

Research Article

Transfer Layers: A Comparison across SWNTs, DWNTs, Graphite, and an Ionic Fluid

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Lubrication is the science of friction at moving interfaces. Nanomaterials acting as interfacial modifiers can minimize friction and thereby improve energy efficiency. To test this hypothesis, single- (SWNT) and double-walled (DWNT) carbon nanotubes and an ionic fluid are tested individually and compared to SWNTs and graphite as additives within the ionic fluid. The minimum coefficient of friction is correlated with the longest lifetime using a ball-on-disc tribometer, in air, at atmospheric pressure. Results are interpreted in terms of the nanotubes' mechanical properties and the formation of transfer layers upon the tribosurfaces.

1. Introduction

At the heart of lubrication, historically a very “mature field,” is material structure and dynamics on the nanoscale. Therein nanotechnology can contribute to designing new lubricants based upon fundamental principles. As interfacial modifiers, nanomaterials can either be used as additives in existing lubricants or be the foundation of entirely new lubricant systems. An area in particular need of new lubricants is near-earth orbiting and outer space mechanisms. Moveable systems for space-based applications include wheels, gyroscopes, solar array gears, antenna drives, actuators for panels, armatures, and latches. Contact stresses can be severe ($>10^6$ Pa) in addition to severe launch vibrations. Additionally maintenance-free lifetimes for these devices, given service difficulties need to be 10–20 years [1].

Space environments in general pose special challenges to lubrication, foremost of which is the vacuum environment, $\sim 10^{-7}$ torr [2]. Liquids are subject to evaporative losses, exacerbated by friction-induced heating. Secondly, wide temperature changes occur, from -120 to 150°C for satellites. Such swings can dramatically change liquid lubricant viscosities. Finally, if exposed, atomic oxygen can oxidize an

organic lubricant. In contrast solids are generally immune to such effects [3]. Solid lubricants, particularly those based on layered 6-H compounds such as graphite, WS_2 , or MoS_2 can form transfer layers upon the component surfaces [4–6]. These layers not only provide wear protection but also inhibit corrosion [7]. Moreover solid lubricants are better suited to high loads, while liquids can undergo rheological thinning, an aspect compounded by the aforementioned conditions [8]. With liquid lubricants, additives are required to provide the formation of protective films and modify the viscosity. Yet liquids can recover rapidly and are superior under high-speed operation.

Of particular advantage to space-based applications is that ionic fluids possess extremely low vapor pressures, are viable over a large temperature ranges, are nonflammable, and have excellent thermal stability and high electrical conductivity [9, 10]. Similar needs arise in MEMs applications for extended life in sealed packages or devices. Several ionic fluids were evaluated by spiral orbit tribometry (SOT) in vacuum and air. The SOT results along with their vapor pressures were reported [11, 12]. In SOT testing, ionic fluids appear to be the only class of base lubricating fluids that have superior lifetimes in vacuum than in air. For the two ionic

fluids evaluated in that study, lifetime improvements of 6.1 and 4.5 in vacuum were reported which was attributed to deleterious effects of water in the tribosystem.

Carbon nanotubes (CNT) have been tested for their lubrication capacity [13]. Systems have included nanotubes directly dispersed as a dry powder and free standing, vertically aligned arrays as grown [14] in air, and vacuum environments. For example, dispersed multiwalled nanotubes (MWNT) exhibited friction coefficients of 0.01 to 0.04 in vacuum and 0.06 in air with long wear life (>1 million orbits in vacuum and >3.5 million orbits in air) using a pin-on-disc apparatus [13]. Evidence for transfer layers was found in wear tracks in these studies and our other studies on nanocarbon lubricants including SWNTs and fluorinated versions, using both physical probes and spectroscopic techniques [13].

