

ARCHIVIO ISTITUZIONALE DELLA RICERCA

Alma Mater Studiorum Università di Bologna Archivio istituzionale della ricerca

Threaded fasteners with applied medium or high strength threadlockers: effect of different tightening procedures on the tribological response

This is the final peer-reviewed author's accepted manuscript (postprint) of the following publication:

Published Version:

Threaded fasteners with applied medium or high strength threadlockers: effect of different tightening procedures on the tribological response / Croccolo, Dario; De Agostinis, Massimiliano; Fini, Stefano; Olmi, Giorgio; Paiardini, Luca; Robusto, Francesco. - In: JOURNAL OF ADHESION. - ISSN 0021-8464. - STAMPA. - 96:1-4(2020), pp. 64-89. [10.1080/00218464.2019.1679630]

This version is available at: https://hdl.handle.net/11585/703645 since: 2024-05-03

Published:

DOI: http://doi.org/10.1080/00218464.2019.1679630

Terms of use:

Some rights reserved. The terms and conditions for the reuse of this version of the manuscript are specified in the publishing policy. For all terms of use and more information see the publisher's website.

(Article begins on next page)

This item was downloaded from IRIS Università di Bologna (https://cris.unibo.it/). When citing, please refer to the published version. This is the final peer-reviewed accepted manuscript of:

D. Croccolo, M. De Agostinis, S. Fini, G. Olmi, L. Paiardini & F. Robusto (2020) Threaded fasteners with applied medium or high strength threadlockers: effect of different tightening procedures on the tribological response, The Journal of Adhesion, 96:1-4, 64-89, DOI:10.1080/00218464.2019.1679630

The final published version is available online at: https://doi.org/10.1080/00218464.2019.1679630

Terms of use:

Some rights reserved. The terms and conditions for the reuse of this version of the manuscript are specified in the publishing policy. For all terms of use and more information see the publisher's website.

This item was downloaded from IRIS Università di Bologna (<u>https://cris.unibo.it/</u>)

When citing, please refer to the published version.

THREADED FASTENERS WITH APPLIED MEDIUM OR HIGH STRENGTH THREADLOCKERS: EFFECT OF DIFFERENT TIGHTENING PROCEDURES ON THE TRIBOLOGICAL RESPONSE

D. Croccolo^a, M. De Agostinis^a, S. Fini^a, G. Olmi^{a, *}, L. Paiardini^a, F. Robusto^a

^a Department of industrial Engineering (DIN), University of Bologna, Viale del Risorgimento 2, 40136 Bologna, Italy

Abstract: Threaded joints are widely used in the automotive industry, where threadlockers can be applied to prevent self-loosening and to possibly achieve a friction drop upon tightening. This study deals with this topic, investigating threadlocker tribological response before polymerization takes place. These lubricating properties are usually not considered in adhesive datasheets, but can be highly beneficial from the point of view of the achievable preload, thus preventing self-loosening. The topic was tackled experimentally, running an extensive campaign that involved a medium and a strong threadlocker (respectively LOCTITE 243 and LOCTITE 270), used for tightening zincplated and black-oxidized screws. The testing procedure was inspired by a protocol being used by most manufacturers in the automotive field: in particular, the impacts of pretightening speed and of the time interval between pre-tightening and final tightening were investigated. The retrieved results indicate a lubricating effect of the medium threadlocker with achievable friction coefficients in the order of 0.11. Moreover, for both the threadlockers, a generally decreasing trend for the thread friction upon tightening for increasing time interval before tightening was observed. This is a particularly original outcome that could have remarkable implications for optimizing the assembly tasks of large sets of bolts.

Keywords: friction (Phenomena), anaerobic (Adhesive materials), automotive (Applications), Threadlocker, Lubrication, Screw Tightening

List of Symbols:

- *d* Nominal thread diameter [mm]
- *d*₂ Pitch diameter [mm]
- F_v Axial preload [N]
- q_H Coefficient for the HSD Test [-]
- q_{N-K} Coefficient for the NKD Test [-]
- *t* Coefficient for the LSD Test based on the Student's distribution [-]
- *T* Tightening torque [Nm]
- *T_b* Tightening torque lost in the underhead [Nm]
- T_{th} Tightening torque in the shank [Nm]
- μ_{th} Friction coefficient in the threads [-]

List of Acronyms:

- ANOVA Analysis of Variance
- ER Engagement Ratio
- F_{calc.} Fisher ratio
- HSD Honest significant difference
- LSD Fisher's Least Significant Difference
- MSQ Mean Squares (general term) for ANOVA
- MSE Mean Squares error per pairwise tests
- NKD Newman-Keuls Significant Difference
- p-v. p-value
- SSBR Sum of Squares Between Rows

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: <u>giorgio.olmi@unibo.it</u>

SSBC Sum of Squares Between Columns

SSQ Sum of Squares (general term) for ANOVA

1. Introduction

The use of adhesives has increased in the last years, as they proved to provide reliable design solutions for the development of mechanical joints. In fact, they make it possible to achieve on one hand, an acceptable strength with respect to specifications, on the other hand lightweight properties, thanks to their high strength over weight ratio. In addition, suitable adhesives ensure the fulfilment of some further features, such as corrosion or fretting resistance [1] features. With particular reference to anaerobic adhesives, i.e. to adhesives that polymerize in the absence of oxygen, several studies have been performed to assess their fatigue response [1], to investigate the effect of the pressure acting on the bonding on its static strength [2-3] and to derive some design tips regarding joint proportioning. In particular, regarding this point, an experimental study has been conducted, to assess the impact of the aspect ratio (coupling length over coupling diameter) on the shear strength of press fitted and adhesively bonded joints [4]. The response of an anaerobic adhesive, when applied under different interference or small clearance levels (no more than 0.05 mm) has been investigated as well. The effectiveness of anaerobic adhesives, when applied to joints with a limited clearance or a not high interference between the mating parts, for instance a shaft and a hub, has been also highlighted in [4]. In this and other References it is pointed out that the joint overall strength takes advantage of the capability of the adhesive of filling the voids among the roughness crests, thus incrementing the actual mating surface to the surface theoretically involved in the coupling. From this point of view, in hybrid joints, when adhesive is added, a remarkably high coupling strength can be achieved, even for not a high

interference level. This outcome makes it possible to reduce the costs needed to accomplish very strict tolerances and may facilitate the coupling (by press or shrink fitting) procedure. Further studies [3, 5] investigated the response of a strong commercial anaerobic adhesive by torsional tests on butt-bonded joints.

As it stems from the previous remarks, one of the main features of an anaerobic adhesive is that it can be used not only to replace a conventional coupling, but also to integrate it, thus improving its mechanical response. A typical example from this point of view is the use of an anaerobic adhesive as a threadlocker that safely locks a threaded fastener against self-loosening, which may be induced by on service loads or vibrations. In addition, they are able to provide an additional protection against moisture and corrosion by sealing the voids between the screw and the nut threads [6-7]. This type of adhesive, namely an anaerobic acrylic one (based on dimethacrylate resin), is able to cure at room temperature: it polymerizes in the absence of air in the pockets between the internal and external threads of the nut and of the screw respectively [8]. Cure is activated by the presence of metal and affected by its physical properties and by the oxygen concentration. From this point of view, the most widely used metals for threaded fasteners, i.e. steel and aluminium alloy (sometimes used for nuts, to achieve lightweight properties, see [9]) are active enough, to harden anaerobics rapidly. The properties of these adhesives are also affected by some additives that are mixed with the basic resin [10]. Their role mainly involve cure acceleration or viscosity increase. In addition, a further and worth noticing property, being yielded by Polytetrafluoroethylene (PTFE) additives, is lubricity. In other words, before polymerization takes place, this additive is able to yield a decrease of the friction coefficient in the threads. Several studies, such as [11], indicate this friction drop has a beneficial impact on the structural response of the threaded joints, as it makes it possible to achieve an incremented axial load for a fixed value of the controlled tightening torque.

A higher preload is usually preferable, as it increases the joint reliability against fretting and fatigue and prevents loosening.

Threadlockers are commonly classified as weak, medium and strong, based on their achievable strength upon polymerization, and also with regard to their effect from the tribological point of view. In particular, high strength threadlockers may lead to an increase of the thread friction coefficient upon tightening. Conversely, medium and weak threadlockers are thought to lead to a slight friction drop with respect to dry conditions, as they act as lubricants before cure takes place [12]. Other studies dealing with the beneficial properties of threadlockers are available in the technical literature and are often provided in the Internet, for instance [13-14]. However, they can be usually regarded as internal studies by suppliers, where few averaged results are reported without any statistical assessment. Moreover, properly regarding the tribological response, friction coefficients are not provided and the lubricity yielded by the added threadlocker can be only guessed by tightening torque decrease for fixed controlled bolt preload. On the other hand, a very low amount of studies on threadlockers are available in the scientific literature, and most of them deal with breakaway untightening torque increment as an effect of threadlocker application. For instance, the study in [15] is focused on the response, in terms of untightening torque, of a strong threadlocker (LOCTITE 270). The effect of thread geometry and applied coatings is investigated, but no tribological analyses are performed. Other studies [16], on a medium threadlocker (LOCTITE 243), have been focused on the effect on strength of pressure acting on the bonding interface. The aforementioned lubricating effect has been partially investigated in [17], where a quite extensive experimental campaign has been run, involving a medium threadlocker (LOCTITE 243), and assessing the effects of the thread nominal diameter, of its aspect ratio (engaged length over the nominal diameter of the threaded joint) and of the nut material. In particular, steel and Aluminium alloy nuts have been considered, thus investigating steel-to-steel and steel-to-Al alloy mates. Suitable testing rigs and instrumented sleeves have been utilized, to simultaneously measure both the tightening or the untightening torques, as well as the axial loads, thus retrieving the frictional coefficients (according to [18]), for fastening and release phases. The results indicate that, regardless of the nut material, the frictional coefficient is reduced (at an average, by 20%) with respect to the dry state, when a threadlocker is added before tightening. In other words, a beneficial lubrication occurs, which is likely to provide a further benefit beyond the well known breakaway torque increase.

