

By carefully orchestrating atomic-scale interactions, one can coax hard surfaces to slide against one another with virtually no resistance.

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riction and wear affect almost all moving mechanical systems. By some estimates, nearly a quarter of the world's energy output is spent in overcoming friction.¹ About 70% of equipment failures are in some way attributable to wear and fatigue. Granted, strong friction forces are sometimes needed; brakes, tires, and continuously variable transmissions, for example, couldn't operate without them. But in many cases, alleviating friction would save energy, help the environment, and boost productivity.

Since prehistoric times, human beings have sought ways to minimize friction and its attendant ills. Before the 1990s, however, tribologists generally assumed that the friction coefficient—the dimensionless ratio of the friction force to the normal load across contacting surfaces—could never fall below 0.1 in most practical systems. The friction coefficient of wood sliding against wood is typically 0.2 or larger; for clean metal surfaces under dry conditions, it can be larger than 1.

A quarter of a century ago, theorists began to consider a curious and tantalizing idea: Through careful atomic-scale design, one can not only reduce friction in some tribological systems but make it vanish. That idea is now a reality. Superlubricity, or something very close to it, has been observed in various experimental systems.

In this overview we will concentrate primarily on superlubricity in systems exhibiting hard contacts—point and line contacts between stiff materials such as metals, ceramics, and hard coatings—operating under high normal loads and high sliding speeds. Hard contacts can be found in valve trains of thermal engines, ball and roller bearings, and gears; they arise in metalforming operations like forging and wire drawing and even in nanotribological settings such as atomic force microscopes and microelectromechanical systems. They can be distinguished from soft contacts by their contact pressures. Soft contacts such as those between polymers, elastomers, and biological tissues, including hip and knee joints—generally exhibit contact pressures of 10 MPa or less. (For more on the lubrication of soft contacts, see the article by Sabrina Jahn and Jacob Klein on page 48 of this issue.) Hard contacts, meanwhile, can exhibit peak contact pressures as high as 3 GPa.

Here we will examine hard contacts under high vacuum, in the presence of gas, and in the presence of liquid lubricants. (Some tribologists consider gases to be lubricants.) All those systems have been shown to exhibit some form of extremely low friction. But as we will show, the underlying physical mechanisms vary significantly.

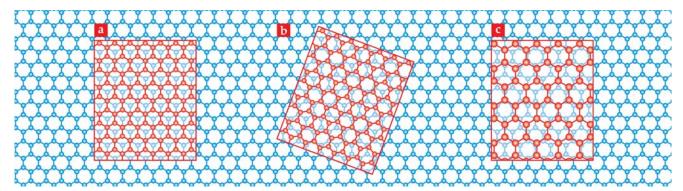
# Structural superlubricity

In the macroscopic sense, the friction coefficient of two sliding surfaces depends on the surfaces' roughness. But studies of atomically smooth surfaces under vacuum clearly illustrate that the true origin of friction is at the atomic scale. The friction force is the sum of atomic-scale forces—coulombic and

van der Waals interactions, ionic and covalent bonds, and so forth—after accounting for all possible interferences between those forces. Thus, under vacuum, friction depends heavily on the arrangement, be it crystalline or amorphous, of a material's surface atoms.

In the early 1990s, Motohisa Hirano, then at Japan's Nippon Telegraph and Telephone Corp, predicted that the friction coefficient should vanish for certain configurations of contacting crystalline surfaces.<sup>2</sup> He coined the behavior superlubricity, in reference to previously discovered dissipationless phenomena such as superconductivity and superfluidity. However, unlike superconductivity and superfluidity, which are highly temperature dependent and correspond to bulk phase transitions, superlubricity is driven entirely by surface properties. It requires atomically clean and smooth surfaces characterized by weak atomic interactions. Temperature is not a critical factor, but lower temperatures are thought to be preferable due to the reduced thermal vibration of surface atoms. As Hirano proposed it, superlubricity is strongly affected by environmental factors and can be obtained only under ultrahigh vacuum. (Interestingly, the largest experimentally measured friction coefficients are also observed under ultrahigh vacuum-between metal surfaces having strong atomic interactions.)