Such experimental observations are consistent with the additional lubrication mechanisms suggested by Molecular Dynamics simulations for nanostructured lubricants [15]. Such numerical calculations also predict rolling friction in addition to sliding. Perhaps reflecting these multiple lubrication modes, fluorinated SWNTs [16], and functionalized MWNTs [17] have shown superior lubricity compared to Teflon and diamond. CNTs have also proved useful tribological additives within composites of Teflon [18], ultra-high density polyethylene [19], copper [20], and carbon [21].

From the perspective of complimenting the respective tribological properties of liquid and solid lubricants, it would appear advantageous to combine them. A good example would be the perfluoropolyether lubricants which generally have good vapor pressures for use as space lubricants but rather poor lifetimes compared to hydrocarbon and ionic liquid lubricants. Together the two materials could provide lubrication over a wider spectrum of loads and speeds than either component separately. In fact our prior work with carbon nanoions dispersed in Krytox (a commonly used space lubricant) demonstrated a marked lowering of the coefficient of friction (CoF) (0.04 to 0.05) for the mixture with an eightfold improvement in the lifetime over Krytox alone in air [22]. In this work preliminary tribological test results of CNT-ionic fluid mixtures are reported.

2. Experimental

2.1. Materials. Single-walled nanotube (SWNT) and double-walled nanotube (DWNT) materials were obtained from CNI Inc. Both nanotube forms were purified by CNI. The ionic fluid, 1-ethyl-3-methylimidazole tosylate (EMT) was purchased from Aldrich Chemical Co. All materials were used as received. To minimize variability in nanotubes quality, we used materials produced by the same company using the same process.

2.2. Sample Preparation. A special solvent was prepared in-house by mixing 5 g of phenol (Alfa Aesar, 99+%) and 1.5 mL of isopropyl alcohol (Aesar Alcohol, 99+%). Then, 3.8 mg of each lubricant sample were dispersed in 1 mL of the solvent using an ultrasonic bath treatment for 1 h. A

TABLE 1: Summary of lubricant systems.

No.	Description	Concentrations ($\mu\text{g}/\text{mm}^2$)	Source
(1)	SWNT	2.52	CNI
(2)	DWNT	2.52	CNI
(3)	SWNT/EMT	2.52/5.04	CNI/Aldrich
(4)	Graphite—KS-10/EMT	2.52/5.04	Lonza/Aldrich
(5)	EMT	5.04	Aldrich

calculated amount of the formed suspension was dispersed on the surface of a 12.7 mm quartz platter and the platter was mounted in a vacuum chamber constructed in-house and maintained at room temperature as described in the literature [13, 14, 16]. Prior to use the quartz platters were cleaned by ultrasonication in isopropyl alcohol for 5 min and dried in air at 50°C. The vacuum chamber was evacuated for 2 h using a regular mechanical pump until all the solvent was evaporated. Coating films of lubricants with a loading of 2.52 $\mu\text{g}/\text{mm}^2$ were fabricated and tested. The different lubricant systems are listed in Table 1.

2.3. Tribological Testing. The evaluation tests were run on a ball-on-disk tribometer as described in the literature [13]. A 6 mm sapphire ball was loaded (1.4 N) and put in contact with a rotating (120 rpm) quartz disk of 12.7 mm diameter to provide the average Hertzian contact pressure of about 0.3 GPa. The force the ball applied on the disk was used to determine the friction coefficient. All experiments were performed at a track diameter of 6 mm and a sliding velocity of 38 mm/s. The wear life of the lubricant was estimated by the number of rotating passes until the friction coefficient increased to 0.15. All tests were run in an air environment of about 40% relative humidity at room temperature ($\sim 23^\circ\text{C}$).

