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Synergetic Effects of Surface Texturing and Solid

Lubricants to Tailor Friction and Wear – A

Review

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Abstract

Surface texturing and solid lubricants have demonstrated the ability to substantially reduce friction and wear under dry conditions. In recent decades, these two technologies have been

combined to leverage the advantages of both for superior tribological performance. This review article first summarizes the state-of-the-art regarding surface texturing and solid lubricants, including soft metals, polytetrafluoroethylene, diamond-like carbon and 2D layered materials. Then, the synergy between surface textures and solid lubricants is discussed, with particular emphasis on the underlying mechanisms. Finally, gaps in the existing understanding of these synergies are identified and opportunities for future research are suggested.

1. Introduction

Tribology connects friction, wear and lubrication with aspects of materials science, chemistry, physics and even biology in a highly inter- and multi-disciplinary manner. Although often not noticed, friction- and wear-related phenomena are present in everyday life and have become more important in recent years due to energy and resource concerns [1-4]. Beyond well-known examples of tribology in the world of engineering [2, 5], including mechanical components such as bearings, gears, brakes and piston rings, tribological phenomena are relevant to a broad range of applications, such as contact lenses [6, 7], lipstick or other cosmetical products [8, 9], artificial hip or knee joints [10, 11] and even drinking wine as oral tribology [12, 13].

The importance of tribology is particularly evident in the context of several global challenges. Due to the rapidly growing population on earth, the demand for energy has been increasing steadily every year, reaching 400 Exajoules in 2019 [2]. From an environmental point of view, continuously decreasing resources and the need to reduce CO₂ emissions to slow global warming need to be addressed in the near future [14]. These environmental concerns are

being partially addressed by new, more stringent legal restrictions on permissible CO₂ limits and the usage of certain lubricant additives. Considering all these challenges, it is evident that greener, smarter, more efficient and environmentally friendly technologies, processes and systems are more urgently needed than ever before [2, 4].

Historically, the easiest approach to reducing friction and wear is to apply a lubricant (oil or grease) between two rolling or sliding surfaces to decrease solid-solid contact, moving the contact from dry to lubricated [15]. Besides the use of liquid lubricants, tribological properties can be greatly improved by changing the involved materials and/or component designs, or by making use of advanced approaches for surface engineering. Among these technological approaches, surface textures [16-19] and solid lubricant coatings [20, 21] are two of the most promising avenues being explored to improve friction and wear.

Surface texturing has gained increasing attention over the last three decades. Boosted by the work of Etsion and co-workers in the 90's [16, 22, 23], surface textures have been shown capable of beneficially affecting friction and wear under dry friction conditions [24, 25] as well as boundary [26-29], mixed [30-33], elastohydrodyamic (EHL) [34-36] and full-film hydrodynamic lubrication [37-39]. Under dry conditions, surface textures help to reduce the real area of contact and trap wear debris, thus lowering friction and extending wear life [18]. Surface coating technologies are based on two general approaches: a hard substrate with a soft coating (soft-on-hard) or a soft substrate with a hard coating (hard-on soft). The first approach is based on the low shear strength of soft materials to facilitate relative motion between surfaces, while the latter intends to reduce the real area of contact [20, 21]. According to Bowden and Tabor [40], friction force is the product of the real area of contact and shear strength, which directly implies lower friction for materials with reduced shear

strengths or interfaces with smaller contact areas. Solid lubricants, often applied as coatings, have received significant attention in the tribological community due to their excellent performance, even under harsh conditions such as high temperature and vacuum [41-44]. Solid lubricants can be classified into four categories: soft metals such as gold; polymers such as polytetrafluorethylene (PTFE); 2D layered materials including graphite and MoS₂; as well as hard carbon-based materials such as diamond-like carbon (DLC) [44, 45].

Despite the promise of these technologies, both surface textures and coatings have limitations, and their limitations become particularly evident under high load and low speed conditions [18-21]. Under such conditions, surface textures and coatings may be worn away quickly, such that they can no longer reduce friction and/or wear. This can also potentially lead to the formation of abrasive wear debris that accelerates wear and the associated degradation processes, thus worsening friction and wear performance [18]. A potential strategy to improve tribological properties under more severe conditions lies in the combination of surface texturing with surface coatings.

There are two conceptual approaches to combining textures with solid lubricant coatings. First, the coatings themselves can be textured. Textured coatings may have lower wear due to the smaller contact area and potentially trap wear debris, thus removing them from the tribological interface and extending the service lifetime. This approach is typically used for hard, wear-resistant coatings. Second, coatings can be deposited on textured substrates. In this case, the synergy lies in either the ability of the surface textures to store and re-supply the solid lubricant material to the contact or the ability of the hard coating to prolong the life of the textures.

Motivated as above, this review article explores the synergy between surface texturing and solid lubricant coatings for dry conditions. Note that this review will not cover studies in which the textures' main role is to induce hydrodynamic effects or improve lubricant supply under lubricated conditions, which has already been addressed in other reviews [17, 18, 46, 47]. After a brief introduction, the state-of-the-art regarding surface texturing and solid lubricants will be concisely summarized. Then, the synergy between surface texturing and solid lubricants will be described for different classes of solid lubricants, with special emphasis on the underlying friction and wear mechanisms. Depending on the coating material, different strategies for combining surface texturing and solid lubricants have been explored and tested, with varying results. Finally, based on the review of existing synergies, short-comings of current approaches will be identified and then corresponding opportunities for potential future research directions will be proposed.

2. Surface Texturing under Dry Conditions

Texturing involves the controlled modification of topography to produce functional surfaces [48, 49]. The ability of surface textures to control friction and wear under dry and lubricated conditions has been exhaustively investigated for more than three decades. Under lubricated conditions, the main mechanisms behind the improved performance of textured surfaces are the increase of the hydrodynamic film thickness and load bearing capacity [19, 37, 50-52], increased lubricant supply [53-56] and increased inlet suction [53, 57]. Gropper et al. presented a thorough review of textured surfaces under hydrodynamic lubrication [17]. Other contact conditions and lubrication regimes have been reviewed by Gachot et al. [18]. For non-conformal contacts, small contact areas and the possibility of inducing contact fatigue

by texturing [58, 59] as well as the need to maintain high viscosity inside the contact demand substantially narrower and shallower textures [60]. In boundary lubrication, the main effects of surfaces textures are to increase the lubricant supply [61] and to increase the contact pressure at the textures' edges which activates lubricant additives via stress-induced tribochemical reactions [62, 63]. Considering that success or failure of surface texturing depends on contact and operation conditions, the use of surface texturing for different machine elements (seals, gears, roller bearings and piston-ring assemblies in automotive systems) has also been reviewed, providing guidelines for optimal texture design for each application and lubricated condition [19]. This review, however, focuses on relative motion in dry conditions.

2.1 Mechanisms for Improved Performance under Dry Conditions

Phenomena occurring at tribological interfaces that dissipate energy during sliding, such as adhesion, ploughing by the asperities or wear debris, elastic/plastic deformation and fracture, are largely influenced by the surface topography [64]. Therefore, surface texturing has the potential to improve friction and wear, even in dry conditions.

Some of the earliest studies of dry friction on textured surfaces were carried out by Suh et al. [64, 65]. Substantially lower coefficients of friction (COFs) were measured for textured copper shafts (0.25) when compared with smooth shafts (0.75) [64]. The authors suggested that the grooves trapped wear debris, thus removing them from the sliding interface. Trapping wear debris both reduces ploughing and inhibits the generation and agglomeration of larger debris that can induce delamination.

Since then, other studies have reported lower friction [66-73] and reduced wear [66, 68-70, 72, 74, 75] on textured surfaces attributed to the ability of textures to trap wear debris. For

instance, different textures (parallel and perpendicular grooves as well as dimples) on brass reduced friction after running-in and the corresponding wear rates for all textures in comparison to smooth brass (Figure 1). This was linked to the entrapment of wear debris in the textures [68].

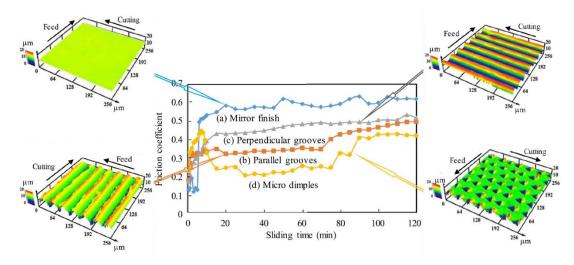


Figure 1: Temporal evolution of COF during dry ball-on-disk tests of smooth and textured brass samples, with corresponding 3D topographical maps. The increased COFs for the textured samples in the first 5 minutes of the experiment can be correlated with the presence with material pile-ups induced by vibration-assisted microcutting. Reprinted (adapted) with permission from [68].

In addition to the entrapment of wear debris, adhesion between moving surfaces can be tuned by surface texturing. Gachot et al. demonstrated that laser-textured stainless-steel surfaces containing parallel grooves sliding against grooved balls have lower friction than smooth surfaces (balls and specimens). Moreover, when the textures on the ball and substrate were oriented at 90°, friction was lower than for parallel alignment [25]. In a follow-up paper, the authors showed that the lower friction for the perpendicular orientation was due to increased plastic flow for the parallel orientation, which increased the real contact area and adhesion, as well as more efficient removal of wear debris for the perpendicular alignment [73].

Another relevant aspect of surface texturing is how textures affect the stress distribution in and around the contact region [76]. The high contact pressures acting at the textures' edges have been identified as a major drawback for their use in dry conditions [46]. Finite element modelling has facilitated the analysis of stress and strain distributions for textured surfaces. Xing et al. proposed a finite element model for the contact of a cylindrical pin (flat geometry) sliding against smooth and textured (parallel and zigzag grooves) ceramic surfaces. These simulations demonstrated that the stress distribution is more uniform for textured surfaces, although the magnitude of the maximum von-Mises-stress was slightly increased relative to the smooth case. However, large stresses occur at the edges of the textures [66].