At its essence, Hirano's superlubricity stems from a mismatch between the atomic lattices of two contacting surfaces. To understand why, consider an atomically smooth and flat surface under vacuum. If the surface atoms are arranged periodically, the envelope of the surface energy resembles an egg crate. If two such "crates" are layered against one another, as in figure 1a, their wells and peaks will fit perfectly together, and the surfaces will resist sliding. More precisely, the surfaces will exhibit an intermittent stick—slip motion—and, depending on the sliding direction, possibly an associated zig-zag motion. The force required to initiate sliding will be high. (In atomic force



**FIGURE 1. STRUCTURAL SUPERLUBRICITY,** in which two dry crystalline surfaces exhibit a vanishingly small friction coefficient, is intimately linked to the relative orientation and spacing of the surfaces' atomic lattices. (a) When the two lattices are identical and share the same rotational orientation, the atoms of one surface can sit in the spaces between the atoms of the other, and the surfaces resist sliding. But if the lattices (b) are rotated relative to one another by an angle incompatible with their rotational symmetry or (c) have mismatched lattice spacings, theory predicts frictionless sliding, provided the surfaces are smooth and the atomic interactions are weak. (Adapted from ref. 10.)

microscopy, stick—slip motion has an advantageous quality: It can be exploited to generate high-resolution atomic-scale images.)

If one surface is rotated by an angle that doesn't correspond to a rotational symmetry, the crates no longer fit. The lattice misfits cancel the lateral forces in every direction, which results in zero net friction force. Rotated at such a misfit angle—say, 30° for a hexagonal lattice, as in figure 1b, or 45° for a square one—the friction coefficient in the sliding direction should effectively disappear. The rotation dependence is an example of what's known as frictional anisotropy. Another route to incommensurability is to pair two lattices having mismatched interatomic spacings, as illustrated in figure 1c. Recently, superlubricity triggered by frictional anisotropy has come to be called structural superlubricity.

Also important is the direction of sliding relative to the crystal orientation. Recent work shows that if two contacting surfaces are allowed to rotate freely, they will spontaneously adopt a rotational misfit angle and thereby minimize friction. In most cases, however, sliding surfaces are not free to rotate. In those instances, the vanishing of the force in a specific sliding direction can be accompanied by motion of areas of registry in directions orthogonal to the sliding direction.

Hirano wasn't the first person to theorize about frictionless sliding. In 1990 physicist Jeffrey Sokoloff predicted that "the frictional force generated when a crystal slides relative to the surface of an incommensurate crystal is . . . 13 orders of magnitude smaller than for the case of shearing of commensurate layers." But whereas Sokoloff's model was essentially one-dimensional, Hirano's described the effect in two and three dimensions. Hirano's work is also better remembered because it was the first to use the term superlubricity.

The works of Hirano and Sokoloff were essentially theoretical. The real challenge in proving or disproving the theories was a practical one: Extremely low friction coefficients are exceedingly difficult to measure. In typical measurements, the friction coefficient isn't smooth over time, in large part because of the springy action of the tribometers used to measure friction force. Even today, most tribometers can't accurately measure friction coefficients below, say, 0.001, except in gas-phase hydrodynamic-lubrication experiments where there is no solid contact. Hirano's own efforts to observe superlubricity in metals and sheet-like minerals known as mica didn't detect the

vanishing of friction but were convincing nonetheless: They showed significant dips in the friction coefficient at rotational misfit angles, which suggested the validity of Hirano's model.<sup>2</sup>

Recently, researchers have managed to overcome many of the technological difficulties Hirano faced. Sophisticated experiments on various systems, particularly materials made up of stacked, atomically thin sheets and known as lamellar compounds, have definitively confirmed Hirano's model.

# Superlubricity under vacuum

The first experimental demonstration of macroscale superlubricity was published in 1993 by a group that included one of us (Martin) at the École Centrale de Lyon. The group deposited a 1-µm-thick film of pure molybdenum disulfide (MoS<sub>2</sub>), or molybdenite, on a flat, highly polished steel substrate. A tribometer held a steel ball against the surface at a contact pressure of 0.5 GPa and measured the force required to slide the two objects past one another. The friction test was conducted in an ultrahigh-vacuum chamber at a gas pressure of 10 nPa.

After a brief run-in period, in which some of the molybdenite coating was transferred to the surface of the steel ball, the friction coefficient fell below 0.01, smaller than the measurement precision of the instrument. (The value 0.01 has come to be regarded as the standard threshold for superlubricity.) For all practical purposes, friction had vanished. At times, the calculated friction coefficient even fell below zero, a physically impossible value that reflects the noisiness of the signal and the difficulty of measuring vanishingly small friction coefficients, particularly in a vacuum chamber.