3. Results and Discussion

Lubricants by necessity interact with the surfaces they are designed to protect. Different materials require different lubricant types and chemistries. Therein it is more appropriate to consider the moving parts and lubricant together as a tribological system [3, 8, 13]. It is well known that carbon-based lubricants form transfer layers [3, 8] which we have directly observed in our prior work involving CNTs [13, 14, 23]. These layers are those molecular layers (derived from the lubricant) that are formed upon bonding to the opposite surface to their initial application. These layers serve to protect the moving surfaces from wear, by creating a more lubricious surface. Hence, the sliding resistance of these layers determines the coefficient of friction. How well these transfer layers adhere to the tribosurfaces depends upon details of surface roughness, chemistry and environment. Nevertheless for carbons in general and nanocarbons in particular, graphene layers are produced by shearing action and can act as a mobile phase lubricant [24]. These values are summarized in Figure 1 and are the average over the lubricant lifetime. The duration over which the lubricant can

continue to furnish these layers in order to maintain low friction is the measure of the lubricant lifetime. End of life was determined as that point at which the friction coefficient exhibited a sharp increase of >50% over a period of <100 orbits. Repetitive tests were conducted to verify consistency of friction and lifetime values. These values are summarized in Figure 2.

We interpret the friction and lifetimes of the lubricant systems listed in Table 1 in terms of the ease of formation of transfer layers and the reactivity of these layers once formed. The SWNTs alone are incapable of forming transfer layers without the degradation of the nanotubes. As the SWNTs adhere to the stainless steel ball and disk surfaces, the relative motion of the two surfaces would not act to form a transfer layer as happens with graphite particles wherein shearing of lamella occurs [25]. With SWNTs the opposing adherence forces would be acting upon the same single graphene layer [26]. With macroscale forces focused at the nanoscale, SWNTs would rupture to form small, planar graphene segments with a predominance of reactive edge sites given their size (reflecting their origin), in effect resembling amorphous carbon. These newly created lamella edge sites would initially be in the form of radicals seeking bond termination [27]. As such they may bond to the tribosurfaces and result in stiction. More likely they would react with other SWNTs and/or ionic liquid given their reactivity. Such reactive amorphous carbon would thereby act more as “glue” than a lubricant, binding SWNTs together and increasing ionic liquid viscosity.

By degrading and immobilizing remaining SWNTs and their bundles, very short lifetime is not surprising. Moreover the graphene segments could even be binding SWNTs (the lubricant) to the tribosurfaces. Therein a high coefficient of friction is probable, as observed.

By comparison, the DWNTs should be able to form transfer layers without suffering complete degradation. Based upon their different physical structure (i.e., single versus double wall), they could “sacrifice” one graphitic shell while yet preserving the inner SWNT core (this sequential degradation is reasonable given the true concentric nature of the tube walls in DWNTs.) This would provide the opportunity for lamella sliding upon SWNTs formed by this stripping action. Moreover the lamella formed by the degradation of the DWNTs would likely be slightly larger than those from SWNTs based on the larger size of the outer cylindrical shell. Overall the result is a lower friction coefficient and a longer lifetime given preservation of nanotube structure, in contrast to the SWNT system. Still, the necessary formation of lamella by degradation of the nanotube wall material yields a relatively short lifetime. As with SWNTs, reactive bonding with neighboring DWNTs would be expected to polymerize the nanotube film. Thus, for either SWNTs or DWNTs, localized wall degradation would lead to frequent occurrences of stiction and reactive bond formation.

We note that observation, let alone “measurement” of transfer layers, is notoriously difficult and generally inconclusive. In the case of SWNTs or DWNTs, planar transfer layers would be formed by crushing upon a substrate. Other studies have also noted this degradation of carbon

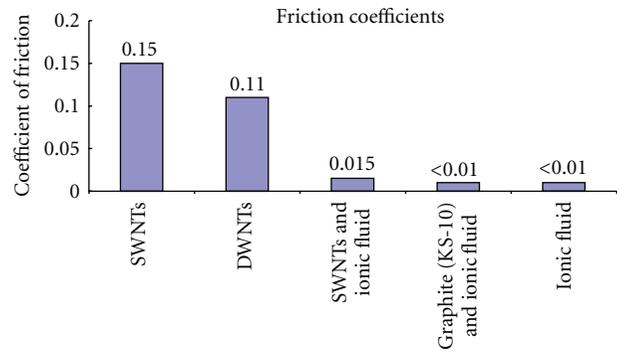


FIGURE 1: Friction coefficients of different lubricant systems.