The effects of surface texturing on heat transfer and surface energy, which can indirectly affect friction, are also relevant. The presence of textures can increase heat transfer by either convection or due to thermocapillary migration induced by differences in localized temperature and surface energy (Marangoni effect) [49]. For tribological systems involving high contact pressures, for which friction leads to high localized temperatures, improved heat transfer away from the contact can potentially reduce wear. In metal cutting of nickel-based alloys, textured tungsten carbide tools reduced tool wear, thus increasing tool life [77], apparently because surface texturing can improve heat exchange in the tool-workpiece interface [77, 78].

Surface texturing can also affect the wettability of surfaces, which may influence the adhesion component of friction. Bico et al. numerically showed that surface texturing increases capillary effects for hydrophilic surfaces, making them more hydrophilic. In contrast, in the case of hydrophobic surfaces, the air entrapped within the textures increases the contact angle, making the surfaces more hydrophobic [79]. This can be important for

applications in which adhesion is relevant, such as micro electrical mechanical systems (MEMS). In such small scale devices, meniscus forces (which depend on wettability) can arise due to the atmospheric humidity and affect friction even in dry conditions [80].

2.2 Detrimental Effects of Surface Texturing under Dry Conditions

Higher friction and/or wear for surface texturing have also been reported. For textured stainless steel with parallel grooves, it was found that, when the area coverage was excessive, friction was higher for the textured surface than for the smooth surface. This was attributed to increased contact pressures, probably inducing a transition from elastic to plastic contact conditions, increasing the deformation component of friction [81]. When sliding textured aluminum surfaces against polydimethylsiloxane (PDMS) balls, the adhesion component of friction increased. This probably occurred due to the compliant nature of the ball, increasing the real contact area for the textured specimen [82]. For laser surface texturing (LST) of polyether-ether-ketone (PEEK), only wider dimples (50 µm) induced friction and wear reduction, while narrower dimples (25 µm) did not reduce friction and increased wear. It was proposed that narrower dimples could not remove the wear debris from the contact and increased contact pressures around the dimples, thus increasing wear [75]. WC inserts textured by electrical discharge machining (EDM) exhibited reduced tool life, which was attributed to large residual stresses induced by EDM due to high temperatures during texturing [83]. The use of surface texturing on aluminum sheets increased friction and adhesive wear during strip drawing tests. For high contact pressures, it was speculated that the reduction in load bearing capacity with texturing caused intense ploughing of the sheet surface, leading to severe adhesive wear [84].

2.3 Design Guidelines for Surface Texturing under Dry Conditions

Since 1990, more than 3,700 articles can be found in the database "Web of Science" on the topic of surface texturing and friction, and more than 2,700 articles on surface texturing and wear. Despite the large number of studies using surface texturing for tribological applications, consensus regarding the effectiveness of surface textures and full understanding of the mechanisms involved have not been achieved yet [18].

Based upon the existing literature, we propose that the design of textures for dry conditions should take into account eight different points. First, if the distance between the textures is too large, efficient entrapment of debris may not occur, leading to agglomeration of wear debris and ploughing. Second, if the area coverage is too high, there might not be enough smooth areas to carry the normal load, thus lowering the load bearing capacity. Third, when the textures become too deep, edge stresses may become detrimental, thus inducing excessive plastic deformation. Fourth, the size of the textures must allow the largest debris to be accommodated. Fifth, the effects of the surface texturing technique on generation of residual stresses should be minimized. Sixth, the effects of the textures on properties that can indirectly affect friction and/or wear need to be understood, in particular heat transfer and surface energy. Seventh, rationally designed textures, particularly those identified using

guidance from simulations, can be used to improve tribological performance. The eighth and most important point in this review is the possibility of combining surface texturing with solid lubricants. Although surface texturing alone can reduce the ploughing and adhesion components of friction under dry conditions, can this be further reduced with a solid lubricant? Design guidelines for textures combined with solid lubricants will strongly depend on the solid lubricant as well as if and how texturing affects its structural and chemical properties.

3. Friction and Wear Mechanisms in Solid Lubricants

There are many technical applications for which liquid lubricants are not feasible that, instead, make use of solid lubricants. These applications often involve one or more of the following conditions: high or low operating temperatures, operation under vacuum or at low pressures, corrosive environments, large loads, high risk of contamination, stringent limits on weight, as well as complications associated with service and/or replacement of liquid lubricants. Solid lubricants help to overcome some or all of these short-comings [44, 85, 86]. The goal of this section is to provide an overview of friction and wear mechanisms for the most prominent classes of solid lubricants, including soft metals, polymers (with the example of PTFE, i.e. Teflon), DLCs and 2D layered materials (in particular, graphite/graphene and MoS₂, as well as MXenes). As extensive reviews that focus on different aspects of solid lubrication exist in literature (see, e.g. [42, 44, 85-90]), the discussion here is restricted to the basics of the underlying mechanisms, serving as an introduction to the main focus of this review: the combination of solid lubricants with textures to improve friction and wear.

3.1 Solid Lubrication with Soft Metals

Soft metal coatings have been employed as solid lubricants in certain applications since the first half of the 20th century [91]. The main physical characteristic making soft metals suitable for use as solid lubricants is their relatively low shear strength, which is further reduced at high temperatures where most other solid lubricants fail. Consequently, metals such as gold, silver, platinum, indium, tin, and lead have been most often employed as solid lubricants. The fact that soft metals feature multiple slip planes and are able to heal microstructural defects effectively through frictional heat further contributes to their effectiveness as solid lubricants.

The classic model of Bowden and Tabor [40] explains the physical mechanism behind the solid lubrication achieved by a thin layer of soft metal on a relatively hard and stiff substrate. According to this concept, friction experienced by an asperity sliding on such a surface can be calculated from the product of the interfacial shear strength and the contact area. In the case of a soft thin film on a hard and stiff substrate, most of the load is carried by the substrate, which results in a small contact area due to its mechanical strength. Moreover, the soft thin film offers a reduced shear strength; the combination with small contact areas leads to low friction, i.e. solid lubrication.

The argument described above means that friction exhibited by a soft metal films depends on film thickness. In fact, as film thickness increases, a greater portion of the total load is carried by the soft metal, which results in larger contact areas and, therefore, higher friction. When the soft metal film becomes too thin, topographical features of the substrate start to interact with those of the counter-body, thus increasing friction. As such, an optimum range of film thickness exists for different scenarios, e.g. the optimum film thickness was reported to be on the order of 1 µm for an indium film deposited on tool steel that has not been highly polished [40]. This value naturally depends on the substrate used, as well as its roughness, and the soft metal employed. It should also be noted that thin films naturally wear out faster than thick films since wear of thick films is typically accompanied by plastic deformation through dislocation motion (see Figure 2 for a representative wear track on a silver film).

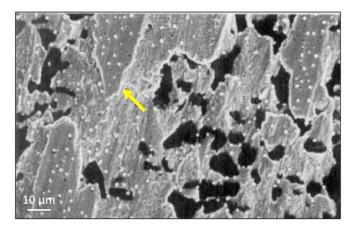


Figure 2: SEM image of a wear track on a 2-μm-thick silver coating deposited on an alumina substrate, recorded after sliding against bare alumina. The dark regions are where the film wore off. The yellow arrow highlights shear patterns attributed to dislocation motion in the silver film in the direction of sliding. Reprinted (adapted) with permission from [92].

A particular point that needs to be taken into account when choosing soft metals for a given application is the operating temperature. While soft metals generally experience a decrease in shear strength at high temperatures, metals such as tin and indium chemically degrade via oxidation at higher temperatures, while gold and silver are more resistant to such effects. Consequently, silver has been widely utilized as a solid lubricant in high-speed ball bearings of X-ray tubes and at ceramic interfaces that experience high degrees of heating through friction [85].

Despite the success of soft metal solid lubricants, they have limitations, in particular with respect to substrate adhesion and wear. The combination of soft metals with surface texturing may overcome some of these limitations, as discussed in Section 4.1.

3.2 Solid Lubrication with PTFE

Polymers are attractive as engineering materials for many applications in terms of cost, weight and ease of manufacturing. Polymeric materials used for solid lubrication include PTFE, PEEK, polyimide (which is typically employed for high temperature applications), and ultra-high molecular weight polyethylene for biomedical implants. Among these, PTFE is the most widely utilized due to its excellent frictional performance [87]. Because of this, and the significantly different tribological mechanisms for other polymers, this review will solely focus on the friction and wear of PTFE.

While PTFE is most commonly associated with non-stick coatings used in cookware, its ability to exhibit low COFs (down to \sim 0.05) has also led to its wide use as a solid lubricant. This behavior can be traced back to the low shear strength between the macro-molecules $(CF_2-CF_2)_n$ that make up PTFE [87, 93], which leads to low resistance to deformation in the direction of sliding and, consequently, low friction. The material's relatively high melting point of about 342 °C, chemical inertness and thermal stability also contribute to beneficial frictional properties.

Since the low friction of PTFE is inherently tied to its structure consisting of individual chains of macromolecules that slide against each other, friction of PTFE depends on speed and temperature. In particular, the COF of PTFE has been shown to increase with increasing sliding speed, in a viscoelastic fashion, as the macro-molecular constituents have less and less time to "relax" at higher sliding speeds [94].

A characteristic feature that enhances PTFE's utility as a solid lubricant is the fact that it easily forms chemically stable transfer-films on a counter-surface, even at low pressures of only a few kPa (Figure 3) [95]. Once the formation of the transfer-film on the counter-surface occurs, the frictional interface consists of PTFE-PTFE contacts, inducing low friction. The ease with which PTFE can be transferred onto surfaces of materials, including metals and glass, provides a practical route for tribological properties to be modified. The molecular-scale mechanisms involved in the formation of transfer films are still not completely understood, making them a subject of active computational [96] and experimental research [95, 97, 98].

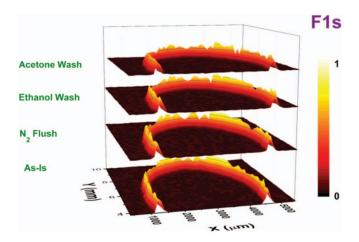


Figure 3: Maps of F_{1s} peak intensity acquired via X-ray photoelectron spectroscopy on an area where circular tracks of PTFE have been transferred onto a SiO₂ substrate via scratching by a piece of PTFE. The transferred PTFE films are resistant to chemical treatments including acetone and ethanol washes. Reprinted (adapted) with permission from [95].