High-resolution transmission electron microscope (TEM) images of wear particles generated during the experiment, shown in figure 2, elucidate the underlying mechanism. Those images show that the molybdenite film organized into a stacked mosaic of 2D crystalline nanosheets, each aligned parallel to the surface. A fast Fourier transform revealed that each sheet was rotated roughly 15°—a misfit angle—from the one below it, in agreement with Hirano's theory.

The result was later confirmed by Akira Miyamoto (Tohoku University) and colleagues, who used computer simulations to model the evolution of an amorphous molybdenite film sheared between iron surfaces.<sup>5</sup> (Iron was used to mimic steel.) The simulated molybdenite film gradually crystallized and self-

organized into three stacked sheets whose lattices were rotated by angles similar to those observed in the experiment. As the layers emerged, the calculated friction coefficient fell below 0.01. Recently, superlubricity of lamellar molybdenite has also been demonstrated by a group at Peking University in China, who used in situ scanning electron microscopy and a silicon nanowire force sensor to sensitively measure friction forces.6

In 2004 a Dutch team led by Martin Dienwiebel demonstrated the superlubricity of another lamellar material: graphite. They used a nanotribometer that consisted of a graphite nanosheet, affixed to a small pin, sliding against a flat graphite surface under high vacuum.7 The setup allowed them to arbitrarily

choose the orientation of the nanosheet relative to the surface. For rotation angles near 0° and 60°—that is, angles compatible with graphite's sixfold symmetry—the researchers saw stick slip behavior and the friction force spiked, as shown in figure 3. But when the angle deviated by more than 10° or so from the angles of rotational symmetry, the system transitioned to smooth sliding, and the friction force dropped nearly to zero. The results provide elegant confirmation of Hirano's model of structural superlubricity. So, too, do experiments by a Tsinghua University group that report rotation-dependent superlubricity between a freestanding microscale graphite mesa sliding against a graphite surface.8

Recently, researchers at Tsinghua have shown structural superlubricity in graphene-coated silica nanoparticles sliding across graphene and hexagonal boron nitride substrates. The exceptionally low friction coefficients, as small as 0.003, could be attributed to the incommensurability of the randomly ori-

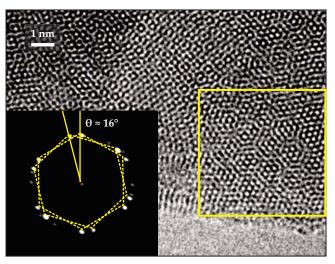


FIGURE 2. HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY reveals that a molybdenite coating sheared between steel surfaces organizes into stacked layers of sheet-like two-dimensional lattices. A fast Fourier transform (inset) of the region of the TEM image within the square indicates that the crystal's top layer is rotated by about 16° from the layer beneath it. Because that rotational angle is incompatible with the lattices' rotational symmetry, the friction force between the layers is vanishingly small. (Adapted from ref. 4.)

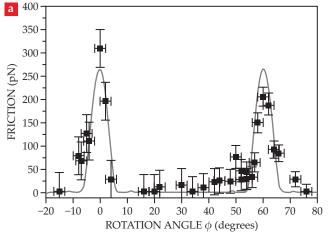
ented graphene nanograins.9 Likewise, a group at the University of California, Berkeley, showed that small graphene nanoflakes could slide freely against a graphene surface when the incommensurability conditions were met and temperatures were sufficiently low.10

At Argonne National Laboratory, researchers, including one of us (Erdemir), slid a graphene-coated surface against an amorphous carbon film and showed that the friction coefficient can fall below 0.01 if the interface is sprinkled with graphene patches and nanodiamond particles: The graphene patches wrap around the particles to form nanoscrolls, which act as miniature ball bearings.11 (The illustration on this article's opening page depicts the ef-

fect.) A Japanese team exploited a similar mechanism to achieve superlubricity between graphite flakes. 12 The team inserted monolayers of C<sub>60</sub> molecules, or buckyballs, between the graphite sheets. Because of the increased interplanar spacing and the ball-bearing behavior of the fullerenes, the friction force all but disappeared.

A wide range of other carbon-based materials have been shown to exhibit superlubricity under dry sliding conditions. In 2013 a Tsinghua team showed that the inner shell of a double-walled carbon nanotube could be effortlessly pulled from the outer shell, regardless of the length of the nanotube. 13 Other studies reported ultralow friction—corresponding to friction coefficients smaller than 0.1 but larger than 0.01—in carbon nanodiamonds and multilayer fullerenes, nano-onions, and other carbon-based structures.