The present study derives its motivations from the lack of studies regarding the lubricating effect of commercial threadlockers, as previous studies are generally focused on their locking properties, disregarding this additional beneficial and counterintuitive property. This research aims at tackling this topic, assessing the tribological properties of threadlockers upon tightening before significant polymerization takes place and an appreciable shear strength of the adhesive is achieved, i.e. before the properties described in every data sheet are finally fulfilled. Issues of novelty, with respect to the only study [17] that highlights this interesting feature arises from the involvement of both zinccoated and black-oxidized screws. Moreover, both a medium threadlocker (LOCTITE 243) and a strong one (LOCTITE 270) are included in the analysis, in order to compare their tribological responses. Regarding this point, dry state is also added as a further reference level, to assess friction evolution, as an effect of threadlocker application. In particular, to the best of the authors' knowledge, no results are available in the literature regarding the effect on friction upon tightening, following the application of a strong adhesive. As remarked above, the lubricating properties arising from PTFE particles in threadlockers are very well known in the technical literature, however quantitative results

dealing with friction are still missing from the scientific point of view. Finally, frictional coefficients are determined in the framework of a bolt tightening protocol, which is widely utilized by several manufacturers in the automotive field. From this point of view, the tribological assessment is addressed in actual industrial conditions. Therefore, manufacturers can take advantage of the retrieved outcomes, to optimize their process and to get a better awareness of the effects of possible deviations from the standard protocol. For this purpose, statistical tools have been widely applied, to process the results and to assess the significance of the observed friction variations.

2. Materials and methods

Standard hexagonal head screws class 8.8 M8 were used (according with ISO 898-1). Two different screw surface treatments were considered: zinc-coated and black-oxidized. The screws were tested by a Kistler ANALYSE system (Manufacturer: Kistler, Winterthur, Switzerland), whose picture is provided in Fig. 1, being able to tight the bolted joint up to 500 Nm with a rotation speed up to 300 rpm. The system is equipped with a torque cell, which makes it possible to measure the tightening torque and with an axial load and a further torque cell. These are able to record the thread torque and the axial preload during the screw fastening task. In addition, the testing device is provided with a steel test-bearing-plate (type HH), to be posed under the screw head, having a 0.5 µm average roughness, 9 mm diameter holes and being conformal to ISO 16047.

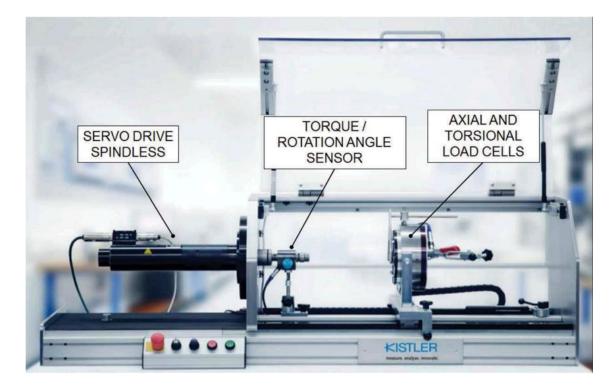


Fig. 1: Kistler ANALYSE system

The testing procedure was inspired to the Volkswagen Standard VW 01131-1:2012, a protocol that is commonly used in the automotive field. According to its recommendations, a minimum turning angle of 360° between the screw head and its counter face at the bearing must be ensured. The tightening of the screw consists of three phases: a first tightening up a torque of approximately 30% of the maximum tightening torque is initially carried out at a remarkably high tightening speed (200 rpm). Afterwards, the spindle is unloaded, in order to trigger the transition from the sliding to the static friction. Finally, the fastening of the threaded joint up to its maximum recommend torque is completed out at a rotational speed of 20 rpm. The test procedure is summarized in Fig.

2.

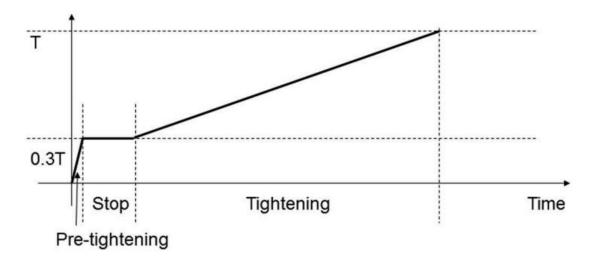


Fig. 2: Testing procedure

The present research involved different factors, while assessing the impact of different threadlockers on the frictional coefficient in the threads upon tightening. The study was framed within the aforementioned protocol and also assessed the tribological response, following some deviations from the described procedure, or the use of different screw types. The involved factors were the threadlocker type (strength), tightening speed with regard to the pre-tightening phase and the stop duration between pre-tightening and final tightening.

As for the threadlockers, LOCTITE 243 and LOCTITE 270 (Manufacturer: Henkel, Düsseldorf, Germany), respectively medium and strong anaerobic adhesives, were selected for testing, due to their wide application, especially in the automotive field. In addition, as received screws, i.e. without added threadlocker (dry conditions), were involved in the campaign for comparison purposes. With regard to the pre-tightening speed, three levels were considered (100 rpm, 200 rpm and 300 rpm), thus including the recommended speed according to the Volkswagen Standard. Finally, with reference to the stop between pre-tightening and final tightening, five levels were considered for its

span (0.2 s, 2 s, 20 s, 200 s and 600 s). These can be regarded as a 10-base logarithmic regression, with an additional higher term, corresponding to 10 min. As above, these durations were selected, so that they included the recommended time interval and were also consistent with the actual durations in industrial fastening procedures, which usually take longer times. In fact, the elapsed time between the pre-tightening and the tightening phases may be dramatically increased, when fastening sets consisting of several screws. The maximum threshold of 10 min arises from the occurrence that, after this elapsed time, both the adhesives start to polymerize according to their datasheets. Therefore, the stop duration should never exceed this time interval.

Forty-five treatment combinations were considered as a total, including 5 stop duration levels, 3 different tightening speeds upon pre-tightening and two different threadlockers (LOCTITE 243 and 270) plus dry state. Moreover, 5 replications were considered (per treatment combination) for statistical evidence reasons, according to [9 18-19]. The experimental design is provided in Table 1: it corresponds to a total of 225 performed experiments. As stated above, the experimental campaign involved both zinc-plated and black-oxidized screws. Therefore, the same plan was repeated for both: in other words, it was doubled, which increased the overall number of tests to 450. All the tests were run at the controlled temperature of 23°C.

| Dury | | Pre-tightening speed [rpm] | | | | | | |
|-------------------|-----|----------------------------|-------------|-------------|--|--|--|--|
| Dry | | 100 | 200 | 300 | | | | |
| | 0.2 | Series 1.1 | Series 1.2 | Series 1.3 | | | | |
| | 2 | Series 1.4 | Series 1.5 | Series 1.6 | | | | |
| Stop duration [s] | 20 | Series 1.7 | Series 1.8 | Series 1.9 | | | | |
| | 200 | Series 1.10 | Series 1.11 | Series 1.12 | | | | |
| | 600 | Series 1.13 | Series 1.14 | Series 1.15 | | | | |
| LOCTITE | 042 | Pre-tightening speed [rpm] | | | | | | |
| LOCITE | 243 | 100 | 200 | 300 | | | | |
| Stop duration [a] | 0.2 | Series 2.1 | Series 2.2 | Series 2.3 | | | | |
| Stop duration [s] | 2 | Series 2.4 | Series 2.5 | Series 2.6 | | | | |

Table 1: Experimental plan

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: <u>giorgio.olmi@unibo.it</u>

| | 20 | Series 2.7 | Series 2.8 | Series 2.9 |
|-------------------|-----|-------------|-------------------|-------------|
| | 200 | Series 2.10 | Series 2.11 | Series 2.12 |
| | 600 | Series 2.13 | Series 2.14 | Series 2.15 |
| LOCTITE | 270 | Pre-t | ightening speed [| rpm] |
| LOCITE | 270 | 100 | 200 | 300 |
| | 0.2 | Series 3.1 | Series 3.2 | Series 3.3 |
| | 2 | Series 3.4 | Series 3.5 | Series 3.6 |
| Stop duration [s] | 20 | Series 3.7 | Series 3.8 | Series 3.9 |
| | 200 | Series 3.10 | Series 3.11 | Series 3.12 |
| | 600 | Series 3.13 | Series 3.14 | Series 3.15 |

The testing rig makes it possible to on-line compute the total, the bearing and the thread friction coefficients at the end of every phase. In this study, the effects of the aforementioned factors on the thread friction coefficients after pre-tightening and final tightening were considered. It appeared to be reasonable to specifically consider the friction coefficient in the threads (instead, for instance, of total friction) because the addition of threadlocker directly affects the thread response.

The tightening torque was chosen according to the Standard ISO 898-1 [20]. This Standard provides the proof loads for each pitch diameter and each screw class. In the case study the proof load recommended by the Standard is F_V =21,200N. The tightening torque *T* was evaluated as follows by the simplified formula in Eq. (1) [21].

$$T = 0.2 \cdot F_{V} \cdot d = 33.92Nm \tag{1}$$

Where d is the nominal diameter.

To perform the tests, according to the Volkswagen Standard, a first rotation of -360° (backwards) was applied by the spindle. Afterwards, the screw was tightened up to 30% of the tightening torque *T*, controlling the spindle speed. Finally, after the stop for the treatment combination dependent duration, the screw was tightened up to the final torque *T* at the speed of 20 rpm. The tightening torque, the thread torque and the axial load were measured during the test. The friction coefficient in the threads was automatically

determined by the testXpert® software, according to the Standard ISO 16047 [18], following recording of the thread torque T_{th} . The Motosh's formula as indicated by Eq. (2), was applied for this purpose:

$$\mu_{th} = \frac{\frac{T_{th}}{F_V} - \frac{p}{2\pi}}{0.577 \cdot d_2} \tag{2}$$

Where p is the pitch and d_2 is the pitch diameter.