Despite its attractive frictional properties, pure PTFE typically suffers from poor wear resistance, reflected by specific wear rates on the order of 10⁻⁴ mm³/Nm [97, 99-101]. The same molecular mechanisms leading to low COFs (weak interactions between macromolecular chains) also result in low cohesion. The onset and progression of wear in pure PTFE are typically correlated with the initiation and growth of subsurface cracks that increase the probability of large wear debris formation. Large wear debris tends to form

lumpy, discontinuous and incomplete transfer films that are not capable of inducing ultralow wear [97, 100, 101].

However, the wear properties of PTFE can be significantly improved using suitable fillers materials, thus producing composites with PTFE as the matrix and the fillers acting as the reinforcement phase [102, 103]. Studies making use of α -alumina nano-particles as filler material demonstrated a reduction of the wear rate by four orders of magnitude, accompanied by the formation of thin, homogeneous, continuous and tenacious transfer films [97, 101, 104, 105]. Since then, many filler materials, including carbon fibers [106], carbon nanotubes [107], graphite flakes [108] and glass fibers [109], among others, have been shown to improve the wear performance of PTFE, resulting in COFs of less than 0.2 and specific wear rates on the order of 10⁻⁷ mm³/Nm. The improved performance has been attributed to the interplay between the polymeric surface, the embedded filler particles, the counter-body (often metallic) and the size of the formed polymeric wear debris [97, 101, 110, 111]. Generally, the successful use of filler materials depends on the size, shape and concentration of the filler materials embedded in the PTFE matrix as well as the experimental testing conditions [112]. In addition to tribo-chemical reactions, tribo-film adhesion to the substrate and the substrate roughness affect the PTFE wear performance [101, 110, 111, 113, 114]. This sensitivity of tribological performance on the substrate roughness provides the foundation on which the combined use of PTFE and surface textures are discussed in Section 4.2.

3.3 Solid Lubrication with Diamond-like Carbon

Since its first synthesis in 1971 [115], DLC has become a successful carbon-based coating with widespread industrial applications. DLC is a metastable form of amorphous carbon with a high ratio of tetrahedral sp³- (diamond-like) to sp²- (graphite-like) bonds [115, 116]. DLC has an unusual combination of properties, including high hardness, wear resistance, low friction, high corrosion resistance, biocompatibility [117], low surface roughness and optical transparency [118]. Moreover, thermal and electrical conductivity can be tuned depending on the relative amount of sp³- and sp²-bonding. These characteristics have led to the widespread use of DLC films in industry, ranging from coatings of complex engine parts to common mechanical components such as gears and pins [119].

The term DLC describes a large family of coatings that are differentiated based on the ratio between sp³- and sp²-hybridization, the amount of hydrogen (H), as well as other dopants [116]. Films with a large content of sp³-bonds are very hard and are called tetrahedral amorphous carbon (ta-C) [116], whereas the almost total prevalence of sp²-bonds leads to graphite-like carbon (GLC) films [120]. Amorphous carbon (a-C) and amorphous hydrogenated carbon (a-C:H) films are also considered DLCs. However, they typically have a much lower content of sp³-bonds compared to ta-C, as well as a large amount of H in the case of a-C:H films, which generally results in lower hardness [116].

Depending on their chemical composition and environmental factors, DLC films can exhibit a wide range of COFs, all the way from 0.001 to as much as 0.7 [88]. An important distinction between hydrogenated and hydrogen-free DLC films exists within this context. While hydrogen-free films require humid conditions to deliver low COFs (~0.1) but do not perform well as solid lubricants under dry conditions or inert atmospheres (with COFs up to ~0.7), hydrogenated DLCs provide low COFs under dry conditions but not at high humidity levels (Figure 4) [44, 88]. The related mechanism has to do with dangling σ-bonds on the surfaces

of DLC, on both the original coating and the transfer film formed on a counter-body sliding against DLC-coated components [121]. While these dangling bonds are mostly passivated in hydrogenated DLCs (i.e. a-C:H), they covalently interact with each other in the absence of environmental oxygen and water in case of hydrogen-free DLC films (i.e. ta-C), thus leading to increased adhesion and high COFs. Moreover, water layers adsorbed on hydrogenated DLC surfaces at high humidity may lead to increased friction through dipole-like interactions [121]. Factors other than hydrogen content that significantly influence friction include the topographic roughness of the surfaces on which the DLC films are applied, the magnitude of the applied loads, as well as sliding speed and temperature [88].

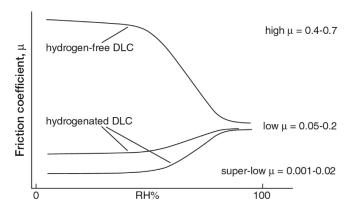


Figure 4: Schematic dependence of the COF on environmental humidity for hydrogen-free and two kinds of hydrogenated DLC films with varying hydrogen content. Reprinted (adapted) with permission from [121].

The wear behavior of DLC films also greatly depends on the chemical composition and testing conditions [88]. In certain cases, low COFs correlate with low specific wear rates (down to values on the order of 10⁻⁹ mm³/Nm). Hydrogen-free DLC films tend to exhibit more wear under dry and inert atmospheres when compared with humid conditions where which their inherently high hardness results in low wear rates. It should also be noted that rougher substrates lead to a higher rate of third body formation and, consequently, wear. The

tribological performance of DLCs can also be improved by chemical doping with metals and other elements [122].

Despite their attractive tribological properties in most circumstances, DLC films may present unsatisfactory performance under highly loaded conditions or when applied to soft substrates, which results in the DLC film carrying a large portion of the load. These conditions can result in excessive spalling [123], or increased ploughing and plastic deformation (more relevant for films with increased sp²-content) [124]. The use of surface textures has the potential to alleviate these issues, as discussed in Section 4.3.

3.4 Solid Lubrication with 2D Layered Materials

2D layered materials, including graphite/graphene as well as transition metal dichalcogenides (TMDs), such as MoS₂ and WS₂, are employed as solid lubricants in applications ranging from electrical contact brushes to bearings in satellites and space probes, as well as constituents in composite solid lubricants with metallic or polymeric matrices [42, 45, 125]. Here, we focus on two of the most frequently utilized 2D layered solid lubricants, graphite and MoS₂, as well as an emerging class of 2D layered solid lubricants, MXenes.

A widely accepted physical argument behind the low friction exhibited by mechanical components coated with layered materials such as graphite and MoS₂ involves their lamellar crystalline structure, as first observed by Bragg through X-ray diffraction performed on graphite [126]. While atoms forming the individual sheets (i.e. layers) of a layered material are covalently bound with each other, the individual lamella are held together only through weak, van der Waals interactions. This characteristic bonding structure, which makes it easy to "peel" individual lamellae from bulk material [127], also results in a remarkably low

resistance to shear. Specifically, much like individual cards in a stacked deck, when subjected to shear, sheets of layered materials slide with relative ease on top of each other, resulting in low friction.

Despite the fact that the basic physical picture described above provides a simple, structural argument for the lubricity exhibited by layered materials, the actual mechanisms are more complex. This is highlighted by the fact that graphite needs a relatively humid environment to function effectively as a solid lubricant. In contrast, it performs poorly under vacuum or at low pressures, resulting in high friction and wear rates accompanied by the phenomenon of "dusting" [125].

The aforementioned general mechanism based on low shear strength in the sliding direction is also responsible for the solid lubrication characteristics of the prototypical TMD, MoS₂ [42]. Transmission electron microscopy studies have shown (i) the formation of an MoS₂ transfer-film on an originally uncoated surface in sliding contact with an MoS₂-coated part, and (ii) that MoS₂ basal planes (which may be originally randomly oriented or even perpendicular relative to the surface of the coated component) become oriented parallel to the direction of sliding through shear forces during relative motion [128, 129]. Experiments have further revealed that individual MoS₂ sheets aligned on top of each other are often rotationally incommensurate, i.e. have mismatching registry, which leads to ultra-low friction through structural superlubricity [130], thus resulting in the experimentally observed low COFs for MoS₂-coated parts.

A key difference between graphite and MoS₂ involves their tribological behavior under different environmental conditions. In contrast to graphite, MoS₂ exhibits low friction and wear in vacuum, but its functionality as a solid lubricant quickly deteriorates in the presence of ambient gases such as H₂O and O₂; see Ref. [42] for a detailed discussion and Figure 5 for

a schematic description of the processes proposed to be involved in the humidity dependence of friction on graphite, as well as TMDs like MoS₂ [131]. It should also be noted that doping, in particular with transition metals, has been shown to improve the tribological performance of MoS₂ [132].

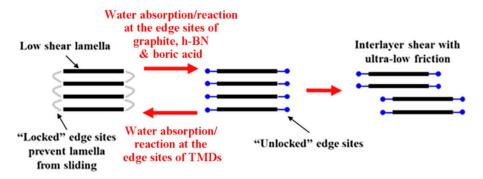


Figure 5: Proposed mechanisms to explain the humidity dependence of friction for graphite/h-BN/boric acid as well as TMDs such as MoS₂. Reprinted (adapted) with permission from [131].

In addition to theoretical and computational work aimed at elucidating the physical reasons behind the lubricative properties of layered materials [133-135], there have also been experimental studies that directly probe the underlying mechanisms with high spatial resolution. In addition to electron microscopy studies that manipulated individual sheets of layered materials in a controlled way while measuring the related forces during sliding [136, 137], atomic force microscopy (AFM) experiments performed on single- or few-layer samples of graphene and MoS₂ have revealed intriguing tribological characteristics on the nanometer length-scale, including but not limited to, layer- [138], speed- [139] and direction-dependence (anisotropy) [140-142]. AFM has also been used to investigate friction mechanisms for other TMDs, including WS₂ and WSe₂ [143] as well as MoSe₂ and MoTe₂ [144].