Naively, one might expect that diamond crystalline interfaces could give structural superlubricity via rotational disorder.



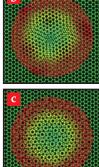
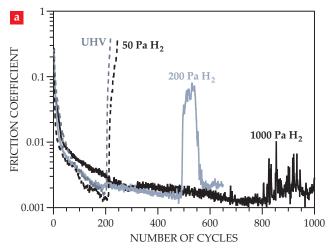
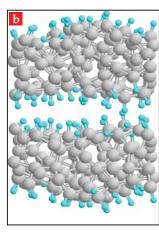


FIGURE 3. THE FRICTION COEFFICIENT of a graphite flake sliding across a flat graphite surface depends on the relative rotation angle between the surfaces' crystalline lattices. (a) Peaks of high friction observed near 0° and 60° correspond to angles of rotational symmetry. Away from those angles, where the lattices are incommensurate, the friction force effectively falls to zero. (Adapted from ref. 7.) (b-c) Computer illustrations depict the superposition of the two surfaces' honeycomb lattices, at relative rotation angles near 6° and 27°. In each drawing, one lattice lays flat and the other coats a hemispherical surface; red indicates increasing distance from the flat surface. (Adapted from ref. 9.)

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**FIGURE 4. HYDROGENATION OF AMORPHOUS CARBON SURFACES** reduces friction by weakening interfacial interactions. **(a)** When such hydrogenated surfaces slide against one another under ultrahigh vacuum (UHV), the friction coefficient initially falls below 0.01, the standard threshold for superlubricity. But the sliding action strips surface C–H bonds, and after about 200 sliding cycles, the coefficient jumps sharply. The system behaves similarly under a hydrogen partial pressure of 50 Pa. But when partial pressure increases to 1000 Pa, the system can remain in the superlubricity regime for 1000 or more cycles; the gaseous hydrogen helps replenish surface C–H bonds. (Adapted from ref. 15, J. Fontaine et al.) **(b)** In this simulated image of two hydrogenated amorphous carbon surfaces under 1 GPa contact pressure, C and H atoms are colored gray and teal, respectively. (Adapted from ref. 14, Y. Wang et al.)

But the unbonded electrons of the surface atoms give rise to strong interactions, and a key requirement of superlubricity is therefore unfulfilled. In theory, incommensurate hydrogenterminated diamond surfaces should give superlubricity under vacuum conditions, but due to the technical difficulty of preparing such surfaces under vacuum, the prediction has yet to be verified.

# **Hydrogen-mediated superlubricity**

Due to their light weight, low cost, and nontoxicity, carbonbased materials are commonly used to control friction and wear. In many carbon-based materials and coatings, the presence or absence of superlubricity hinges on the makeup of the surrounding gases in the test environment.

That's especially true for amorphous materials such as diamond-like carbon (DLC), whose individual atoms are mostly tetrahedrally coordinated but lack long-range symmetry. The surface carbon atoms in a DLC film are typically terminated with hydrogen atoms. The hydrogen termination is critical for superlubricity; it effectively passivates the dangling bonds of the surface carbon atoms. In the absence of hydrogen, the atoms' orbitals could hybridize, which would give rise to strong attractive interactions between opposing surfaces and, in turn, trigger strong adhesion and friction.

When hydrogen atoms adsorb on the surface, however, their electrical charge density shifts toward the underlying carbon, and the surface acquires a small positive charge. Such a dipole configuration at the sliding interface is thought to lead to repulsive forces between surfaces, which would undoubtedly reduce friction. Indeed, molecular dynamics and *ab initio* computer simulations have shown the existence of such repulsive forces among fully hydrogenated DLC surfaces. <sup>14</sup> Because the surfaces are not completely smooth, the friction coefficient cannot vanish. But it can become very small, on the order of 0.01

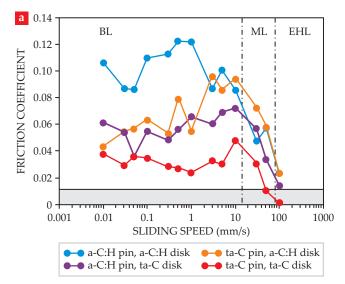
at the beginning of a sliding test.