During the experimental tests, the screw was combined with a washer at its underhead before applying the threadlocker. It was applied on the screw threads, starting from the bottom side of the screw (Fig. 3). Afterwards, the screw was inserted in the test fixture and the previously described procedure for tightening was started. As an effect of rotation, while engaging the screw into the nut, the adhesive got spread on the entire screw thread, thus remaining trapped between the internal and the external threads with tolerances 6H/6g. The nuts, the screws and the washers were changed at each test.

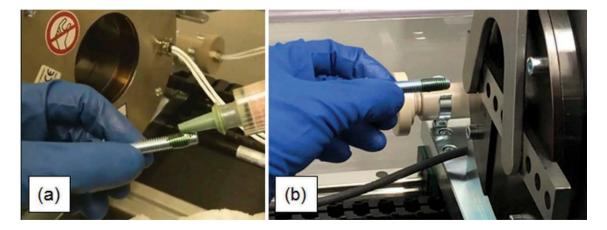


Fig. 3: (a) Threadlocker application and (b) screw mounting

3. Results

The results in terms of the friction coefficients in the threads upon pre-tightening and final tightening, when testing zinc-plated screws, are collected in Tables 2-4 respectively. The same results for black-oxidized screws are reported in Tables 5-7. Considering grand mean values, pre-tightening μ_{th} were 0.16 (with 0.013 standard deviation) and 0.17 (0.021 standard deviation) for zinc-coated and black-oxidized screws respectively. The same values upon tightening, again for the same screws, were respectively 0.14 (0.025 standard deviation) and 0.12 (0.013 standard deviation).

In the next section, the friction coefficient in the threads is going to be referenced as just "friction coefficient" for the sake of synthesis.

| | DRY | | | | | | | | | | | |
|-------------------|-------------------------------------|-----------|---------------------|-------|-------------------|---------|---------|---------------------|-------|--|--|--|
| | AFTER | R PRE-TIC | GHTENIN | NG | | AFTER | FINAL T | IGHTEN | NG | | | |
| | μ_{th} | Pre-ti | ghtening : [rpm] | speed | μ_{th} | | Pre-ti | ghtening : [rpm] | speed | | | |
| | <i>p</i> -tit | 100 | 200 | 300 | | Pan | 100 | 200 | 300 | | | |
| | | 0.178 | 0.149 | 0.137 | | | 0.204 | 0.131 | 0.122 | | | |
| | | 0.180 | 0.148 | 0.160 | | | 0.161 | 0.137 | 0.168 | | | |
| | C 0.170 0.139 0.164 C | 0.158 | 0.145 | 0.168 | | | | | | | | |
| | | 0.158 | 0.159 | 0.160 | Stop duration [s] | | 0.134 | 0.171 | 0.159 | | | |
| | | 0.162 | 0.146 | 0.144 | | | 0.146 | 0.147 | 0.132 | | | |
| \mathbf{s} | | 0.150 | 0.143 | 0.191 | | 6 | 0.126 | 0.120 | 0.182 | | | |
| on | | 0.172 | 0.186 | 0.160 | | | 0.180 | 0.194 | 0.141 | | | |
| ati | 7 | 0.151 | 0.158 | 0.152 | | | 0.135 | 0.116 | 0.123 | | | |
| Inp | | 0.177 | 0.153 | 0.156 | Inp | | 0.152 | 0.121 | 0.145 | | | |
| Stop duration [s] | | 0.160 | 0.169 | 0.168 | do | | 0.122 | 0.123 | 0.147 | | | |
| S | | 0.152 | 0.161 | 0.169 | S | | 0.117 | 0.145 | 0.135 | | | |
| | | 0.169 | 0.155 | 0.160 | | | 0.128 | 0.123 | 0.110 | | | |
| | 50 | 0.149 | 0.159 | 0.172 | | 20 | 0.114 | 0.117 | 0.159 | | | |
| | | 0.161 | 0.165 | 0.156 | | | 0.132 | 0.125 | 0.106 | | | |
| | | 0.203 | 0.173 | 0.152 | | | 0.179 | 0.133 | 0.119 | | | |
| | 20 0 | 0.153 | 0.155 | 0.154 | | 20 0 | 0.107 | 0.110 | 0.111 | | | |

Table 2: Experimental results in terms of friction coefficient in the thread after pretightening and after final tightening for zinc-coated screws in dry conditions

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: giorgio.olmi@unibo.it

| | 0.159 | 0.167 | 0.153 | | 0.124 | 0.154 | 0.161 |
|-----|-------|-------|-------|-----|-------|-------|-------|
| | 0.180 | 0.181 | 0.162 | | 0.200 | 0.165 | 0.149 |
| | 0.154 | 0.141 | 0.144 | | 0.104 | 0.160 | 0.099 |
| | 0.154 | 0.170 | 0.175 | | 0.128 | 0.180 | 0.168 |
| | 0.163 | 0.146 | 0.158 | | 0.104 | 0.121 | 0.105 |
| | 0.169 | 0.171 | 0.164 | _ | 0.140 | 0.169 | 0.141 |
| 600 | 0.157 | 0.159 | 0.189 | 600 | 0.106 | 0.133 | 0.110 |
| • | 0.170 | 0.155 | 0.161 | • | 0.132 | 0.175 | 0.142 |
| | 0.178 | 0.187 | 0.178 | | 0.133 | 0.142 | 0.104 |

Table 3: Experimental results in terms of friction coefficient in the thread after pretightening and after final tightening for zinc-coated screws treated by LOCTITE 243

| | LOCTITE 243 | | | | | | | | | | | | |
|-------------------|----------------------------|-----------|---------------------|-------|------------------------|------------|---------|---------------------|-------|--|--|--|--|
| | AFTER | R PRE-TIC | GHTENIN | ١G | AFTER FINAL TIGHTENING | | | | | | | | |
| | μ_{th} | Pre-tiş | ghtening s [rpm] | speed | | μ_{th} | Pre-tiş | ghtening s [rpm] | speed | | | | |
| | - | 100 | 200 | 300 | | - | 100 | 200 | 300 | | | | |
| | | 0.190 | 0.187 | 0.176 | | | 0.145 | 0.143 | 0.131 | | | | |
| | | 0.190 | 0.216 | 0.174 | | | 0.153 | 0.159 | 0.127 | | | | |
| | C 0.156 0.159 0.182 | 0.2 | 0.129 | 0.125 | 0.156 | | | | | | | | |
| | | 0.204 | 0.206 | 0.208 | | | 0.157 | 0.152 | 0.145 | | | | |
| | 0.213 0.188 0.183 | 0.142 | 0.150 | 0.158 | | | | | | | | | |
| | | 0.180 | 0.202 | 0.179 | | 5 | 0.159 | 0.147 | 0.126 | | | | |
| | | 0.180 | 0.140 | 0.228 | Stop duration [s] | | 0.136 | 0.107 | 0.181 | | | | |
| | 7 | 0.183 | 0.192 | 0.190 | | | 0.135 | 0.147 | 0.117 | | | | |
| S | | 0.216 | 0.174 | 0.154 | | | 0.159 | 0.122 | 0.115 | | | | |
| on | | 0.177 | 0.190 | 0.202 | | | 0.129 | 0.130 | 0.148 | | | | |
| Stop duration [s] | | 0.197 | 0.230 | 0.198 | ati | | 0.125 | 0.164 | 0.151 | | | | |
| Inp | | 0.205 | 0.179 | 0.194 | Inp | | 0.134 | 0.141 | 0.150 | | | | |
| do | 20 | 0.221 | 0.192 | 0.146 | do | 20 | 0.160 | 0.146 | 0.131 | | | | |
| St | | 0.187 | 0.210 | 0.158 | St | | 0.148 | 0.169 | 0.119 | | | | |
| | | 0.189 | 0.198 | 0.221 | | | 0.137 | 0.155 | 0.170 | | | | |
| | | 0.196 | 0.153 | 0.250 | | | 0.158 | 0.108 | 0.241 | | | | |
| | | 0.175 | 0.168 | 0.210 | | | 0.127 | 0.124 | 0.176 | | | | |
| | 200 | 0.185 | 0.161 | 0.206 | | 200 | 0.145 | 0.115 | 0.194 | | | | |
| | | 0.193 | 0.154 | 0.205 | | | 0.155 | 0.114 | 0.140 | | | | |
| | | 0.201 | 0.135 | 0.158 | | | 0.205 | 0.098 | 0.129 | | | | |
| | 600 | 0.184 | 0.149 | 0.155 | | 600 | 0.170 | 0.126 | 0.119 | | | | |
| | 9(| 0.189 | 0.118 | 0.127 | | 9(| 0.130 | 0.121 | 0.109 | | | | |