Lastly, a recently discovered class of layered materials - MXenes - are being explored for solid lubrication. MXenes are obtained by selective etching of the A layers from $M_{n+1}AX_n$

(where M: early transition metal, A: group IIIA or IVA element and X: C or N with n=1, 2 or 3) [145, 146]. While their lubricious properties by themselves [147-149] or as part of composite coatings [150-153] have been studied on the macroscopic scale, AFM studies have recently quantified the nanoscale solid lubrication achieved by individual Ti₃C₂T_X nanosheets (Figure 6) [154] and demonstrated the temperature dependence of adhesion and friction on Ti- and Nb-based MXenes [155]. These studies pave the way for further fundamental work on the potential of this exciting new material system for solid lubrication. Much like the other solid lubricants discussed earlier, various attempts have been made to improve the tribological performance of layered materials through surface texturing, as discussed in detail in Section 4.4.

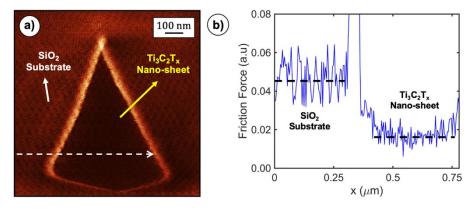


Figure 6: (a) Friction force map recorded on a few-layer MXene nano-sheet on a SiO_2 substrate. Darker colors correspond to lower friction. (b) Friction force profile extracted along the white dashed arrow in (a). The average friction recorded on the MXene nano-sheet is ~35% of that recorded on the SiO_2 substrate. Reprinted (adapted) with permission from [154].

3.5 Summary of Solid Lubricants

The field of solid lubrication has reached a certain level of maturity, but a number of major challenges still remain. Soft metals may work well at high temperatures, but do not provide sufficiently low friction levels like other solid lubricants, and frequently suffer from adhesion-related problems. Polymer-based solid lubricants, while cost effective and capable

of reducing friction considerably, typically rely on complex tribo-film formation processes and provide only limited wear resistance, which restricts their long-term application in scenarios where replacement of parts is not possible. DLC coatings do not perform well when applied on soft substrates or under the presence of high contact loads. In addition, hydrogen-free DLCs are generally more wear-resistant than hydrogenated DLCs but require ambient humidity for good tribological performance. 2D layered materials can be used to achieve very low COFs and significant wear resistance in certain scenarios but do not perform well under changing environmental conditions. For instance, MoS₂ oxidizes at high temperatures and loses its lubricious capabilities under ambient conditions, whereas graphite requires a certain level of humidity to provide effective lubrication.

The discovery of a solid lubricant with outstanding capabilities of friction reduction and wear resistance over a wide range of operating conditions (including but not limited to temperature, humidity, contact load, and sliding/rolling speed) is the holy grail of solid lubricant research. A more realistic short term goal continues to be the combination of different solid lubricants, thus forming hybrid/composite solid lubricant systems that blend the advantages of their individual constituents [156, 157], which could be enhanced by chemical functionalization. Further, solid lubricants can be combined with surface texturing to alleviate their inherent short-comings, as discussed next.

4. Synergetic Effects between Solid Lubricants and Surface Textures

4.1 Soft Metals and Surface Textures

As outlined in Section 3.1, some metals can be used as solid lubricants because of their inherently low resistance to shear, particularly since they soften at elevated temperatures.

However, one challenge with leveraging this property for lubrication is that the material is easily removed by shear, leading to short useful lifetimes. This issue can be partially overcome by combining soft metals with hard textured surfaces to trap the softer material such that it can be released during sliding to lubricate the contact.

Several different soft metals have been studied in combination with textured hard surfaces. The most commonly reported metal used in combination with texturing is silver (Ag). Silver has been used for many years as part of composite coatings in which it is believed to segregate at higher temperatures to provide a lubricious film for low friction sliding [43, 158-160]. Silver has been applied as a solid lubricant to textured hard surfaces either on its own [161, 162] or in combination with MoS₂ [163, 164]. Other metals that have been used as solid lubricants on textured surfaces are copper (Cu) [165], indium (In) [166, 167], magnesium (Mg) [168], or multiple metals combined in Sn-Ag-Cu [169-171]. Textured hard surfaces have also been embedded with soft metal-containing inorganic compounds, including Ag₃VO₄[172] and BaSO₄[173].

Metallic solid lubricants have been deposited onto various hard materials and coatings with surface textures, including steel [164, 169, 174, 175], WC [165], VN [172], Ni₃Al [170], TiN [166, 167], TiAlCN [163], Ni [161] and Ta [162]. Textures in the form of dimples/pores [162-164, 166, 167, 169, 172, 173] or grooves [170] have been studied. The solid lubricants were used to fill the textures with lubricious material. Textures have been filled with metallic materials using various techniques, including sputtering [162, 163, 166, 167], electrodeposition/electroless plating [161, 164], hand burnishing [172, 173], spraying followed by grinding and polishing [165], or additive manufacturing of the material in powder form [169, 170].

The resulting surfaces have been then tested for their friction and wear properties using ball, pin- or ring-on-disk tribometers. As discussed later, many of these studies have been
performed at high temperatures to best leverage the lubricious nature of metals when they
soften. Several studies have also explored the effect of texture size and distribution on the
surface and shown that performance improves and then deteriorates with increasing dimple
or groove density [161, 162, 164, 167, 170]. This observation is attributable to the fact that
higher texture densities provide more lubricating material, but there is a limit to this benefit
as the surface approaches a flat surface from which the solid lubricant is easily removed
during sliding.

Studies often report improved friction or wear achieved through the combination of textures and metallic solid lubricants. The most commonly cited mechanism to explain this synergy is that the textures act as lubricant reservoirs that release lubricious material to the sliding contact or to the counter-body gradually during sliding [161, 163, 164, 166, 167, 169]. An illustration of dimples acting as reservoirs for silver is shown in Figure 7. Gradual release of metallic solid lubricants has been proposed to have a secondary benefit for self-healing. Specifically, it was reported that Sn-Ag-Cu can spread slowly from textures across the surface and fill cracks formed during sliding [169]. In most cases, this process is said to be facilitated by high temperatures that help the metallic solid lubricant flow or facilitate segregation of composite materials into lubricating metallic clusters [172]. It has also been proposed that textures can provide more surface area for the solid lubricant to interact with than a flat surface, thereby improving the adhesion of the lubricant to the surface and improving wear [172]. Lastly, it has been proposed that metal-based solid lubricants may react chemically with the counter-bodies or chemical species in the environment to form compounds that facilitate sliding [167, 170, 173].

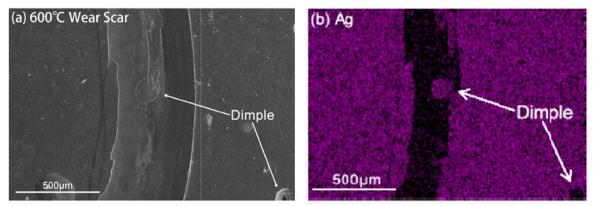


Figure 7: (a) SEM and (b) EDS images of a textured Ta-coated steel surface that has been covered with Ag after sliding against a silicon nitride ball at 600°C. Although the Ag is easily removed from the wear track, the solid lubricant remains in the dimples where it can replenish the interface during sliding. Reprinted (adapted) with permission from [162].

As mentioned above, metallic solid lubricants are often considered for high temperature applications because metals soften with increasing temperature. Most studies of Ag or Agbased solid lubricants report that friction decreases with increasing temperature [161-163, 169]. This decrease in friction with silver-filled textures has been observed up to 700°C, as illustrated in Figure 8 [161]. Similarly, friction was lower on a textured surface with BaSO₄ solid lubricant than without up to 800°C [173]. However, these solid lubricants may not provide any benefit over a texture-only surface at lower temperatures such as those found under ambient conditions [162]. In contrast, indium has a much lower melting temperature that the other materials discussed so far and it was reported that indium sold lubricant improved friction from room temperature up to 450°C, but then had an adverse effect on tribological performance from 450 to 1200°C [166]. Coatings performing well across a wide temperature range can be achieved by combining soft metals with layered materials. Ag was combined with Mo/MoS₂, such that the MoS₂ facilitated sliding at lower temperatures and silver or silver molybdates enabled easy sliding at high temperatures [163].

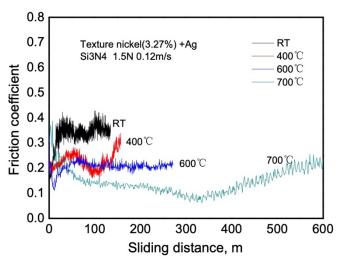


Figure 8: Friction coefficient for silver coated textured nickel at room temperature (RT) and three elevated temperatures. Reprinted (adapted) with permission from [161]

4.2 PTFE and Surface Textures

To combine PTFE and surface textures, most studies have used smooth PTFE counter-bodies rubbing against textured specimens. To understand the synergy between PTFE and textures combined in this way, it is necessary to first discuss the influence of stochastically rough surfaces on transient and steady state wear of PTFE [176]. For steady state conditions reached after the formation of a thin and homogenous transfer film, Burris and Sawyer showed that the wear performance of PTFE nano-composites did not depend on the roughness of the counter-body [104]. Li et al. studied the effect of the grinding orientation on the ability to form low-wear transfer-films. They demonstrated that homogeneous and complete transfer-films were formed when the sliding direction was aligned perpendicular to the grinding direction. For the parallel orientation, lumpy and discontinuous films with a poor wear resistance were observed. It has been also shown that the underlying surface roughness influenced the adhesive strength between the formed transfer layer and the substrate [177].

These observations were supported by early studies conducted by Laux and Schwartz for PEEK films [176].

To assess the influence of substrate roughness on the formation of ultra-low wear transferfilms, Ye et al. systematically studied the effect of the peak height (varying the arithmetic surface roughness R_a while keeping the skewness constant) and valley depth (varying the skewness while keeping Ra constant) on film formation [178]. Depending on the counterbody's roughness, two different running-in behaviors and film formation mechanisms were observed. In the case of counter-bodies without pronounced protruding surface asperities (Figure 9 (a)), the formation of the wear-resistant tribo-film was initiated by nano-scale lubricious wear debris deposited on smooth plateau regions. These individual areas grew over time to form continuous films. In this case, the valley depth was of minor importance for the overall film formation. For rougher counter-bodies (Figure 9 (b)), the polymeric counterbody initially abraded the substrate's highest asperities thus inducing surface topographies with preferential orientation. Nano-scale wear debris, which is generally beneficial for transfer-film formation, was deposited in the valleys, becoming nucleation centers for film formation. Specifically, valleys orientated perpendicular to the sliding direction helped to initiate and establish tribo-film formation [178]. The results indicate that the directionality of the surface topography affects the ability and efficiency of low-wear transfer-film formation. This is important for the use of PTFE with surface textures, which usually are inherently directional.