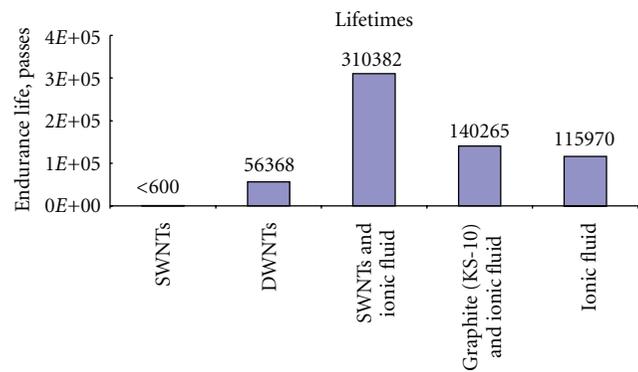


FIGURE 2: Lifetimes as measured by the number of cycles for the indicated lubricant systems.

lubricants [28]. Alternatively they could be non-adherent and dispersed, being then in greater concentration at the edge of the wear track. TEM in situ is not possible and SEM does not possess sufficient resolution for graphene segments of a few nanometers. Traditionally, optical microscopy or micro-Raman has been applied to studies with micron+ size carbon lubricants, carbon or diamond-like films [24, 28]. In these cases measurable amounts of carbon are formed in the wear track. Even in these cases, several complimentary analyses (e.g., Raman, XPS, optical microscopy) are necessary [25, 28]. To note is that these analyses are performed upon dry, solid-phase systems, not liquid-phase lubricants. For studies such as these with a mixed phase lubricant, sampling from the wear track is difficult and leads to considerable uncertainty as to the spatial origin and time (lubricant) history of the sampled material.

In contrast, the ionic liquid, possessing an aromatic constituent (the tosylate moiety), can furnish a molecular transfer layer in its nascent form. Both physical adsorption and chemically reacted films have been formed from ionic fluids [29]. Additionally the ionic nature of the molecule necessarily creates a strong dipole moment that may facilitate its attraction to sliding surfaces but without significant loss of lubricant. We note that formation of transfer layers has been observed with other related

ionic fluids, for example, 1-ethyl-3-hexylimidazolium-bis(trifluoromethylsulfonyl)-imide [30], suggesting this traditional lubrication mechanism to be applicable to other ionic fluids as well. Finally the platelet nature of the ionic fluid molecules could promote their relative molecular motion past bound transfer layers or films. As with other liquid lubricants, the ionic fluid lifetime may be limited by reactions with asperities and local heating at shear points.

The molecular resistance of ionic fluid molecules to undergo translation may arise from the strong dipoles in each molecule created by the separated charges. In contrast to these “molecular lamellae,” those derived from graphite particles would exhibit far less attractive force. Graphite in the form of powders has been thoroughly tested and can yield a friction coefficient of <0.3 , in air [3, 8]. Graphite particles readily shear to produce lamella without undergoing significant degradation [3], unlike closed-shell nanocarbons (in fact mechanical erosion is one method for making graphene [31]!) In this system transfer layer interactions can occur between the ionic liquid molecules, between the carbon lamellae and between these two systems. In this case the ionic fluid and graphite lamellae may act as transfer layers relative to each other. The dissimilarity in size between lamella formed by the graphite flake and the ionic liquid molecules may further facilitate low friction as edge sites of the lamella derived from the graphite are not encountered frequently. Both the larger carbon lamella and the preservation of their originator (namely, the graphite flake reflecting its micron size scale) are consistent with the lower friction coefficient and longer lifetime of this lubricant system relative to SWNTs or DWNTs without any other lubrication sources. Such synergies between the two lubricants could account for the enhanced lifetime relative to the pure ionic fluid.