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: <u>giorgio.olmi@unibo.it</u>

| 0.133 | 0.120 | 0.094 | | 0.123 | 0.094 | 0.085 |
|-------|-------|-------|--|-------|-------|-------|
| 0.046 | 0.125 | 0.080 | | 0.075 | 0.094 | 0.084 |
| 0.081 | 0.114 | 0.052 | | 0.087 | 0.102 | 0.074 |

| Table 4: Experimental results in terms of friction coefficient in the thread after pre- | |
|---|--|
| tightening and after final tightening for zinc-coated screws treated by LOCTITE 270 | |

| ugn | tening an | | | LOCTI | | | s treated by | LOCIII | L 270 |
|-------------------|----------------------|-------------------|--------|-------|-------------------|------------|--------------|---------------------|-------|
| | AFTER | Re-TIG | HTENIN | ١G | | AFTER | FINAL TI | GHTENI | NG |
| | Pre-tightening speed | | | speed | | μ_{th} | Pre-tig | ghtening s [rpm] | peed |
| | <i>p</i> | 100 | 200 | 300 | | <i>[</i> | 100 | 200 | 300 |
| | | 0.248 | 0.220 | 0.203 | | | 0.239 | 0.219 | 0.213 |
| | | 0.229 | 0.220 | 0.226 | | | 0.229 | 0.214 | 0.212 |
| | 0.2 | 0.189 | 0.269 | 0.168 | | 0.2 | 0.201 | 0.271 | 0.211 |
| | | 0.222 0.249 0.204 | 0.219 | 0.242 | 0.220 | | | | |
| | 0.257 0.248 0.246 | 0.237 | 0.235 | 0.250 | | | | | |
| | | 0.221 | 0.227 | 0.215 | | | 0.243 | 0.243 | 0.231 |
| | | 0.275 | 0.201 | 0.191 | | 7 | 0.252 | 0.204 | 0.191 |
| | 5 | 0.190 | 0.210 | 0.166 | | | 0.211 | 0.204 | 0.187 |
| | | 0.228 | 0.211 | 0.231 | | | 0.246 | 0.218 | 0.245 |
| _ | | 0.208 | 0.219 | 0.224 | _ | | 0.180 | 0.222 | 0.224 |
| S. | | 0.234 | 0.214 | 0.248 | Stop duration [s] | | 0.217 | 0.228 | 0.246 |
| tior | | 0.215 | 0.179 | 0.208 | | | 0.212 | 0.194 | 0.209 |
| ura | 20 | 0.214 | 0.210 | 0.221 | | 20 | 0.219 | 0.213 | 0.244 |
| ıp d | | 0.235 | 0.217 | 0.205 | | | 0.236 | 0.216 | 0.196 |
| Stop duration [s] | | 0.207 | 0.227 | 0.217 | Stol | | 0.203 | 0.208 | 0.212 |
| •1 | | 0.172 | 0.190 | 0.186 | •1 | | 0.201 | 0.202 | 0.215 |
| | | 0.173 | 0.214 | 0.196 | | | 0.180 | 0.243 | 0.222 |
| | 200 | 0.198 | 0.192 | 0.180 | | 200 | 0.226 | 0.195 | 0.194 |
| | | 0.198 | 0.193 | 0.189 | | | 0.201 | 0.210 | 0.190 |
| | | 0.196 | 0.209 | 0.183 | | | 0.203 | 0.228 | 0.192 |
| | | 0.125 | 0.129 | 0.160 | | | 0.157 | 0.176 | 0.199 |
| | | 0.165 | 0.125 | 0.183 | | | 0.184 | 0.174 | 0.202 |
| | 600 | 0.128 | 0.149 | 0.158 | | 600 | 0.144 | 0.187 | 0.192 |
| | _ | 0.183 | 0.182 | 0.177 | | _ | 0.199 | 0.206 | 0.194 |
| | | 0.142 | 0.153 | 0.194 | | | 0.182 | 0.173 | 0.221 |

Table 5: Experimental results in terms of friction coefficient in the thread after pre-tightening and after final tightening for black-oxidized screws in dry conditions

| | | | | DF | RY | | | | | | |
|-------------------|------------|---------|---------------------|--------|------------------------|--------------|---------|---------------------|-------|--|--|
| | AFTER | PRE-TIC | HTENIN | ١G | AFTER FINAL TIGHTENING | | | | | | |
| | μ_{th} | Pre-tig | ghtening s [rpm] | speed | | μ_{th} | Pre-tig | ghtening s [rpm] | speed | | |
| | <i>p</i> | 100 | 200 | 00 300 | | <i>μ</i> υιν | 100 | 200 | 300 | | |
| | | 0.175 | 0.153 | 0.179 | | | 0.110 | 0.100 | 0.117 | | |
| | | 0.174 | 0.159 | 0.173 | | | 0.112 | 0.119 | 0.110 | | |
| | 0.2 | 0.121 | 0.188 | 0.155 | | 0.2 | 0.104 | 0.118 | 0.089 | | |
| | | 0.176 | 0.173 | 0.121 | | | 0.121 | 0.122 | 0.095 | | |
| | | 0.187 | 0.182 | 0.201 | | | 0.124 | 0.130 | 0.153 | | |
| | | 0.183 | 0.165 | 0.170 | | | 0.112 | 0.113 | 0.116 | | |
| | | 0.134 | 0.138 | 0.174 | | | 0.116 | 0.131 | 0.121 | | |
| | 7 | 0.179 | 0.175 | 0.117 | Stop duration [s] | 7 | 0.108 | 0.122 | 0.099 | | |
| | | 0.200 | 0.165 | 0.141 | | | 0.116 | 0.147 | 0.114 | | |
| | | 0.172 | 0.192 | 0.189 | | | 0.129 | 0.107 | 0.124 | | |
| Stop duration [s] | | 0.195 | 0.181 | 0.163 | | | 0.132 | 0.099 | 0.124 | | |
| tion | | 0.174 | 0.158 | 0.156 | | | 0.107 | 0.116 | 0.118 | | |
| ura | 20 | 0.162 | 0.168 | 0.153 | | 20 | 0.103 | 0.099 | 0.136 | | |
| p d | | 0.157 | 0.154 | 0.180 | | | 0.101 | 0.118 | 0.117 | | |
| Sto | | 0.144 | 0.205 | 0.185 | Sto | | 0.116 | 0.097 | 0.093 | | |
| | | 0.120 | 0.130 | 0.204 | | | 0.098 | 0.100 | 0.103 | | |
| | | 0.142 | 0.172 | 0.165 | | | 0.120 | 0.116 | 0.117 | | |
| | 200 | 0.174 | 0.151 | 0.173 | | 200 | 0.129 | 0.136 | 0.122 | | |
| | | 0.193 | 0.159 | 0.142 | | | 0.106 | 0.112 | 0.098 | | |
| | | 0.190 | 0.162 | 0.165 | | | 0.107 | 0.124 | 0.104 | | |
| | | 0.174 | 0.142 | 0.187 | | | 0.120 | 0.122 | 0.125 | | |
| | | 0.161 | 0.170 | 0.173 | | | 0.122 | 0.131 | 0.115 | | |
| | 600 | 0.198 | 0.179 | 0.168 | | 600 | 0.129 | 0.125 | 0.110 | | |
| | 9 | 0.167 | 0.152 | 0.166 | | | 0.089 | 0.135 | 0.106 | | |
| | | 0.169 | 0.201 | 0.199 | | | 0.122 | 0.130 | 0.110 | | |

| | LOCTITE 243 | | | | | | | | | | | |
|-------------------|-------------|-------------------|---------------------|---------|------------------------|------------|---------|---------------------|-------|--|--|--|
| | AFTER | R PRE-TIC | GHTENIN | ١G | AFTER FINAL TIGHTENING | | | | | | | |
| | μ_{th} | Pre-tiş | ghtening s [rpm] | speed | | μ_{th} | Pre-tig | ghtening s [rpm] | peed | | | |
| | pan | 100 | 200 | 200 300 | | pun | 100 | 200 | 300 | | | |
| | | 0.261 | 0.171 | 0.201 | | | 0.191 | 0.172 | 0.180 | | | |
| | | 0.165 | 0.169 | 0.220 | | | 0.175 | 0.166 | 0.191 | | | |
| | 0.2 | 0.162 | 0.195 | 0.170 | | 0.2 | 0.181 | 0.174 | 0.168 | | | |
| | | 0.165 | 0.181 | 0.205 | | | 0.197 | 0.177 | 0.183 | | | |
| | | 0.139 | 0.182 | 0.201 | | | 0.172 | 0.171 | 0.199 | | | |
| | | 0.191 | 0.181 | 0.213 | | | 0.169 | 0.203 | 0.205 | | | |
| | | 0.270 0.242 0.198 | 0.234 | 0.219 | 0.174 | | | | | | | |
| | 7 | 0.250 | 0.161 | 0.189 | | 7 | 0.197 | 0.167 | 0.191 | | | |
| | | 0.220 | 0.219 | 0.215 | | | 0.191 | 0.210 | 0.202 | | | |
| | | 0.261 | 0.227 | 0.197 | | | 0.215 | 0.202 | 0.192 | | | |
| ı [s] | | 0.200 | 0.208 | 0.144 | Stop duration [s] | | 0.157 | 0.158 | 0.115 | | | |
| tio | | 0.222 | 0.207 | 0.233 | | 20 | 0.175 | 0.159 | 0.157 | | | |
| ura | 20 | 0.199 | 0.230 | 0.246 | | | 0.155 | 0.172 | 0.172 | | | |
| p d | | 0.205 | 0.214 | 0.212 | p d | | 0.154 | 0.171 | 0.160 | | | |
| Stop duration [s] | | 0.185 | 0.185 | 0.242 | Sto | | 0.146 | 0.151 | 0.160 | | | |
| | | 0.192 | 0.175 | 0.134 | | | 0.151 | 0.122 | 0.114 | | | |
| | | 0.215 | 0.168 | 0.144 | | | 0.151 | 0.111 | 0.126 | | | |
| | 200 | 0.214 | 0.214 | 0.204 | | 200 | 0.158 | 0.134 | 0.150 | | | |
| | | 0.221 | 0.192 | 0.192 | | | 0.155 | 0.135 | 0.143 | | | |
| | | 0.201 | 0.181 | 0.132 | | | 0.144 | 0.134 | 0.113 | | | |
| | | 0.174 | 0.159 | 0.138 | | | 0.124 | 0.106 | 0.097 | | | |
| | 600 | 0.154 | 0.161 | 0.272 | | | 0.115 | 0.114 | 0.165 | | | |
| | | 0.160 | 0.191 | 0.136 | | 600 | 0.102 | 0.111 | 0.095 | | | |
| | | 0.227 | 0.188 | 0.122 | | | 0.133 | 0.114 | 0.107 | | | |
| | | 0.171 | 0.168 | 0.163 | | | 0.104 | 0.124 | 0.110 | | | |

Table 6: Experimental results in terms of friction coefficient in the thread after pretightening and after final tightening for black-oxidized screws treated by LOCTITE 243