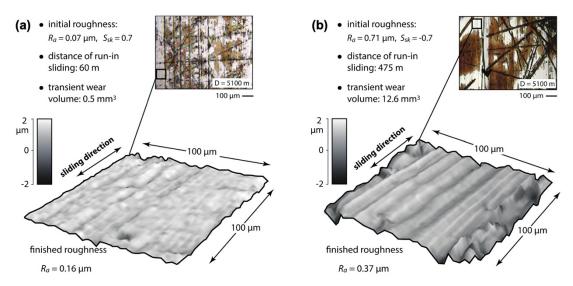


Figure 9: Measured surface roughness, distance of run-in and transient wear volume for α -alumina PTFE nanocomposites for a (a) smooth and (b) rough counter-face. Reprinted (adapted) with permission from [178].

Surface textures have been used to improve friction and wear of PTFE nano-composites. Most of these employed hard textured substrates such as stainless steel [179-183], bronze [184-186], silicon [187] or laminated ceramics [188, 189], which were rubbing against an untextured PTFE nano-composite. Surface textures were mainly produced by LST using lasers with pulse durations ranging from micro- [179, 181, 184-186] and nano- [188, 189] to pico- [182] and femtoseconds [180, 187]. Regarding the geometry of the textures, research effort has focused on dimples [179, 181-183, 185, 186, 188, 189], unidirectional grooves [180], grid-like textures, and circumferentially or radially aligned grooves [184]. Moreover, individual studies can be found in which textured wear-resistant hard coatings [190] or laminated ceramics [188, 189] were filled with PTFE solid lubricant via electrophoretic deposition [190] or spray coating [188, 189].

In the case of an untextured PTFE counter-body (mainly pins) sliding against textured substrates (usually metallic), significant contributions have been made by Ye et al. [179-181]. In a systematic study, dimples with variable diameters (20 - 1200 μ m), depths (5 - 18 μ m)

and densities (0.02 - 0.3) on stainless steel substrates were characterized in terms of their ability to form stable, continuous and low-wear transfer-films. Textures with large diameter, low depth and low area density were shown to be beneficial for the formation of stable transfer-films, thus reducing wear volume and wear rate (Figure 10 (a-c)). However, it must be emphasized that the best surface texture just reached the wear performance of the untextured reference but did not outperform it [179].

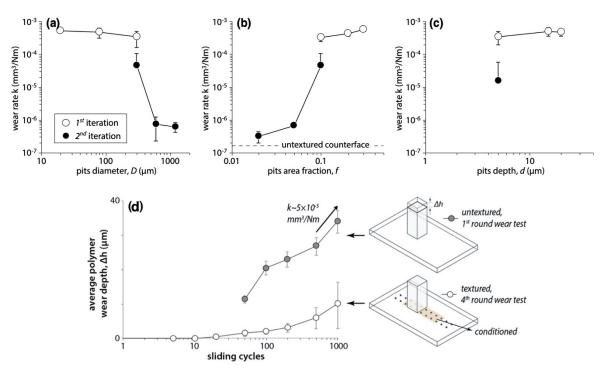


Figure 10: Dependence of wear rate on dimple (a) diameter, (b) density and (c) depth for α -alumina PTFE nano-composites. Figure from [179]. (d) Comparison of the average wear depth measured for α -alumina PTFE pins when rubbed against a reference steel substrate (untextured and unconditioned) and a pre-conditioned steel substrate (texture combined with beneficial transfer film). Reprinted (adapted) with permission from [181].

The full potential of textures to improve the system's wear performance (substrate and PTFE counter-body) was demonstrated in "swapping" tests (Figure 10 (d)). In these tests, homogeneous and stable transfer films were generated on different textures. Once formed, the worn polymeric counter-body was replaced with a fresh one to evaluate the performance of this pre-conditioned system (surface texture with stable transfer-film). For these pre-

conditioned systems, the wear rates of the polymeric counter-body and the textured steel substrate were reduced by 69 and 550%, respectively [181]. The low wear rates were traced back to improved ability to store lubricious wear debris in the textures, which then acted as debris reservoirs thus further promoting the formation of the transfer film. A reduction of wear debris' size and fewer cracks were observed, both of which likely contributed to the superior wear performance. The effects of area density and depth on wear were also reported in a recent study of Ding et al. [182]. They observed that shallow dimples with low area densities demonstrated enhanced film formation ability, thus compensating for negative edge effects that increase abrasion (Figure 11). Although the experimental trends for area density and dimple depth reported in both studies were consistent, they contradicted each other with respect to the effects of dimple diameter. Ye et al. reported the best performance with larger diameters of about 1200 µm, while Ding et al. found the best behavior for considerably small diameters of about 20 µm. These differences may be explained by differences in the PTFE filler material (α-alumina nano-particles for Ye et al. versus glass-fibers for Ding et al.) and experimental conditions such as contact pressure (6.4 MPa in case of Ye et al. and 40 MPa for Ding et al.) and sliding velocity (0.05 mm/s for Ye et al. and 0.12 m/s for Ding et al.).

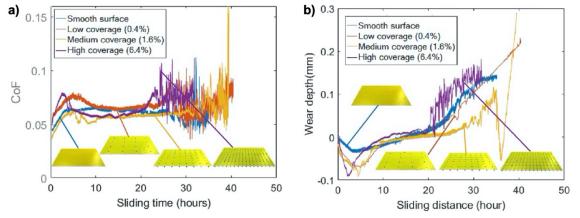


Figure 11: Temporal evolution of the (a) COF and (b) wear depth for narrow and shallow dimples having different area densities ranging from 0.4 to 6.4%. Reprinted (adapted) with permission from [182].

In an innovative study, Wang et al. recently electro-deposited MoS₂/PTFE mixtures (PTFE content between 0 and 20 wt.-%) in textures fabricated on a laser-cladded, wear-resistant Fealloy [190]. The best frictional performance was found for a powder mixture containing 10 wt.% of PTFE with a texture density of 20%. Although this was an interesting approach, the underlying friction and wear mechanisms are not yet completely understood. Moreover, no detailed analyses of the wear rates, transient wear volumes and formed tribo-films have been conducted, which encourages further research in this direction.

In contrast to the beneficial effects of dimples, detrimental effects have been observed for groove-like textures that considerably increased wear rates and transient wear volumes for un-conditioned systems [180]. This was correlated with a decreased ability to store debris in the grooves and accelerated abrasion on the counter-body. Increased wear rates were also observed for grid-like textures as well as circumferentially and radially aligned grooves.

In addition to the observed effect on wear, textures rubbing against PTFE counter-bodies may also lower the COF (reduction on the order of 10%) [183, 184] and improve the energy efficiency in ultrasonic motors (improvement by about 30%) [185, 186]. These improvements were explained by the storage of lubricious wear debris in textures, thus reducing abrasion and adhesion due to the discontinuous contact area.

4.3 Diamond-like Carbon and Surface Textures

The possibility of improving the tribological properties of DLC by surface texturing has raised much interest in recent years. In fact, the overall idea of combining surface texturing with coatings having low elastic modulus-to-hardness ratios (E/H) was proposed more than 25 years ago by Suh and colleagues [64]. They proposed to combine textures that reduce the

ploughing component of friction (by trapping wear debris) with thin coatings with low E/H ratios, such as some ceramics and polymers, that could significantly reduce the deformation component of friction by decreasing the amount of plasticity. However, it took almost ten years until the first attempts to combine surface texturing and DLC were carried out [61, 191]. One aspect not addressed in Suh's work was that surface texturing also decreases the real area of contact, which helps to reduce the adhesion component of friction. This is particularly important for DLC films due to their high surface energy [192].

One complex aspect of combining surface texturing and hard coatings such as DLC concerns how surface texturing affects the stress distribution on the coated surface. Gong and Komvopoulos proposed a model of a spherical asperity on a textured rigid and hard coating deposited onto a soft and compliant substrate, showing that the contact was formed by several micro-contacts. Textures with sharp edges induced significantly higher peak pressures at the borders of the micro-contacts. The increase in contact pressure was less for sinusoidal-like textures than textures with sharp edges. Residual stresses induced by sliding were more severe for textured than smooth coated surfaces, increasing the likelihood of crack propagation [193].

When Dumitru et al. first proposed to combine DLC and LST, they suggested two experimental approaches: direct and indirect structuring. Indirect structuring refers to a textured surface that is subsequently coated with DLC, while direct structuring refers to the texturing of the DLC coating itself [191]. LST has been the most widely used technique for DLCs, for both indirect texturing [117, 194-206] and direct texturing [118, 124, 207-211]. Since DLC films tend to be rather thin, conventional laser texturing techniques have not been very successful for direct texturing, leading to severe spalling [191]. Most successful reports

of direct texturing of DLCs have been carried out using femtosecond pulsed lasers (due to reduced thermal effects) [124, 209, 212]. One alternative is direct laser interference patterning (DLIP) allowing for a fast fabrication of periodic patterns on macroscopic areas based on the interference of two coherent laser beams [118, 192, 210, 211, 213, 214]. Conventional LST has been used mostly for indirect texturing [117, 191, 194-196, 198-203, 205, 215]. Nonlinear laser lithography, as a newly emerging technique capable to produce textures at a nanometric level, may be particularly interesting to texture thin DLCs [49, 216]. Lithography followed by either reactive ion beam etching [80, 217, 218] or chemical etching [61, 219], micro-machining [220] and polymer replication [221] have also been used for indirect texturing of DLCs. Direct texturing of DLCs using alternative approaches other than LST has been mainly achieved by masking the DLC coating and then etching with a reactive ion beam [222-224]. A few other approaches do not fit the classification as direct and indirect texturing, but rather refer to simultaneous texturing during coating deposition, such as the deposition of DLC through a mask, thus producing a textured coating [225]. Another example is the production of textured DLC using atmospheric pressure plasma enhanced chemical vapor deposition that naturally forms a stochastic texture composed of nanopillars during deposition [226].