The final system considered is the SWNT plus ionic fluid mixture. Similar to the graphite flake-ionic fluid system, this mixture potentially could also form three transfer layer systems. The fact that this mixture possesses the longest lifetime points to a synergy between the ionic fluid and SWNTs. The SWNTs have high friction and very short lifetime. While the pure ionic fluid has a significantly lower friction coefficient, its lifetime is nearly 300% lower relative to that of the SWNT-ionic fluid mixture. We postulate that the ionic fluid may form a strong physisorbed layer upon the SWNTs. Such physical adherence is the basis by which surfactants disperse SWNTs with those surfactants possessing an aromatic moiety, for example, Sodium Dodecyl Benzene Sulfonate, being the best [32]. This reflects energetically favorable π - π orbital overlap. The high dipole moment of the ionic fluid would further facilitate molecular attraction. This molecular coating would act to substantially reduce SWNT mechanical degradation. Moreover the ionic fluid, as observed elsewhere [30], could itself form a transfer film.

Therein the ionic fluid could favorably promote the SWNT lubricant contribution via three synergetic processes. With a greater than 10x smaller size, the ionic fluid molecules would be expected to intervene between the SWNTs and tribosurfaces. Secondly, the sidewall “functionalization” of the nanotube by the ionic fluid could serve to separate

reactive sites (tribocreated defects) from the tribosurfaces. Finally, this functionalization could act to separate the SWNT bundles, the analogy acting as an exfoliant. More nanotubes and/or bundles (aka “ropes”) with fewer numbers of tubes may provide a larger solid-phase contribution to the lubrication system. To enable these interactions defines the requirements of the base lubricant, namely, that it protects the solid-phase lubricant while also providing lubrication itself. Such synergistic actions were also found in a mixed lubricant containing a mixture of an ionic fluid and non-polar liquid crystal [33].

In contrast to the graphite flake, the SWNTs (when their physical structure is preserved) can act as an extended carbon lamella (i.e., as transfer layers) without any reactive edge sites. Retention of their physical structure may be aided by their high Young’s modulus. Molecular dynamic simulations have shown the capability for Stone-Walls defects in the sidewall to “heal” by conversion of the C5-C7 ring pair to a C6-C6 structure. Other simulations have shown that mechanically deformed SWNTs can return to their initial state upon release of the load [34–36]. Again, even if defects form, the ionic fluid may well act as a molecular “band aid” for the site. Moreover the high SWNT radius of curvature would maintain minimal contact with opposing transfer layers thereby leading to minimal friction. In summary, with an ionic fluid providing molecularly based transfer layers and serving to protect and disperse SWNTs, their degradation is inhibited. With their high tensile strength and elastic modulus, the SWNTs can contribute complimentary lubricant properties to the binary lubricant system. In the context of the present system, an extended lubricant lifetime is realized.

The subject of transfer layers and their nanosize scale and formation from nanomaterials is relevant to the Nanotechnology field and therein presents a new aspect of nanotechnology that yet to date has received little if any attention. Despite the science of lubrication and wear being one of the oldest engineering fields, it is particularly relevant to energy conservation needs of today, as power generation, for example, wind turbines, transportation, for example, autos, trains, and manufacturing, for example, conveyors, robotics, and so forth, as all depend upon moving parts and components. Future work remains to understand the detailed tribochemistry of mixed nanolubricants for space- and energy-related applications [37].

4. Conclusions

Ionic fluids can have superior vapor pressures to conventional lubricant base fluids, and CNTs have negligible volatility, a primary requirement for space lubricants and longevity in general usage. The combination of SWNTs with ionic fluid EMT yielded a >500 and 2.7 times lifetime improvement over the SWNTs alone and EMT, respectively. It can be expected that additional lifetime improvement will be achieved in vacuum due to the superior performance of ionic fluids in vacuum as opposed to air. DWNTs may prove superior to SWNTs combined with ionic fluids, but, due

to their considerably greater cost, we have not considered them for further development. These results illustrate the promise of carbon nanomaterials as lubricants and their potential contribution to energy efficiency for a wide range of applications.

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