Table 7: Experimental results in terms of friction coefficient in the thread after pretightening and after final tightening for black-oxidized screws treated by LOCTITE 270

| LOCTITE 270 | | | | | | | |
|-------------|-------------------------------|------------------------|-------------------------------|--|--|--|--|
| AFTER | PRE-TIGHTENING | AFTER FINAL TIGHTENING | | | | | |
| μ_{th} | Pre-tightening speed [rpm] | μ_{th} | Pre-tightening speed [rpm] | | | | |

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: <u>giorgio.olmi@unibo.it</u>

| | | 100 | 200 | 300 | | | 100 | 200 | 300 |
|-------------------|-----|-------|-------|-------|-------------------|-----|-------|-------|-------|
| | | 0.229 | 0.174 | 0.228 | | | 0.197 | 0.180 | 0.204 |
| | | 0.242 | 0.175 | 0.201 | | | 0.218 | 0.174 | 0.192 |
| | 0.2 | 0.196 | 0.185 | 0.126 | | 0.2 | 0.181 | 0.181 | 0.148 |
| | | 0.217 | 0.178 | 0.185 | | | 0.216 | 0.193 | 0.196 |
| | | 0.267 | 0.195 | 0.214 | | | 0.236 | 0.199 | 0.196 |
| | | 0.202 | 0.219 | 0.157 | | | 0.190 | 0.172 | 0.150 |
| | | 0.199 | 0.138 | 0.171 | | | 0.179 | 0.153 | 0.157 |
| | 7 | 0.186 | 0.179 | 0.125 | | 7 | 0.173 | 0.185 | 0.149 |
| | | 0.147 | 0.217 | 0.174 | | | 0.155 | 0.195 | 0.190 |
| | | 0.184 | 0.177 | 0.154 | | | 0.161 | 0.173 | 0.175 |
| Stop duration [s] | | 0.175 | 0.190 | 0.213 | נ <u>ו</u> צ] ו | | 0.161 | 0.166 | 0.192 |
| tion | | 0.158 | 0.218 | 0.182 | Stop duration [s] | | 0.148 | 0.193 | 0.163 |
| ura | 20 | 0.219 | 0.201 | 0.163 | | 20 | 0.185 | 0.187 | 0.152 |
| p d | | 0.214 | 0.224 | 0.152 | | | 0.168 | 0.200 | 0.165 |
| Sto] | | 0.261 | 0.189 | 0.165 | | | 0.216 | 0.157 | 0.175 |
| | | 0.097 | 0.152 | 0.099 | | | 0.126 | 0.150 | 0.123 |
| | | 0.198 | 0.155 | 0.162 | | | 0.210 | 0.169 | 0.161 |
| | 200 | 0.134 | 0.201 | 0.198 | | 200 | 0.155 | 0.199 | 0.169 |
| | | 0.242 | 0.159 | 0.179 | | | 0.214 | 0.151 | 0.165 |
| | | 0.166 | 0.208 | 0.225 | | | 0.158 | 0.194 | 0.198 |
| | | 0.177 | 0.196 | 0.171 | | | 0.150 | 0.176 | 0.167 |
| | 600 | 0.211 | 0.238 | 0.121 | | | 0.189 | 0.205 | 0.148 |
| | | 0.153 | 0.200 | 0.149 | | 600 | 0.159 | 0.160 | 0.152 |
| | | 0.176 | 0.134 | 0.209 | | - | 0.167 | 0.151 | 0.157 |
| | | 0.146 | 0.173 | 0.123 | | | 0.155 | 0.164 | 0.156 |

4. Discussion

The experimental campaign involved both zinc-plated and black-oxidized screws: these two types were selected, based on their wide applications in dry conditions and also on their suitability to threadlocker application [17]. The effects of zinc coatings on friction and wear were investigated in [22] by trials on tribological testing rigs, however very few studies, apart from [19], are available regarding screws. Black oxidization, based on a chemical process developed at the beginning of 1900, is well known for yielding esthetic properties, lubricity, corrosion and galling resistance [23].

The observed frictional coefficients in the as received conditions can be related to the outcomes of a closely related study [19]. The friction coefficients for zinc-coated screws, both for pre-tightening and upon tightening (0.16-0.14), are well consistent with the corresponding ranges reported in [19]. As for the black-oxidized ones, the coefficient after pre-tightening (0.17) is well consistent with the yields in the same reference, whereas, it remarkably drops down (to 0.12) upon final tightening. This value is a bit lower than that at the bottom of the range in [19].

The retrieved values in dry conditions were processed by the tools of ANOVA and Fisher-Test [24-25], to assess the significance of the pre-tightening speed. All the outcomes of the performed analyses (here omitted for the sake of synthesis) indicate that this factor is not significant at the 5% significance level.

Then, the study was moved to the retrieved data following the applications of the two threadlockers. The first task consisted in the preliminary assessment of the potential impact of the pre-tightening speed on the tribological response. For this purpose, it seemed to be reasonable to account for the frictional coefficients after pre-tightening. In fact, these data appeared to be potentially the most affected by this factor. Average values of the retrieved friction coefficients for the two threadlockers and the two screw types are plotted in the bar graphs in Fig. 4. Their differences (for each of the four cases) appear to be not significant, as also confirmed by the statistical tests at high confidence levels.

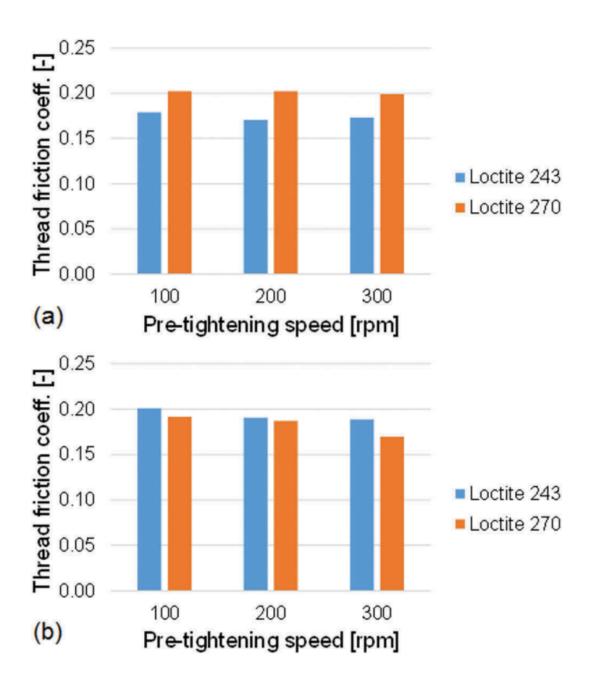


Fig. 4: Thread friction coefficient upon pre-tightening for different values of the pretightening speed measured after the application of the two threadlockers for (a) zincplated and (b) black-oxidized screws

Following this outcome, consistent with that for the as received screws, the analysis was moved to the assessment of the further operative factor, i.e. the stop duration between pre-tightening and tightening. This appeared to be the most interesting, as it may considerably vary, when large sets of screws have to be tightened. Moreover, when using an anaerobic adhesive, a friction increase with time may be intuitively expected, accounting for an initial polymerization, triggered by the reduced clearance in the pockets between the internal and the external threads. On the other hand, a possible lubricating effect, depending on the screw type and on the general threadlocker properties highlighted in the technical literature, deserved a deep investigation [17].

In order to suitably assess the impact of this factor on the threaded fastener, the friction coefficient upon final tightening was considered. In fact, this is the friction value that actually affects the induced preload for selected and controlled tightening torque [11, 18]. The responses under the application of the two threadlockers were initially considered separately for both the screw types.

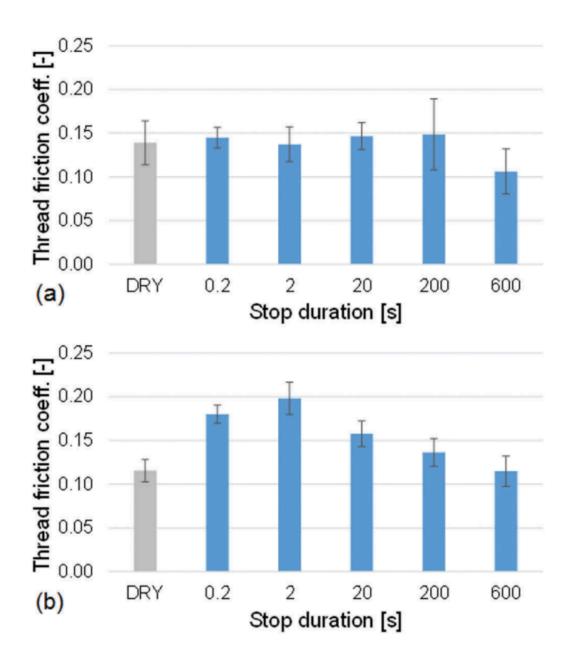


Fig. 5: Thread friction coefficient upon final tightening, following the application of LOCTITE 243 for (a) zinc-plated and (b) black-oxidized screws

The response under the medium threadlocker LOCTITE 243 was considered first: the results for different stop durations are collected in the bar graph in Fig. 5 for zinc-coated and black-oxidized screws. Variation intervals, accounting for one standard deviation, are appended as well, to take result scattering into account. The results were processed by the

tool of two-factor ANOVA, considering the two factors of pre-tightening speed and stop duration, and repeating the analysis for LOCTITE 243 applied to zinc-plated and black oxidized screws. Pre-tightening speed already proved to be not significant, but was considered as well, mainly to assess the presence of interaction between these two factors, as recommended in [24-25]. The results of the first analysis, for zinc-coated screws, is provided in Table 8, where the symbols and the acronyms retain the meanings described in the Nomenclature and in the aforementioned references. The stop duration is regarded as the row-factor and the pre-tightening speed as the column-factor.