For direct LST of DLCs, the occurrence of localized graphitization is an important issue. The reduced density of graphite is responsible for surface swelling, thus leading to the formation of textures composed of periodic patterns of hill-like micro-bulges. Localized nanocrystalline graphite bulges have been reported for both a-C and ta-C films after LST. The textured region had a porous structure with larger grains than the amorphous and denser untextured film [207, 208, 210-212]. In general, the laser fluence determines whether graphitization or ablation

occurs. Graphitization of DLC films starts at lower fluences, accompanied by surface swelling due to reduction of the material's density, thus creating localized graphitized bulges as texture [118, 207, 227]. For DLIP, graphitization occurs only at the regions of maximum interference [192, 208, 210, 211]. For higher fluences, which can be obtained by increasing either the energy density or the number of laser pulses, ablation dominates, thus locally removing the carbon film to generate grooves or dimples [124, 228]. For DLIP, ablation occurs at the regions of maximum interference, with significantly thinner films remaining [211]. If excessive fluence is used, the film can be completely removed in the regions of ablation and the substrate can be affected as well [212].

4.3.1 Direct Structuring of DLC

The first experimental findings of improved frictional performance due to direct laser texturing of DLCs under dry conditions were reported for hard ta-C films deposited on steel surfaces. Roch et al. suggested that texturing of DLCs could only reduce friction when the fluence was above the ablation limit, leading to well-defined grooves or dimples. For lower fluences, increased friction for textured DLCs was attributed to a reduced hardness due to graphitization and increased plastic deformation [211]. However, in subsequent work, Roch et al. measured lower friction for DLC coatings on Si with a texture composed of graphitized bulges when compared with smooth DLC. These findings were traced back to a reduced contact area, and thus lower adhesive forces. The authors argued that beneficial effects of the reduced contact area probably overcompensated for the lower hardness due to graphitization [192]. In a follow-up work, the authors proposed that graphitization width could be controlled with laser fluence. The COF was more stable for DLC with graphitized bulges than smooth

DLC. This was attributed to entrapment of wear particles by the textures, since for smooth surfaces the debris can temporarily participate in the contact, affecting the ploughing component of friction. Regarding texture periodicity, the smallest period resulted in the highest COF (0.152 compared to 0.09 for untextured DLC), whereas patterns with larger periods considerably reduced friction (0.057). These findings were explained based on the formation of very thin thermally-induced graphitized layers for patterns with short periods (below 5 μ m), thus increasing plastic deformation. When the graphitic layers were removed by a fluoride-based plasma etching, friction decreased to values below the untextured DLC, probably due to reduction in contact area and the entrapment of wear debris [210].

Despite the reduction of surface hardness accompanying graphitization, when AFM was used to measure directly textured ta-C films on steel substrates, friction in the localized graphitized bulges was lower than outside the bulges. This implies that graphitization does not necessarily lead to increased friction for DLCs, probably because graphite is a good solid lubricant. Some friction reduction was also observed during scanning outside the graphitized bulges when compared with the untextured films, which was associated with transfer of graphitic, lubricious material to the original surface [208].

Similar effects of reduced friction on graphitized bulges have been observed for direct texturing of a:C films on silicon wafers. In this case, texturing reduced friction when rubbing the DLC-coated surface against a steel counter-body under different normal loads and the worn surfaces showed a higher degree of graphitization for the textured films. The authors claimed that the adhesion component was reduced at low normal loads because the contact area of the graphitized bulges was smaller than for smooth DLC. For higher normal loads,

parts of the graphitized bulges were worn, transferred into the contact and spread over the sliding surfaces as a tribo-layer, reducing ploughing and friction [207].

Diamond-like nano-composite (DLN), a-C:H, Si:O films on Si substrates, have also been textured by direct LST. DLNs can be regarded as two atomic-scale networks (diamond-like carbon a-C:H network and glass-like a-Si:O network), the combination of which reduces internal stress and improves adhesion compared to a-C:H films. The friction force measured by LFM inside the graphitized bulges were significantly lower than the untextured surfaces (Figure 12 (a) and (b)). Also, force-distance curves were different inside and outside the bulges. Figure 12 (c) shows that the pull-off force was significantly higher on untextured DLC (original film) than inside the bulge, attributed to an increase in hydrophobicity due to air cushioning liquid droplets on top of the bulges. Due to the presence of humidity, the predominant component of the pull-off force was due to the water meniscus between the AFM tip and the film surface [212].

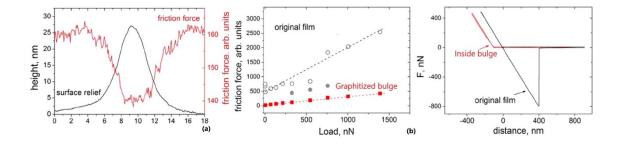


Figure 12: LFM measurements: (a) scanning across a graphitized bulge; (b) friction force as a function of load along a graphitized bulge (squares) and the original film surface (circles); (c) force-distance curves inside and outside the bulges. Reprinted (adapted) with permission from [212].

For direct texturing of DLCs, the height of the graphitized bulges is typically between 10 to 250 nm. For heights around 10 nm, no beneficial effect of surface texturing was observed, probably because the height was the same magnitude as the surface roughness of the film [124]. For bulges with heights around 100 nm and above, beneficial effects of texturing were

often observed [207, 208, 210, 212]. The width of the bulges changes with the laser fluence, varying from nanometers [118, 192, 209, 210] to a few micrometers [208, 211, 212]. The main effects of the spacing between bulges are the change in real contact area and the amount of graphitization inside and outside the bulges. A compromise between both effects needs to be achieved to optimize the friction and wear behavior. However, due to the difficulty in calculating the area coverage of the texture when it is composed of bulges, none of the published studies reported numerical values of the texture percentage when surface texturing was carried out below the ablation threshold of DLC.

Besides LST, photolithography followed by reactive ion beam etching has also been used to texture DLC films, which changes the topography but does not lead to structural changes in the film. For instance, photolithography was used in magnetic recording heads to texture DLC-coated sliders with 9 nm laser bumps on the landing zone, which presented low and uniform friction and stiction forces even after 40,000 cycles [80].

When DLC was deposited by physical vapor deposition onto WC-Co tools, a textured DLC film containing circular pillars with height of 300 nm was produced [225]. To explain a reduction in ploughing due to the reduced apparent contact area for the textured surface, the authors suggested that debris twice the size of the apparent contact area can detach from the surface, whereas debris at the center of the contact is more likely to get trapped and plough the surfaces [229]. Reduced wear and more stable friction values throughout the test were demonstrated due to entrapment of wear debris outside the pillars. Moreover, a reduction in real contact area increased the real contact pressure and, therefore, wear. To achieve stable friction and low wear, textures should efficiently eject wear debris from the contact interface and reduce the generation of wear debris, without substantially increasing stress

concentrations. For textures containing pillars, smaller diameters give lower coverage areas, resulting in higher contact pressures. The best compromise was found for a pillar diameter of 50 μ m and distance between the pillars of 15 μ m [225].

4.3.1 Indirect Structuring of DLC

For the majority of studies, texturing has been carried out prior to DLC deposition [117, 191, 194-203, 205, 217-221, 230, 231]. For titanium alloys, He et al. found lower COFs during running-in for laser-textured specimens, which was attributed to smaller real contact areas. Further, textured DLC showed more stable COFs after running-in, which was correlated to the entrapment of wear debris. The lowest friction was found for the highest area coverage because the entrapment of wear debris and reduction of contact area were more significant (Figure 13 (a)). Surface texturing also reduced the wear rate of DLC, except for the highest area coverage (Figure 13 (b)). Another observed effect of texturing was more intense graphitization on the worn surfaces due to sliding [198]. Smooth DLC exhibited graphitization induced by sliding, as normally reported for DLCs [116], but to a much lesser extent than textured DLC. The authors suggested that higher contact stresses for the textured DLC caused additional tribochemically induced graphitization. For high area coverages (44%), the wear rates were higher than for smooth DLC, probably due to the reduced bearing capacity and excessive softening resulting from the graphitized layer [198]. For DLC coatings doped with tungsten (W-DLC), Arslan et al. found that texturing of a hardened steel substrate led to reduced friction and entrapment of wear debris [205]. However, contrary to the suggestion in He's work, Raman spectroscopy revealed higher graphitization for the smooth DLC than for the textured DLC. The authors hypothesized that the presence of large wear debris in the contact could intensify the graphitic transformation since contact should occur between the large debris and the moving surfaces, leading to high contact pressures and thus more graphitization. For textured DLC, the textures remove the wear debris from the contact, reducing graphitization. The difference between these two studies may lie in the texture depth, which was substantially larger in He's work (25 μ m) [198] than in Arslan's work (6-8 μ m), resulting in more intense stress concentrations [205]. Further, W-DLC is harder than undoped DLC, and the film was deposited onto a substantially harder substrate (hardened steel compared to a titanium alloy) in Arslan's work [205].

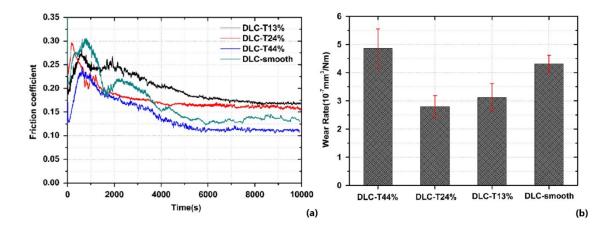


Figure 13: Dry reciprocating ball-on-flat tests of indirectly textured DLC on titanium substrates: (a) COFs; (b) wear rates. Reprinted (adapted) with permission from [198].

Improved friction and wear were found when circular nanopillars were obtained via texturing before DLC deposition by colloidal lithography with silica particles and subsequent plasma deposition of DLC. The authors traced their positive results back to less debris participating in the contact due to entrapment of wear debris and reduced real contact area. However, they found higher wear rates for textured DLC under higher normal loads, probably due to higher contact pressures [217]. A similar approach employed mask-less etching to produce nanowells (inverted pyramids) and nanodomes on Si surfaces prior to the deposition of a-

C:H films. Reciprocating ball-on-flat tests showed no beneficial frictional effects, but wear of the textured DLC was substantially lower than for smooth DLC. The authors hypothesized that surface texturing changed the distribution of internal stresses during the deposition of the DLC film [231].