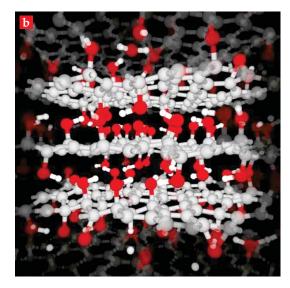
To ascertain the effect of hydrogen gas on the tribology of DLC coatings, a team at École Centrale de Lyon measured the friction of a bearing steel pin having a radius of curvature of 8 mm sliding across a flat sample of hydrogenated DLC, under various partial pressures of hydrogen gas.15 As shown in figure 4, under ultrahigh vacuum the friction coefficient rapidly decreased to the ultralow friction regime, down to about 0.007. But after 200 sliding cycles, it suddenly jumps to more than 0.7. Superlubricity was similarly short-lived when the hydrogen partial pressure was increased to 50 Pa. But at a partial pressure of 200 Pa, the system remained in the superlubricity regime for nearly 500 cycles; at 1000 Pa, superlubricity persisted for the duration of the test, 1000 cycles.

From triboscopic images, which show how the friction coefficient varies at different stages in each sliding cycle, it was clear that the jumps in the friction coefficient were correlated with the emergence of deep grooves and film failures, which in turn were likely due to adhesive or abrasive phenomena that removed bits of the DLC coating. The implication was that the severe shearing action severed carbon–hydrogen bonds at the surface, exposing dangling carbon bonds that triggered strong adhesion. However, those adhesive phenomena were curtailed in the presence of environmental H<sub>2</sub>, which rapidly replenished the surface with fresh C–H bonds.

A repeat experiment that substituted gas-phase hydrogen with molecular deuterium produced further evidence supporting that explanation. Posttest surface analyses performed using time-of-flight secondary ion mass spectroscopy revealed high concentrations of deuterium in the wear tracks left by the sliding contacts; that finding confirmed that deuterium had chemically interacted with the sliding surfaces. Similar tribochemical interactions occur in the presence of oxygen. But because oxygen is polar and very reactive, it leads to stronger adhesion, higher friction, and more extensive wear damage.

Superlubricity in DLCs can also be influenced by the material's own bulk and surface chemistry. <sup>16</sup> At Argonne, researchers used plasma-enhanced chemical vapor deposition to synthesize DLC films from increasingly hydrogenated gas-discharge plasmas. They found that the films synthesized from the most hydrogenated plasmas gave the lowest friction coefficients, between 0.001 and 0.005. Moreover, when hydrogen-poor or hydrogen-free DLC films were subjected to hydrogen plasma bombardment or treatment, those films could exhibit superlubricity even in inert gas environments. It has been proposed that, mechanistically, when a DLC film is synthesized from a highly hydrogenated gas-discharge plasma or subjected to hydrogen ion bombardment, it acquires a high density of hydrogenical chemical values and the superlubricity of hydrogenical companies.





**FIGURE 5. SUPER LUBRICANTS.** (a) The friction coefficient for an oleic-acid-lubricated pin-disk contact depends on sliding speed: It generally falls as the system transitions from a so-called boundary-lubrication (BL) regime to a mixed-lubrication (ML) regime and reaches a minimum in an elastohydrodynamic-lubrication (EHL) regime. When both pin and disk are coated in hydrogen-free tetrahedral amorphous diamond-like carbon (ta-C) rather than hydrogenated amorphous diamond-like carbon (a-C:H), the friction coefficient dips into the superlubricity regime (shaded gray) in the ML and EHL regimes, as a result of forces arising from hybridization and hydroxylation at the surface. (Adapted from ref. 18.) (b) Similar hydroxylation can be seen in lamellar graphene oxide, depicted in this simulated image. Here, carbon is gray, oxygen is red, and hydrogen is white. (Adapted from S. Kim et al., *Nat. Mater.* 11, 544, 2012.)

gen, as high as 40 atomic percent, in the bulk film and at the surface. Much like in the case of a hydrogen gas environment, the hydrogen serves to diminish the number of dangling bonds.<sup>17</sup>

# **Liquid-mediated superlubricity**

Superlubricity means something fundamentally different for wet, lubricated contacts than it does for dry contacts. It is conceptually more akin to superelasticity, superhardness, and superhydrophoby encountered in materials science: A lubricated system is said to exhibit superlubricity whenever its friction coefficient reaches values significantly lower than those typically encountered in pure sliding at low sliding speeds.

Friction in lubricated systems is commonly illustrated with a Stribeck curve, which plots the friction coefficient as a function of sliding speed or of some dimensionless parameter that includes the sliding speed. The Stribeck curves of lubricated systems tend to share some general features.