Table 8: Analysis of variance on the effects of pre-tightening speed and of stop duration on the thread friction coefficient, following the application of LOCTITE 243 for zinc-

| | SSQ | DoF | MSQ | F _{calc.} | p-v |
|---------------------------------------|--------|-----|--------|--------------------|--------------------|
| SSBR (effect of stop duration) | 0.0186 | 4 | 0.005 | 9.39 | 5.10-6 |
| SSBC (effect of pre-tightening speed) | 0.0017 | 2 | 8.10-4 | 1.67 | 0.2 |
| Interaction | 0.0117 | 8 | 0.001 | 2.95 | 7·10 ⁻³ |
| Error | 0.0296 | 60 | 5.10-4 | | |
| Total | 0.0615 | 74 | | | |

plated screws

The outcome of the Fisher Test indicates the stop duration is highly significant with a p-value in the order of 10⁻⁶. Regarding black-oxidized screws, a similar outcome was retrieved with a very low p-value, whose order of magnitude is 10⁻²². These results indicate that the friction variations in Fig. 5 are all significant. Pre-tightening speed keeps not significant, whereas a moderate interaction was sometimes observed.

The analysis was deepened, following two strategies: first, the retrieved values were compared to those corresponding to the as received conditions: these were provided in Section 3 and are included in the same graph for the sake of readability. For zinc-coated screws, friction is initially unchanged, following the application of LOCTITE 243. Conversely, for black-oxidized screws, it is incremented, but this noticeable increment, from approximately 0.12 to 0.18, is mainly due to the very low value retrieved for these fasteners in dry conditions. Moreover, the incremented value is very close to the retrieved value upon pre-tightening. Secondly, the trend for increasing stop duration, which is also plotted in Fig. 6 for both screws types, considering a logarithmic scale for time, was assessed by pairwise tests, to allocate the differences. These tools made it possible to determine if the general decreasing trend for increasing stop duration was significant, thus indicating a lubricating effect of the adhesive. Regarding this point, it is worth mentioning that, according to the datasheets of both the threadlockers, polymerization needs longer stop duration to start with noticeable effects in terms of shear strength.

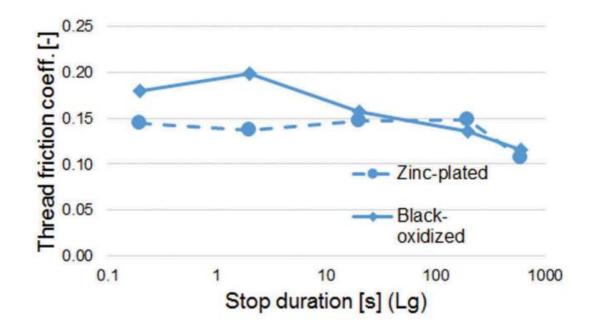


Fig. 6: Thread friction trend vs. stop duration, following LOCTITE 243 application for

the two screw types

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: giorgio.olmi@unibo.it In particular, three test types were applied: the Least Significant Difference, the Tukey's Honestly Significant Difference Test (HSD) and the Newman-Keuls Tests (NKD). The first one compares the difference of two row means (considering the results in Tables 3, 6) to a fixed threshold (LSD). It depends on an experimental uncertainty-related term (MSE), on a value *t* depending on the Student's distribution, and on the number of results (N) for the same stop duration, i.e. for the same level of the row factor. The second one also compares the difference of row means to a fixed threshold (HSD), where the t term is replaced by a different parameter (q_T), depending on the number of items to be compared, i.e., to the number of stop durations to be compared. This is a highly conservative test, meaning that, a pass outcome indicates that differences are highly significant. Finally, the third test adopts a variable threshold, based on the variable parameter (q_{N-K}), which makes this test more conservative than the first and less conservative than the second one. The three thresholds are provided in Eqs. (3)-(5). A 5% significance level was used for all these tests [24-25].

$$LSD = t \cdot \sqrt{\frac{2MSE}{N}}$$
(3)

$$HSD = q_H \cdot \sqrt{\frac{MSE}{N}} \tag{4}$$

$$NKD = q_{N-K} \cdot \sqrt{\frac{MSE}{N}}$$
(5)

Table 9: Pairwise tests applied to the data regarding the thread friction coefficient for

| | Differences | LSD | Sign.? | HSD | Sign.? | NKD | Sign.? |
|------------------|-------------|-------|--------|-------|--------|-------|--------|
| t=0.2s vs t=2s | 0.008 | 0.018 | No | 0.025 | No | 0.018 | No |
| t=0.2s vs t=20s | 0.002 | 0.018 | No | 0.025 | No | 0.018 | No |
| t=0.2s vs t=200s | 0.004 | 0.018 | No | 0.025 | No | 0.022 | No |
| t=0.2s vs t=600s | 0.039 | 0.018 | Yes | 0.025 | Yes | 0.022 | Yes |

LOCTITE 243 application, zinc-plated screws

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: giorgio.olmi@unibo.it

| t=2s vs t=20s | 0.009 | 0.018 | No | 0.025 | No | 0.022 | No |
|------------------|-------|-------|-----|-------|-----|-------|-----|
| t=2s vs t=200s | 0.011 | 0.018 | No | 0.025 | No | 0.024 | No |
| t=2s vs t=600s | 0.040 | 0.018 | Yes | 0.025 | Yes | 0.018 | Yes |
| t=20s vs t=200s | 0.002 | 0.018 | No | 0.025 | No | 0.018 | No |
| t=20s vs t=600s | 0.040 | 0.018 | Yes | 0.025 | Yes | 0.024 | Yes |
| t=200s vs t=600s | 0.042 | 0.018 | Yes | 0.025 | Yes | 0.025 | Yes |

Table 10: Pairwise tests applied to the data regarding the thread friction coefficient for

| | Differences | LSD | Sign.? | HSD | Sign.? | NKD | Sign.? |
|------------------|-------------|-------|--------|-------|--------|-------|--------|
| t=0.2s vs t=2s | 0.018 | 0.011 | Yes | 0.016 | Yes | 0.011 | Yes |
| t=0.2s vs t=20s | 0.022 | 0.011 | Yes | 0.016 | Yes | 0.011 | Yes |
| t=0.2s vs t=200s | 0.044 | 0.011 | Yes | 0.016 | Yes | 0.014 | Yes |
| t=0.2s vs t=600s | 0.065 | 0.011 | Yes | 0.016 | Yes | 0.015 | Yes |
| t=2s vs t=20s | 0.040 | 0.011 | Yes | 0.016 | Yes | 0.014 | Yes |
| t=2s vs t=200s | 0.062 | 0.011 | Yes | 0.016 | Yes | 0.015 | Yes |
| t=2s vs t=600s | 0.043 | 0.011 | Yes | 0.016 | Yes | 0.016 | Yes |
| t=20s vs t=200s | 0.021 | 0.011 | Yes | 0.016 | Yes | 0.011 | Yes |
| t=20s vs t=600s | 0.043 | 0.011 | Yes | 0.016 | Yes | 0.014 | Yes |
| t=200s vs t=600s | 0.021 | 0.011 | Yes | 0.016 | Yes | 0.011 | Yes |

LOCTITE 243 application, black-oxidized screws

The results are collected in Tables 9 and 10 for zinc-plated and black-oxidized screws respectively, where significance is also highlighted. For the first ones, the frictional properties are initially unaffected by stop duration, up to 200 s. However, when it is extended to 600 s, a significant drop occurs, which makes the friction coefficient decrease to 0.11. This is a very low value, being consistent with that observed when tightening with lubricant addition [11] and consequently indicates a lubricating property, probably arising from PTFE particles, yielded by the applied threadlocker. As for black-oxidized screws, after an initial increase, friction gradually decreases, with significant differences even when comparing adjacent levels. The final value, for a 600 s stop is well comparable to that for zinc-plated screws, thus confirming the highlighted lubricating effect. The described outcome indicates that running a pre-tightening with a following stop is

beneficial for two reasons: pre-tightening promotes threadlocker adjustment in the pockets between the threads, with consequent friction drop. Secondly, this effect is enhanced a by a longer stop duration before final tightening, which makes it possible to conduct several assembly tasks in parallel. As an effect of lower friction, higher preloads may be achieved for fixed values of tightening torque. Moreover, the repeatability of the process under torque control is generally incremented [11].

Afterwards, the same tools were used to process the results involving LOCTITE 270. The results related to this strong threadlocker are collected in Fig. 7 for the two screw types, again with their mean values and variation intervals (corresponding to one standard deviation). The results in dry conditions are again appended to the bar graphs for comparison purposes. It must be remarked that the frictional coefficient for the shortest stop duration appears to be significantly incremented with respect to as received conditions: from 0.14 to 0.23 for zinc-plated screws and from 0.12 to 0.19 for those, which underwent black oxidization. This outcome could be due to the different viscosity of LOCTITE 270 with respect to LOCTITE 243. According to the datasheets, the strong threadlocker has 450 MPa s viscosity, determined according to the cone-plate test that well reproduces threadlocker actual insertion into the narrow pockets between the threads. Conversely, the medium strength threadlocker viscosity is 350 MPa s: the related difference may provide an explanation for the observed friction increment, immediately following the application of a more viscous (25% increment) threadlocker. However, longer stops after pre-tightening, lead to frictional coefficient decrease with a quite gradual trend (more regular than that for LOCTITE 243) to values being comparable (but slightly higher) to those measured in dry conditions.

