capture the sticking and slipping process that occurs throughout the sliding process.

In the context of having an Ag coating on stainless steel substrate, the relatively soft Ag or Ag-PTFE is able to provide low friction with the detriment of having higher wear<sup>24</sup>. A soft coating on a hard substrate will function by firstly increasing the pressure contact area through deformation and as a result decreases the interfacial shear strength for the hard substrate<sup>25</sup>. The soft coating on the nut provides a low friction during make-up; however, the life of the coating will be short due to high wear of the coating as the rough surfaces rotate, creating abrasive wear on the coating. As such, manufacturers typically recommend that nuts are not reused after it has been disassembled.

## 4.0 Conclusions

Experiments were carried out on non-cyanide Ag, Ag-PTFE along with commercially available cyanide Ag coatings. Torque-angle signatures were captured for the make-up process which were used to characterise the performance of the coatings. A detailed analysis for the coatings called for the use of the torque-angle slope. This provided the much needed resolution in aiding the evaluation of each coating set. The non-cyanide Ag coating exhibited erratic performance over the experiments and lagged significantly behind the cyanide Ag coating. Overall, the commercial cyanide Ag exhibited the best performance which was closely followed by the non-cyanide Ag-PTFE coating. The results of the Ag-PTFE coating which was approximately 5 times thinner than the commercial cyanide Ag coating suggests its potential use as a viable replacement when deposited to a sufficient thickness.



Prior to and during the course of the experiments, several key observations were made in relation to the make-up instructions provided by manufacturers. Firstly, the finger tightening operation stipulated is a variable, where different installers will exert different forces onto this. Secondly, the make-up process is only given an angle target. Inadequate lubrication can result in a large proportion of applied torque subjected to losses within the system and not on the crimping process. The dissection of the parts after the experiments highlighted the fact that inadequate lubrication can result in irreversible damage to the system. It is recommended that the make-up of the compression fittings be to both a torque and angle specification, especially for critical applications where component damage is not an option.

Potential future work in relation to this could include the use of servo motors in applying torque at a consistent velocity from an experimental point of view and also testing equivalent electroplated thickness coatings over a larger number of make-up attempts.

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