Some studies reported that the synergy between texturing and carbon films was not in terms of texturing improving the tribological performance of DLC, but DLC helping to improve the textures' useful life due to reduced wear. Tani et al. verified that DLC increased dimple life from 20 to more than 8,000 cycles with only minor scratches [230]. Grewal et al. fabricated textures mimicking butterfly wings on Si, subsequently coated with DLC and fluorine-incorporated DLC (F-DLC). For uncoated Si, all textures showed higher friction than the reference, probably because texturing increases hydrophilicity for hydrophilic surfaces [79]. DLC films tended to reduce adhesion and friction, in particular F-DLC [218]. Wang et al. proposed the fabrication of hard, flexible and superhydrophobic DLC films via replicating the texture of lotus leaves followed by electrodeposition of a metallic layer. The textures featured circular pillars with average diameters of about 5 to 10 µm and heights of about 10 to 20 µm. Friction was slightly higher for textured DLC than for smooth DLC, probably due to increased ploughing. The durability of the superhydrophobic surfaces obtained after texturing was excellent due to the presence of DLC and the entrapment of wear debris [221].

Texturing can also fracture long chips that form when cutting soft metals into smaller sized chips. This is beneficial because long chips tend to adhere to the cutting tool, increasing tool wear and reducing machining performance. Texturing DLC-coated tools with wide (20 to 50 µm) and deep (5 µm) grooves has been shown to fracture the chips generated during cutting

of aluminum, thus preventing the formation of adhesion layers and reducing cutting forces [197, 219]

Some of the above-mentioned studies have emphasized the importance of texturing for adhesion between the DLC film and the substrate. The adhesion of DLC films may be poor for relatively soft substrates such as carbon steel due to the generation of large residual stresses as a consequence of the hardness difference between coating and substrate [123]. Liu et al. laser-textured steel followed by the deposition of multi-layer Cr/CrN/DLC films. Less flaking off of the DLC film deposited by a vacuum arc system was observed for lower area densities, for which the film filled the textured surface without spalling. Scratch tests verified improved adhesion [200]. Changes in the electric field distribution leading to ion energy variation during the DLC deposition might have increased the amount of sp³-bonds (confirmed by Raman spectroscopy) and released internal stresses. Textured coatings produced smoother wear tracks, with debris filling the textures, whereas smooth DLC showed grooving with flaking fragments.

However, studies showing no beneficial effect of texturing of DLCs have also been reported. In one of the pioneering attempts to combine surface texturing and DLC, Pettersson and Jacobson found higher friction and wear for textured DLCs [61]. Since the textures were produced by photolithography and presented rather sharp edges, they attributed the adverse performance to a possible scraping of protective tribo-films by the texture edges. DLC films deposited onto stainless steel and textured by fs DLIP showed COFs and wear track widths similar to untextured DLC films [118]. The authors hypothesized that, during sliding, the thin graphitized layer resulting from DLIP filled the valleys of the bulges so that the textures became too shallow to affect friction and wear.

Another unsuccessful attempt was to improve the adhesion of DLC films to polymeric substrates via surface texturing. For a-C:H-coated PEEK, surface textures did not affect the adhesion between the DLC film and PEEK, but reduced the scratch resistance, which was attributed to increased deformation of the soft substrate. Regarding the temporal evolution of the COF, the textured DLC initially had lower friction, probably due to the trapping of wear debris and reduced contact area. However, as the test evolved, both smooth and textured DLC coatings exhibited intense delamination due to the large mismatch in hardness between substrate and coating. Therefore, surface texturing was not able to sufficiently improve the durability of hard DLC deposited onto soft PEEK [202].

For applications on soft substrates, an alternative to DLC has been graphite-like-coatings (GLCs). Like DLCs, GLCs exhibit good chemical inertness, low friction and wear rate, without resulting in large stress concentrations when deposited onto soft materials. Ren et al. textured PEEK prior to GLC deposition and verified by Raman analysis that the deposited film had similar graphite-like structures inside and outside the dimples. Untextured GLC films suffered from more spalling and wear, resulting in wear depths greater than the GLC film thickness that indicated the film was completely removed. In contrast, the textured DLC films exhibited narrower and shallower wear tracks [232]. As previously hypothesized by Arslan et al. for DLC films [205], the authors suggested that wear debris in the contact could induce graphitization [232]. GLC has also been deposited onto hard textured Si substrates, resulting in more stable friction and lower wear than smooth GLC [233].

4.4 2D Layered Materials and Surface Textures

2D layered materials are known to exhibit low-friction sliding between their weakly bonded planes of atoms. These materials have also been combined with texturing in an attempt to improve performance beyond what is achievable with coatings or texturing alone. The layered materials that have been most often combined with texturing are graphite, MoS₂ and WS₂. In all cases, this approach is implemented as recessed textures that are then filled with the layered material. The earliest paper reporting this approach was by Voevodin et al., who filled grooves on a TiC/DLC disk with MoS₂, see Figure 14 [234]. The goal of that study was to combine DLC, which performs well in high humidity conditions, with MoS₂ that performs well at low humidities. The textures were used to combine two different materials in order to achieve an adaptable coating that provides low friction across a range of humidity conditions [234]. After that study, the approach was not pursued again until the late 2000s, when a series of studies combined textures with MoS₂ [124, 235-239] or graphite [240]. However, it was not until after 2010 that the number of tribological studies exploring the combination of surface texturing and 2D layered materials increased dramatically.

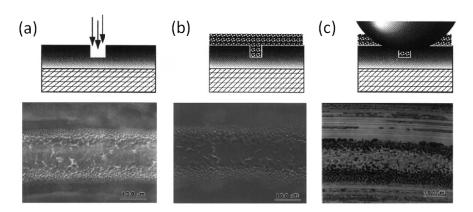


Figure 14: Schematic diagrams and surface topographies for (a) grooves produced on a Ti–TiC–DLC functionally gradient layer with nanocomposite TiC/DLC topcoat, (b) MoS₂ deposition via sputtering from a sintered target and (c) ball-on-disk tests where a steel ball is slid along the MoS₂ filled groove. The surface image in (c) corresponds to 1,600,000 sliding cycles at a contact pressure of 1 GPa in humid air. Reprinted (adapted) with permission from [234].

Surfaces that are both textured and covered by layered materials benefit from the advantages of both technologies, i.e. the surface textures trap wear debris and decrease contact area while the layered materials facilitate sliding. Further, nearly all studies that report synergy between texturing and layered materials where the textures act as reservoirs that continually resupply the contact with lubricious material. This mechanism is also described as the lubricant being stored in textures and then transferred to the untextured regions during sliding. Further, it has been suggested that texturing can improve the adhesion of the layered material coating to the substrate by mechanical engagement of the layered material in the textures, thereby increasing the area available for adhesion [237, 241]. The benefits of combining layered materials with textures are usually quantified using standard ball-on-disk or pin-on-disk test procedures to characterize friction, wear or wear life. Also, some studies specifically focused on machining measure performance improvements in terms of temperature increases, cutting forces and chip thickness ratio [239, 241-244].

The most common layered material used in combination with texturing is MoS₂. However, studies have been performed on WS₂ [241, 245-248], WS₂/Zr [245, 249], Cr-WS₂ [250], graphite/graphene/carbon-nanotubes (CNTs) [167, 233, 240, 251-255], CaF₂ [248, 256], and hBN [248]. Images of textured surfaces coated with MoS₂ and graphite are shown in Figure 15. To deposit the layered materials into the surface features, a variety of approaches have been used. The three most common approaches are burnishing [235, 237, 238, 246, 248, 251, 252, 257, 258], sputtering [124, 167, 234, 235, 241, 249, 259, 260], and manual application processes [236, 239, 242-244, 253, 261]. In addition, recent studies have used electrohydrodynamic atomization [247], atomic layer deposition [262], and electrophoretic deposition [254]. Studies have also shown that good adhesion is critical to the success of 2D

layered coatings embedded in surface textures. Consequently, some deposition processes use a binder or thin adhesion layer [236, 245, 249, 255, 263], or surface treatment such as anodizing [264] to improve coating-substrate adhesion.

Studies have been performed to compare the tribological performance of different layered materials on textured surfaces. One study compared MoS₂, WS₂ and WS₂/Zr, and showed that the latter was superior in terms of friction and wear [245]. MoS₂ in textures was also compared to graphite [248, 252], CaF₂ and hBN [248]. The performance of both graphite and MoS₂ were found to be superior to CaF₂ and hBN, but comparisons between MoS₂ and graphite yielded inconsistent results [248, 252, 255]. In another study, graphite was found not to decrease friction [167]. The inconsistencies in results from comparisons between graphitic materials and MoS₂ are likely due to the environment sensitivity of these two materials, since MoS₂ performs better in vacuum and graphite better in air. Moreover, studies have combined multiple solid lubricants, as done in studies of textures with MoS₂/graphite/Sb₂O₃ [235], Mo/MoS₂/Ag [163] and MoS₂/CNTs [253]. Lastly, studies have evaluated the effect of the initial size of solid lubricant particles before application to texture and found that nanoscale MoS₂ or graphite particles yield lower friction than the same material deposited in the form of microscale particles [253, 255].

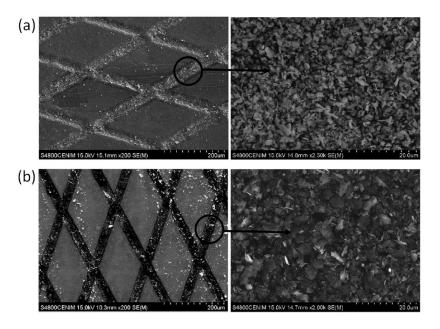


Figure 15: SEM images of textures on a Ti6Al4V alloy surface with (a) MoS₂ and (b) graphite embedded in the grooves. Close-up views of the MoS₂ and graphite flakes/particles inside the grooves are shown on the right. Reprinted (adapted) with permission from [252].