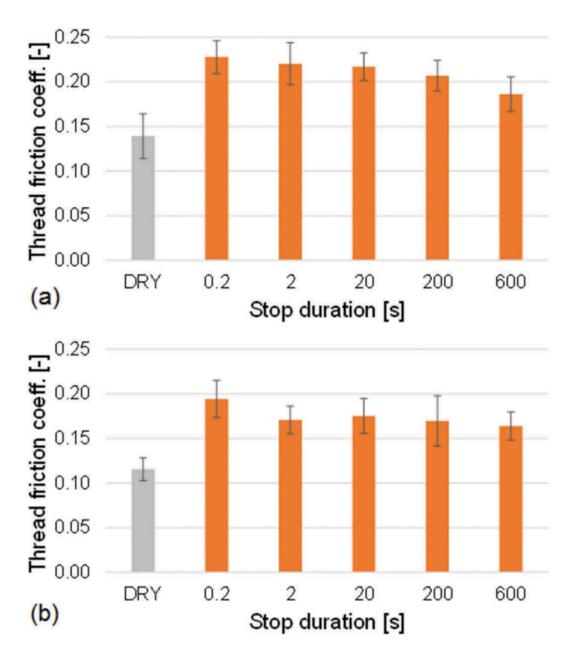


Fig. 7: Thread friction coefficient upon final tightening, following the application of LOCTITE 270 for (a) zinc-plated and (b) black-oxidized screws

These results have also been processed by the tool of two-factor ANOVA. The outcomes of the two tests, for zinc-plated and black-oxidized screws, confirmed the high significance of the stop duration on the retrieved tribological behavior upon tightening. Afterwards, the analysis was deepened, trying to allocate the differences highlighted by the analysis. The generally decreasing trend for friction is plotted in Fig. 8, where a 10base logarithmic scale is used for the stop elapsed time, considering its logarithmic progression of the first four levels.

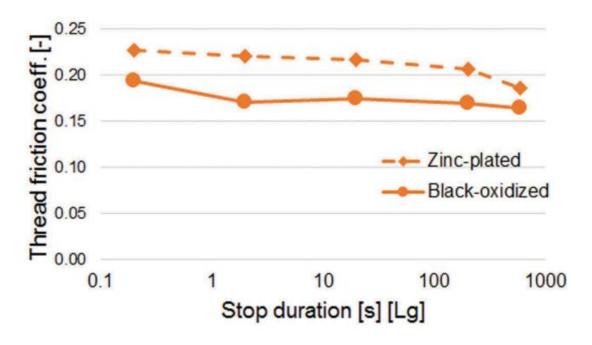


Fig. 8: Thread friction trend vs. stop duration, following LOCTITE 270 application for the two screw types

In order to allocate the differences, the aforementioned tools for pairwise tests were utilized. The outcomes of this analysis (which processed the results in Tables 4, 7) are provided in Tables 11-12 and make it possible to emphasize that the friction trend for zinc-plated screws is nearly constant, with not significant differences, up to a 200 s stop duration. Conversely, significant differences emerge for a stop duration of 600 s. On the other hand, when considering black-oxidized screws, friction drop is concentrated between 0.2 and 2 s stop duration levels, whereas longer stops lead to a further not significant decrease.

Table 11: Pairwise tests applied to the data regarding the thread friction coefficient for

| | Differences | LSD | Sign.? | HSD | Sign.? | NKD | Sign.? |
|------------------|-------------|-------|--------|-------|--------|-------|--------|
| t=0.2s vs t=2s | 0.007 | 0.014 | No | 0.019 | No | 0.014 | No |
| t=0.2s vs t=20s | 0.011 | 0.014 | No | 0.019 | No | 0.017 | No |
| t=0.2s vs t=200s | 0.021 | 0.014 | Yes | 0.019 | Yes | 0.018 | Yes |
| t=0.2s vs t=600s | 0.042 | 0.014 | Yes | 0.019 | Yes | 0.019 | Yes |
| t=2s vs t=20s | 0.003 | 0.014 | No | 0.019 | No | 0.014 | No |
| t=2s vs t=200s | 0.013 | 0.014 | No | 0.019 | No | 0.017 | No |
| t=2s vs t=600s | 0.031 | 0.014 | Yes | 0.019 | Yes | 0.018 | Yes |
| t=20s vs t=200s | 0.010 | 0.014 | No | 0.019 | No | 0.014 | No |
| t=20s vs t=600s | 0.031 | 0.014 | Yes | 0.019 | Yes | 0.017 | Yes |
| t=200s vs t=600s | 0.021 | 0.014 | Yes | 0.019 | Yes | 0.014 | Yes |

LOCTITE 270 application, zinc-plated screws

Table 12: Pairwise tests applied to the data regarding the thread friction coefficient for

| | Differences | LSD | Sign.? | HSD | Sign.? | NKD | Sign.? |
|------------------|-------------|-------|--------|-------|--------|-------|--------|
| t=0.2s vs t=2s | 0.023 | 0.015 | Yes | 0.021 | Yes | 0.018 | Yes |
| t=0.2s vs t=20s | 0.019 | 0.015 | Yes | 0.021 | No | 0.015 | Yes |
| t=0.2s vs t=200s | 0.025 | 0.015 | Yes | 0.021 | Yes | 0.020 | Yes |
| t=0.2s vs t=600s | 0.030 | 0.015 | Yes | 0.021 | Yes | 0.021 | Yes |
| t=2s vs t=20s | 0.005 | 0.015 | No | 0.021 | No | 0.015 | No |
| t=2s vs t=200s | 0.001 | 0.015 | No | 0.021 | No | 0.015 | No |
| t=2s vs t=600s | 0.011 | 0.015 | No | 0.021 | No | 0.018 | No |
| t=20s vs t=200s | 0.006 | 0.015 | No | 0.021 | No | 0.018 | No |
| t=20s vs t=600s | 0.011 | 0.015 | No | 0.021 | No | 0.020 | No |
| t=200s vs t=600s | 0.006 | 0.015 | No | 0.021 | No | 0.015 | No |

LOCTITE 270 application, black-oxidized screws

The described results highlight a different behavior of a strong threadlocker, LOCTITE 270, if compared to that of a medium one, LOCTITE 243. On one hand, a weaker threadlocker exhibits lubricating properties, meaning that the friction coefficient upon tightening is not initially altered by the addition of threadlocker, but tends to significantly decrease after approximately 10 min. The minimum value of the friction coefficient is consistent with that for lubricated mating surfaces. Therefore, a long stop time before

final tightening can be useful to complete pre-tightening tasks of several screws or other assembly operations to be run in parallel and also to achieve a lubricating effect. On the other hand, when using a strong threadlocker like LOCTITE 270, the friction coefficient is initially abruptly incremented with respect to dry conditions. However, the elapsed time before final tightening promotes friction drop, but this decrease is not sufficient to compensate the initial steep increment.

The complete tribological response versus the three involved factors, i.e. the stop duration, the pre-tightening speed, and the threadlocker type, was finally assessed by the tool of three-factor ANOVA. In particular, running this analysis made it possible to confirm the significance of the differences between the responses of the medium and high strength threadlockers. In fact, the application of a stronger threadlocker leads to generally higher friction coefficients for both the screw types. For the sake of synthesis, the ANOVA table containing the outcomes of the statistical assessment is reported for zinc-plated screws only in Table 13.

Table 13: 3-factor ANOVA to assess the effects of stop duration, pre-tightening speed, threadlocker type and presence (and related interactions) on the thread friction coefficient upon tightening with reference to zinc-plated screws

| | SSQ | DoF | MSQ | F _{calc.} | p-v |
|--------------------------------|--------------------|-----|--------------------|--------------------|---------------------|
| Effect of stop duration | 0.0291 | 4 | 0.0072 | 14.92 | 1.5.10-10 |
| Effect of pre-tightening speed | 4·10 ⁻⁵ | 2 | 2.10-5 | 0.04 | 0.96 |
| Effect of threadlocker type | | | | | |
| and presence | 0.2707 | 2 | 0.1354 | 278.2 | 9·10 ⁻⁵⁶ |
| Stop duration-pre tightening | | | | | |
| speed interaction | 0.0027 | 8 | $3 \cdot 10^{-4}$ | 0.69 | 0.70 |
| Stop duration-threadlocker | | | | | |
| type interaction | 0.0102 | 8 | 0.0013 | 2.62 | 9·10 ⁻³ |
| Pre-tightening speed- | | | | | |
| threadlocker type interaction | 0.0023 | 4 | 6·10 ⁻⁴ | 1.20 | 0.31 |
| 3-factor interaction | 0.0169 | 16 | 0.0011 | 2.17 | 8·10 ⁻³ |

| Error | 0.0876 | 180 | 5.10-4 | |
|-------|--------|-----|--------|--|
| Total | 0.4195 | 224 | 0.0019 | |

It is worth mentioning that the p-value being associated to the effect of stop duration is in the order of 10⁻¹⁰, whereas that related to the impact of threadlocker type is around 10⁻⁵⁵, which indicates the utilized threadlocker highly affects the achievable friction coefficients upon final tightening. The interaction between these factors is also beyond the significance threshold. In the authors' opinion, the outcome of 3-factor ANOVA also confirms it is more suitable to consider the response under the two threadlockers separately, as done above, to better understand their different properties. Finally, this overall study also confirms that the pre-tightening speed does not affect friction at all, as the related Fisher ratio is close to zero and the corresponding p-value is very close to 100%. The bar graphs in Fig. 9 resume the average overall responses for zinc-plated screws and well highlight the negligible effect of the pre-tightening speed and the different features of the threadlockers.