At low speeds, in what's known as the boundary-lubrication regime, the friction force is high and almost entirely the result of direct solid contacts between surface asperities. As the sliding speed increases into what's known as the mixed-lubrication regime, the surfaces partially separate, the role of the viscous liquid grows, and the friction coefficient begins to fall.

As the sliding speed increases further, the system enters the elastohydrodynamic-lubrication regime, in which a thin lubricant film completely separates the surfaces. The friction coefficient typically reaches a minimum of a few hundredths in the elastohydrodynamic-lubrication regime. At higher sliding speeds, the system enters the hydrodynamic-lubrication regime, where the friction force depends primarily on the viscous properties of the liquid lubricant and the friction coefficient increases with speed as a direct consequence of Newton's law. Stribeck curves can also be obtained for systems that exhibit a mixture of sliding and rolling. However, friction is generally

more severe under pure sliding, and it is more severe for reciprocal, back-and-forth sliding than for unidirectional sliding.

Superlubricity in lubricated systems is indicated by abnormally low friction coefficients that span multiple regimes in the Stribeck curve. Importantly, the low friction values must not be the mere result of, say, a change in lubrication regimes or a drastic decrease in contact pressure caused by corrosive wear. To qualify as superlubricity, wear should be minimal and the contact pressure should remain relatively high, up to 300 MPa.

Figure 5 illustrates an example from an experiment by a group that included one of us (Martin).<sup>18</sup> The figure shows Stribeck curves for four DLC contacts lubricated with oleic acid, a model molecule present in the chemical formula of glycerol monooleate. (Glycerol monooleate, or GMO, is a common additive to lubricants used in internal combustion engines.) All four Stribeck curves correspond to pure, reciprocating sliding of a carbon-coated pin against a carbon-coated disk. In one case, both the pin and the disk are coated with hydrogenated amorphous DLC (a-C:H). In another case, both are coated with hydrogen-free tetrahedral amorphous DLC (ta-C). In the remaining two cases, a-C:H and ta-C pins are paired with ta-C and a-C:H disks, respectively. Test results were obtained by decreasing the sliding speed gradually from 100 mm s<sup>-1</sup> in the elastohydrodynamic-lubrication regime to 0.01 mm s<sup>-1</sup> in the boundary-lubrication regime.

None of the contacts exhibit superlubricity in the boundarylubrication regime. But when both the pin and the disk are coated in ta-C, the friction coefficient is uncharacteristically low at all sliding speeds. In the mixed- and elastohydrodynamic-lubrication regimes, the coefficient falls below 0.01, the threshold for superlubricity.

A close look at ta-C's surface chemistry sheds light on the behavior. Synchrotron x-ray imaging reveals that under friction, the material's surface carbon atoms adopt a planar, graphene-

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like configuration, in which dangling electrons are free to bond with OH and H groups supplied by the oleic-acid lubricant. The effect of the OH termination is to increase the distance between the carbon atoms of the opposing ta-C surfaces and to provide enough repulsion to prevent covalent C-C bonds from forming across the interface. (There is some indication that friction also modifies the subsurface of the ta-C film, increasing the overall fraction of planar coordinated atoms relative to tetrahedrally coordinated atoms.)

Computer simulations indicate that the mix of C-H and C-OH bonds slightly increases the atomic roughness of the sliding interface, which offsets some of the benefits of the increased interfacial distance. But when substantial portions of the carbon surface are flat and graphene-like, the roughness effect is minimal.

Of course, the presence of a liquid at the interface precludes the complete elimination of friction, but extremely low friction values have been reported in the above and other lubricated systems over the past 10 years. The number of articles dealing with liquid-mediated superlubricity is increasing every year, and there is little doubt that superlubricity will soon be reached under boundary-lubrication conditions.

### The future of friction

As we've seen, there's more than one route to superlubricity: the incommensurate crystalline counterfaces of Hirano's structural superlubricity, the hydrogenated surfaces of H-mediated superlubricity, and liquid-mediated superlubricity. In every case, the phenomenon's origins can be traced to complex atomic and tribochemical interactions at the contact interface.

Admittedly, structural and H-mediated superlubricity have

limited potential for practical applications due to the stringent experimental conditions they require. But they could potentially exist in space applications, hermetically sealed computer hard disks, microelectromechanical systems, and other specialized systems. Thermal engines using hydrogen gas could also benefit from hydrogen-mediated superlubricity. Liquid-mediated superlubricity, on the other hand, could save significant energy and have important practical applications in common systems, particularly one that many of us rely on every day: the automobile engine.

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