2D layered materials have been combined with textures on a variety of different surface materials, including textures imposed directly on a bulk material as well as textures on hard coatings. Bulk materials that have been textured and filled with 2D layered materials include steel [233, 236, 238, 248, 258, 265], Ti₆Al₄V [252, 257, 263, 266], TiAlCN [163, 267], WC/Co [239, 242, 243, 251, 261, 268], Ni-Cr-W-Al-Ti-Ag alloy [269] and Al-Si alloy [264]. In many cases, textures have been imposed on hard coatings, including DLC [124, 234], TiCN/TiN/TiC [167, 235, 240, 241, 244, 245, 247, 249, 259, 260, 262] and TaC [246]. Most of the surface features have been produced via LST. Other methods have also been employed such as micro-milling [264], micro-EDM [239], pulsed air arc treatment [236], or use of ceramic beads that are then removed via sonication to create voids [167, 240]. These technologies have been used to produce surface textures in a variety of shapes and sizes. The most common texture features used with layered materials are grooves and dimples. Grooves used for these applications are, on average, about 60 μm deep and 60 μm wide

(averages calculated from sizes reported in studies reviewed in this section). Grooves have been produced such that they lie either perpendicular [233, 242, 245, 248, 259-261] or parallel [234, 242] to the sliding path of the counter-body, or in a crossed pattern [252]. One study of the effect of the direction of grooves relative to sliding on friction reported that parallel alignment was preferred to perpendicular, possibly because more of the sliding occurred on the MoS₂-filled textures [262], and another showed that a 60° crossing angle is best for crossed grooves (see Figure 15) [252]. Almost as commonly studied as grooves are dimples [167, 235-237, 246, 251, 253, 257, 263, 265, 266, 269]. Dimples used with layered materials are, on average, about 15 μm deep and 70 μm wide. Other shapes, including rectangles, squares and triangles have also been explored [258, 264], but there is no evidence that these different shapes yield better performance. One study tested both grooves and dimples and found that, together, their performance was better than either texture individually [244].

A primary focus of many of the previous layered material/texture studies was optimization of the texture density. Most studies showed that the density was the dominant parameter in terms of friction, wear and wear life [258]. However, the magnitude of the coverage identified as best varied widely from study to study. Friction has been reported to either decrease with increasing density [237, 265, 266] or exhibit a non-monotonic relationship (decrease and then increase with increasing density) [233, 253, 257]. One study also reported that friction increased with increasing density [260]. Density has also been shown to affect wear life, with life either increasing with increasing density [237, 252, 257, 265] or first increasing and then decreasing with density [233, 235, 266]. An example of the latter is shown in Figure 16. These different trends do not appear to depend on texture shape, e.g. grooves vs. dimples, although they have been reported to depend on temperature [269]. Tribological performance

can vary non-monotonically with density because a higher density provides a more ample supply of solid lubricant, but high density also increases surface roughness and decreases the area of the hard surface available to support contact stresses and resist abrasive wear [235, 266]. Therefore, it is likely that studies showing consistent increase or decrease trends were only carried out over a limited range of density conditions such that they capture just one part of an overall non-monotonic trend.

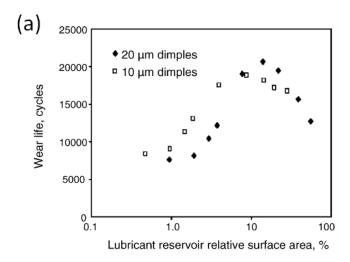


Figure 16: Wear life as a function of texture density for MoS₂ filled dimples as a function of dimple density for dimples of two different depths. Both depths have an optimum density for maximum wear life. Reprinted (adapted) with permission from [235].

5. Challenges, Opportunties and Concluding Remarks

As outlined in Sections 2 and 3, surface texturing and solid lubricants are excellent ways to improve friction and wear under dry conditions, but certainly have some limitations. In the case of surface textures, consensus regarding their effective use and complete understanding of the mechanisms involved have not been established. This is due to the complex interdependence of the tribological performance and the operating conditions as well as the insufficient use of simulation-guided texture optimization. To avoid wear-induced loss of functionality of textures and ensure good tribological performance over time, texture

parameters (e.g. width, depth, density) need to be optimized to effectively store wear debris, minimize acting residual and edge stresses.

Solid lubricants typically exhibit excellent tribological performance under specific testing conditions, but generally fail when used across a wider range of operating and environmental conditions (including temperature, humidity, contact load, and sliding/rolling speed). Moreover, solid lubricants with inherently low shear strength tend to be removed quickly from the surface they are intended to protect, which severely limits their useful lifetime. This issue is exacerbated by differences in the mechanical properties of the coating and the substrate material that may induce delamination. For hard coatings like DLCs, the key limitations are delamination when the coatings are applied on soft substrates or excessive spallation that occurs under high contact pressures.

The combination of surface textures and solid lubricants helps to overcome several of the aforementioned short-comings. Specifically, textures act as reservoirs that continually resupply the contact with lubricious material. This has been cited as the key synergy mechanism for textures with various solid lubricants, including soft metals and 2D layered materials. This mechanism has been supported by pre- and post-test surface characterization that demonstrated textures serve as reservoirs of solid lubricant. Textures filled with solid lubricant reduce friction and/or wear compared to smooth surfaces because low shear strength material is always present in the interface, and, more importantly, increase the useful lifetime of the system because the lubricious material is available longer.

Based on the widely accepted synergy mechanism described above, i.e. textures acting as solid lubricant reservoirs, optimization of tribological systems with textures and solid lubricants should focus on designs that enable the solid lubricant to be released from texture reservoirs at an ideal rate. If the lubricant is released too quickly, the coating will wear away

and the component lifetime will be short. If the solid lubricant is released too slowly, not enough lubricious material will be available to minimize friction and wear during sliding. This is clearly evidenced by studies that showed friction, wear or life vary non-monotonically with texture density, since the lubricant was released too quickly at very high densities and too slowly at very low densities.

The rate of lubricant release depends not only on surface design parameters, such as texture density, but also on operating conditions, such as load and speed. Therefore, the ideal texture/solid lubricant combinations will depend on operating conditions. This is likely why there is so much variation in the results of parametric studies of texture features including shape, width, depth and density. This observation suggests that optimization studies should include operating conditions as systematically varied parameters to fully characterize a given texture/solid lubricant combination. Further, it may be possible to combine ideal textures optimized for different conditions (e.g. loads, velocities, among others) to enable good performance across a range of operating conditions.

For smooth, untextured PTFE counter-bodies rubbing against textured substrates, the complex interplay between the texture design parameters, the temporal evolution of the surface topography and the ongoing tribo-film formation is essential. Pre-conditioned surface textures with large diameters, low depths and low area densities improve the ability of the surface to store lubricious wear debris, thus promoting the formation of wear-resistant, long-lasting transfer films. The rates at which beneficial tribo-films are formed and how surface textures contribute to that formation need to be studied systematically. This requires the use of advanced materials characterization and numerical simulation tools to assess the structural and chemical properties of the tribo-films in their early stages of formation as well as their evolution with time. This may lead to a better understanding of the underlying mechanisms,

thus paving the way for enhanced tribological properties achieved by combining surface textures and PTFE.

For hard DLC coatings deposited on softer substrates, both direct and indirect texturing can be used. Direct LST of DLC can lead to pronounced graphitization, reducing surface hardness but conferring lubricious characteristics. In this context, graphitization of DLCs may be also induced by tribochemical reactions during sliding. The amount of graphitization, which depends on the energy input during surface texturing (only for direct texturing) and on tribochemical reactions, is an important parameter to tailor in order to leverage beneficial effects of texturing and DLC coatings. In addition, textures reduce the real contact area, remove wear debris, improve adhesion between the substrate and DLC, and increase surface energy (hydrophobicity) of DLC. However, when textures are too shallow (of the order of the coating surface roughness), increased surface roughness, graphitization and reduction of load bearing capacity are not compensated for by removal of wear debris, thus worsening the tribological performance. For large area densities, textures can induce excessive contact pressures, reducing or even eliminating any beneficial effect. Some open questions remain regarding design of textures combined with DLC. For instance, there are contradictory results in literature regarding how the texture density affects graphitization during sliding. Application-oriented simulations that predict the amount of graphitization as well as the resulting stress distribution, combined with advanced surface characterization, should enable the positive synergy between surface texturing and DLC coatings to be maximized.

Generally, future improvements to the design of surfaces with textures and solid lubricants may focus on the solid lubricants themselves. There are a few studies that directly compare different solid lubricants with textured surfaces and those primarily focus on graphite vs.

MoS₂. With the emergence of new materials with great potential as solid lubricants, including MXenes and TMDs beyond MoS₂, there are now opportunities to explore a wider range of possible synergies with textures. Such studies could systematically vary both the solid lubricant and the texture design. Regarding solid lubricants, the use of hybrid/composite solid lubricants combining two or even more materials with solid lubrication ability thus blending the advantages of their individual constituents is a promising avenue for further research. However, as mentioned above, the results are likely to be condition-dependent, so multiple operating conditions relevant to the target application should be considered. For instance, graphite and hydrogen-free DLCs exhibit lower friction at high humidity while MoS₂ and hydrogenated DLCs show better friction behavior at low humidity. Similar dependencies are likely exhibited by other promising solid lubricant materials. Therefore, atmospheric conditions, particularly humidity and temperature, should be included in studies attempting to optimize solid-lubricated textured surfaces.

Summarizing, this review has demonstrated that the combination of surface textures and solid lubricants is a promising approach capable of inducing synergistic effects thus significantly improving friction and wear. Specific applications that can potentially benefit from the synergy between solid lubricants and surface texturing include orthopedic and other biomedical implants, microelectronic devices, magnetic storage discs, forming and cutting tools, smart screens, space applications, industrial seals, sensors, among others. The proper function of all these applications depends on the precise control of relative motion in dry conditions. Therefore, the combination of textures and solid lubricants may be a promising way to achieve surfaces with tunable friction and/or wear behavior. Finally, as outlined in this last section, we believe that there are still many opportunities for further improvement

and optimization of this technology to maximally leverage the synergy between textures and solid lubricants. Towards this goal, the use of complementary materials characterization and advanced numerical tools is expected to shed light on the underlying mechanisms, thus paving the way for significant improvements to the energy efficiency and useful lifetime of mechanical components.

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