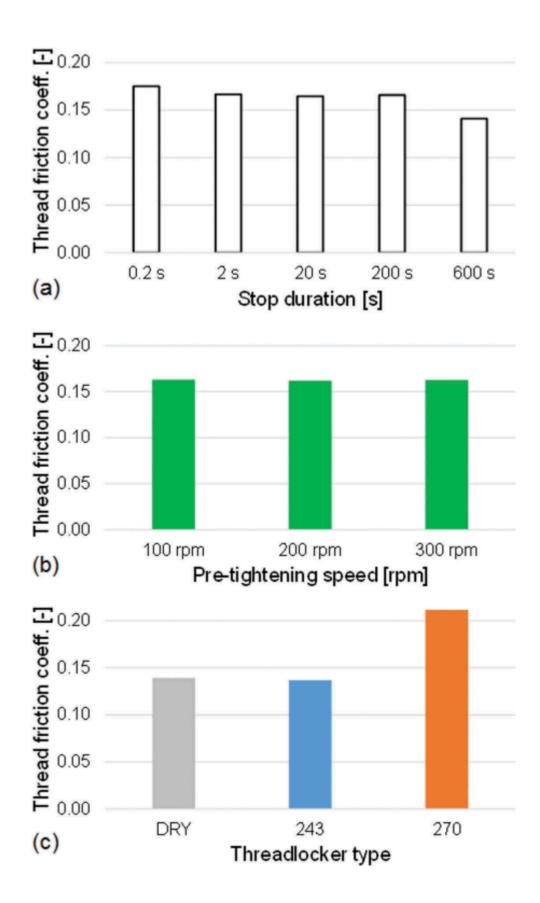


Fig. 9: Average overall response in terms of thread friction coefficient upon tightening versus (a) stop duration, (b) pre-tightening speed and (c) threadlocker type for zinc-

coated screws

A common outcome for both the considered adhesives with different strength, and for both the screw types, is that a lubricating effect occurs after tightening. This is also confirmed by Fig. 9 (a) that shows a globally decreasing trend of friction versus the stop duration, as an overall response accounting for both the threadlockers. The remarked friction decrease can be surely related to the presence of PTFE particles. However, a counter-intuitive outcome is that friction drop is generally not significant just after pretightening and a short subsequent stop duration. Conversely, it becomes significant, as proved by statistical tests, after a longer stop up to 600 s (10 min). According to the datasheets of both the adhesives, this duration is not sufficient to cure the studied adhesives, as their shear strength starts to weakly increase from zero after approximately 10 min. However, this time interval is likely to be sufficient for the occurrence of some physical/chemical processes being able to alter the tribological properties of the compound. The hypothesis is that the high pressure between the internal and external threads promotes the rapid polymerization of very thin films wrapped around the mating surfaces. Threadlocker being still in its liquid phase remains trapped at the interface between these films covering the steel surface. This occurrence modifies the tribological properties at the contact, which is now between the thin films and the threadlocker in its liquid phase. As a matter of a fact, friction decreases. A photo of some screws, which were treated by LOCTITE 270, after untightening, is shown in Fig. 10: the deposited threadlocker (green color) is well visible in all the threads even after untightening, which may support the adhesion of a very thin film.

It is clear that, if the stop gets longer than 10 min, then polymerization starts and a friction increment must be expected [17]. It must be remarked that after this time interval, the adhesive polymerization in the pockets between the threads takes place regardless of the tightening procedure (and related parameters) at the previous stage. Therefore, a conventional polymerization, having the capability of strengthening the fastener, increasing its breakaway torque, as highlighted in [17], can be reasonably expected.



Fig. 10: Screws treated by LOCTITE 270 after untightening: the deposited threadlocker (green color) is well visible in all the threads and highlighted by the arrows in the magnified view.

5. Conclusions

The described research had dealt with a particularly novel and original topic: the lubricity properties of threadlockers, i.e. of acrylic anaerobic adhesives being applied to screws, in order to increase the breakaway torque of the threaded fastener, thus preventing self-loosening. From this point of view, the present study has investigated an important additional effect of threadlockers, at reducing the thread frictional coefficient upon bolt

tightening. An extended experimental campaign was conducted by a testing rig for tribological tests, involving two screw types, zinc-plated and black-oxidized, and two threadlockers, with medium and high strength. The tightening process was inspired to the protocol adopted by Volkswagen and many other manufacturers in the automotive field. Regarding possible deviations from this protocol, the impacts of pre-tightening speed and of a longer stop duration between pre-tightening and tightening were investigated. The results, processed by statistical methods, indicate that pre-tightening speed is not significant, whereas stop duration is highly affecting, with consistent outcomes for the two screw types.

A medium strength threadlocker is able to lead to remarkably low friction coefficients (in the order of 0.11), following a 10 min stop before tightening. The observed occurrence has important industrial applications. In fact, on one hand, from the operative point of view, it is possible to take advantage of this time interval, to run several assembly tasks in parallel. On the other hand, higher preloads preventing self-loosening and fatigue failures may be achieved, as an effect of friction drop, for a fixed value of the tightening torque.

Conversely, a strong threadlocker does not have lubricating properties, if compared to the tribological response in dry conditions. However, a longer stop is again beneficial, to achieve a friction decrease that almost compensates its initial increase, following threadlocker application.

Finally, a longer stop up to 10 min is not expected to alter the adhesive polymerization that starts after 10 min, regardless of the previous tightening procedure, and is expected to increase the breakaway torque of the threaded joint.

References

*Corresponding Author: Giorgio Olmi Telephone +39 051-2093455 e-mail: giorgio.olmi@unibo.it

- Croccolo, D.; De Agostinis, M.; Fini, S.; Morri, A.; Olmi, G. Analysis of the influence of fretting on the fatigue life of interference fitted joints, Proceedings (IMECE) 2014, 2B, 38128.
- [2] Dragoni, E.; Mauri, P. Intrinsic static strength of friction interfaces augmented with anaerobic adhesives. *Int. J. Adhes. Adhes.* **2000**, *20* (4), 315–321.
- [3] Castagnetti, D.; Dragoni, E. Experimental investigation and model validation of the shear strength of hybrid interfaces up to complete failure. *J. Adhes.* 2016, 92 (7-9), 679–697.
- [4] Croccolo, D.; De Agostinis, M.; Mauri, P.; Olmi, G. Influence of the engagement ratio on the joint strength of press fitted and adhesively bonded specimens. *Int. J. Adhes. Adhes.* 2014, 53, 80–88.
- [5] Ragni, M., Castagnetti, D.; Dragoni E. Experimental validation of a simple shear strength model for hybrid friction-bonded interfaces. *Int. J. Adhes. Adhes.* 2018, 83 (6), 130–136.
- [6] Dragoni, E.; Mauri, P. Science of friction-adhesive joints. In *Hybrid Adhesive Joints (Advanced Structured Materials), Second Edition*; Da Silva, L. F. M.; Pirondi, A.; Ochsner, A.; Springer-Verlag: Berlin, Heidelberg, Germany, 2018; pp 201–225.
- [7] Haviland, G. S. *Machinery Adhesives for Locking, Retaining, and Sealing*. Marcel Dekker, Inc. New York and Basel, 1986.
- [8] George, B.; Grohens, Y.; Touyeras, F.; Vebrel, J. New elements for the understanding of the anaerobic adhesives reactivity. *Int. J. Adhes. Adhes.* 2000, 20, 245–251.

- [9] Croccolo, D.; De Agostinis, M.; Fini, S.; Olmi, G.; Robusto, F.; Vincenzi, N. Steel screws on aluminium nuts: Different engagement ratio tapped threads compared to threaded inserts with a proper tolerance choice. *Tribol. Int.* **2019**, *138*, 297–306.
- [10] Murray, B. D. Keeping Bolts Tight with Anaerobics. *Machine Design* 1976, 48 (7), 135–139.
- [11] De Agostinis, M.; Fini, S.; Olmi, G. The influence of lubrication on the frictional characteristics of threaded joints for planetary gearboxes. *P. I. Mech. Eng. C-J. Mec.* 2016, 230 (15), 2553–2563.
- [12] Loctite Corporation. Loctite Worldwide Design Handbook. Düsseldorf: Erasmusdruck, Mainz; 1998.
- [13] LOCTITE® WEBINAR SERIES Threadlocking & the Torque-Tension Relationship. Available on-line at http://henkeladhesivesna.com/knowledgecenter/wp-content/uploads/2017/02/Reliable-Threaded-Fastener-Assemblies-Torque-Tension-Relationship-K-Factor-1.pdf (last visited on 6th October 2019).
- [14] Dube, M. J., Gamwell, W. R. Performance Characterization of Loctite® 242 and 271 Liquid Locking Compounds (LLCs) as a Secondary Locking Feature for International Space Station (ISS) Fasteners. NASA Internal Report NASA/TM-2011-217068 NESC-RP-04-092. Available on-line at: https://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20110011064.pdf (last visited on 6th October 2019).
- [15] Sekercioglu, T.; Kovan, V. Torque strength of bolted connections with locked anaerobic adhesive. P. I. Mech. Eng. L-J. Mater. Des. Appl. 2008, 222 (1), 83–89.
- [16] Castagnetti, D; Dragoni, E. Adhesively-bonded friction interfaces: macroscopic shear strength prediction by microscale finite element simulations. *Int. J. Adhes. Adhes.* 2014, 53, 57–64.

- [17] Croccolo, D.; De Agostinis, M.; Fini, S.; Olmi, G. An experimental study on the response of a threadlocker, involving different materials, screw dimensions and thread proportioning. *Int. J. Adhes. Adhes.* **2018**, *83*, 116–122.
- [18] ISO 16047:2005(E). Fasteners torque/clamp force testing. International Organization for Standardization: Geneva, Switzerland, 2005.
- [19] Croccolo, D.; De Agostinis, M.; Fini, S.; Olmi, G. Tribological properties of bolts depending on different screw coatings and lubrications: an experimental study. *Tribol. Int.* 2017, 107, 199–205.
- [20] ISO 898-1:2013(E). Mechanical properties of fasteners made of carbon steel and alloy steel. International Organization for Standardization: Geneva, Switzerland. 2013.
- [21] Nassar, S. A.; Matin, P. H.; Barber, G. C. Thread friction torque in bolted joints. J. Pressure Vessel Technol. 2005, 127, 387–393.
- [22] Sriraman, K. R.; Strauss, H. W.; Brahimi, S.; Chromik, R. R.; Szpunar, J. A.; Osborne, J. H.; Yue, S. Tribological behaviour of electrodeposited Zn, Zn–Ni,Cd and Cd–Ti coatings on low carbon steel substrates, *Tribol. Int.* **2012**, *56*, 107–120.
- [23] Farrell. R. Jr. Blackening of ferrous metals. Met. Finish. 2007, 105 (10), 390–396.
- [24] Berger, P. D.; Maurer, R. E. Experimental design with applications in management, engineering and the sciences. Duxbury Press, Belmont, CA, 2002.
- [25] Montgomery, D. C. Design and analysis of experiments. Wiley, New York, 2001.