

Diamond-like Carbon Coatings with Ionic Liquid Lubricants

Subjects: **Nanoscience & Nanotechnology**

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Owing to the solid lubrication characteristic of Diamond-like carbon coatings (DLCs), which reduces wear and friction and protects contacting surfaces from degradation, DLCs are often used in industrial machinery and harsh environments. DLC coatings are optimized by adjusting operating and deposition parameters as well as doping them with other elements to improve performance, such as thermal stability and chemical resistance. Ionic liquid (ILs) are a promising green lubricant option due to their low melting temperature, superior thermal stability, and acceptable miscibility with organic substances. ILs have been studied as main lubricants and additives to the main lubricants, and their tribological properties have been investigated, including their use as extreme temperature lubricants. The tribological properties of pure/doped DLC coatings with ILs have also been explored, although limited research has been conducted in this area. The combined synergistic effect of DLCs and ILs shows great promise in reducing energy loss due to friction, promoting longevity, and conserving energy.

diamond-like carbon coating

ionic liquid

tribology

DLC

sustainable lubricants

tribofilm

1. Introduction

Concerns about energy and environmental sustainability have led to a need for more efficient and cleaner industrial activities and transportation systems. Fossil fuels are the main source of global energy consumption, which has a significant negative impact on the environment ^[1]. Vehicles alone consume about 30% of all energy produced ^[2], and a third of that is lost due to wear and friction. Friction losses and wear were identified as the primary culprits that restrict the lifespan and diminish the effectiveness of machinery used in the industry. As a result, these factors have a significant influence on the economy. This has a major impact on national economies, as shown by studies from several countries (United Kingdom ^[2], United States of America ^[3], Japan ^[4], and Canada ^[5]). Considerable resources have been dedicated globally to enhance the energy efficiency and wear resistance of moving mechanical parts. These endeavors, coupled with the increasing prevalence of electrically powered modes of transportation, represent a major stride towards meeting the demands for decreased carbon dioxide emissions set forth by the Kyoto ^[6], Paris ^[7], and EURO 7 ^[8] guidelines.

To decrease friction between sliding components and prevent mechanical wear, the conventional method is to utilize lubricants, typically mineral or synthetic oils in internal combustion engines or water-based emulsions in machining fluids. Additives are frequently incorporated into these fluids to enhance their existing properties or

introduce new ones. Chemical compounds that can adsorb and/or interact with solid surfaces to generate low shear-strength reaction layers (known as “tribofilms”), are among the additives utilized to reduce friction (called “friction modifiers”), wear (known as “anti-wear additives”) [9][10], and oxidation to modify rheology or even to add fragrances [11].

Zinc dialkyldithiophosphates (ZDDPs) have been utilized as additives in engine oil formulations to reduce wear since the 1940s. For many years, scientists have investigated the lubrication mechanism of ZDDP. The predominant view is that ZDDP functions as an anti-wear agent through a surface reaction that results in the formation of uneven phosphate films with a glass-like appearance. This theory has been widely accepted by researchers studying ZDDP’s lubrication properties [12]. Although ZDDPs have proven to be highly effective in reducing wear across various conditions, their usage has also highlighted environmental concerns due to the significant levels of phosphorus, sulfur, and zinc they contain. These elements are recognized for their ability to cause clogging in filters and catalyst degradation in the exhaust after-treatment systems used in gasoline and diesel engines. As a result, the permissible levels of sulphated ash, phosphorus, and sulfur have gradually been restricted in modern engine lubricant formulations to address these environmental concerns.

Also, the oil-based lubricant constitutes an environmental threat. Moreover, the biodegradability of mineral oils is very low and the lubricant is released to the ambient environment in the form of microdroplets and accumulates on plants, animal, and groundwater tissues. The water pollution may be very aggressive since the oxygenation may be altered, leading to a disorder in the ecosystem. Soils also suffer serious damage from oil pollution due to the physical and chemical processes that lead to changes in the forms and distribution of organic matter and the range of carbon, water, nitrogen, and phosphorus, which can alter their entire ecosystem [11]. Because of all these concerns, researchers and companies have focused on developing natural-based oils to infer biodegradability in this product. Existing vegetable-based lubricants have good lubricity, ensuring a high cleaning effect. However, oxidative stability and low-temperature properties are their main drawbacks and require the integration of additives [11].

In recent years, there has been a growing interest in the use of ionic liquids (ILs) as a replacement or supplement to ZDDPs and other molecules in lubrication science and engineering, as well as in machining fluids. Ionic liquids are molten salts which consist of an organic cation and a weakly coordinating anion. They have become increasingly desirable in lubrication due to their exceptional properties, including high thermal stability [13], low flammability [14], and negligible vapor pressure [15]. Moreover, air-stable ILs with various structures and functionalities can be synthesized, making them a promising option for use in applications involving extreme conditions in space for components in space-shuttle [16], and micro-electromechanical systems [17][18]. As a result, ILs have garnered considerable attention as a potential solution for lubrication in such demanding situations.

Numerous studies have been conducted since 2001 when ILs were first suggested as potential lubricants [19][20][21]. In a recent study by Zhou et al. [22], it was demonstrated that phosphorus-based ILs react with cast iron surfaces and create phosphate-rich tribofilms that possess a similar chemical composition to those generated by other organophosphates, which are commonly used as anti-wear additives. Another area of research to reduce the use

of lubricants has been the development of surfaces modified with self-lubricating coatings, such as diamond-like carbon (DLC) or transition metal dichalcogenides (TMDs). In the past few years, there has been significant interest in the research community towards combining diamond-like carbon (DLC) coatings with ILs due to their exceptional tribological properties such as resistance to wear and high levels of protection against corrosion thanks to their inert properties [23]. DLC coatings are composed of an amorphous network of carbon atoms that are in sp^3 and sp^2 hybridization and have hydrogen attached to terminate the dangling bonds [24][25][26]. The flowchart in **Figure 1** explains the benefits of using DLC coatings along with ILs.

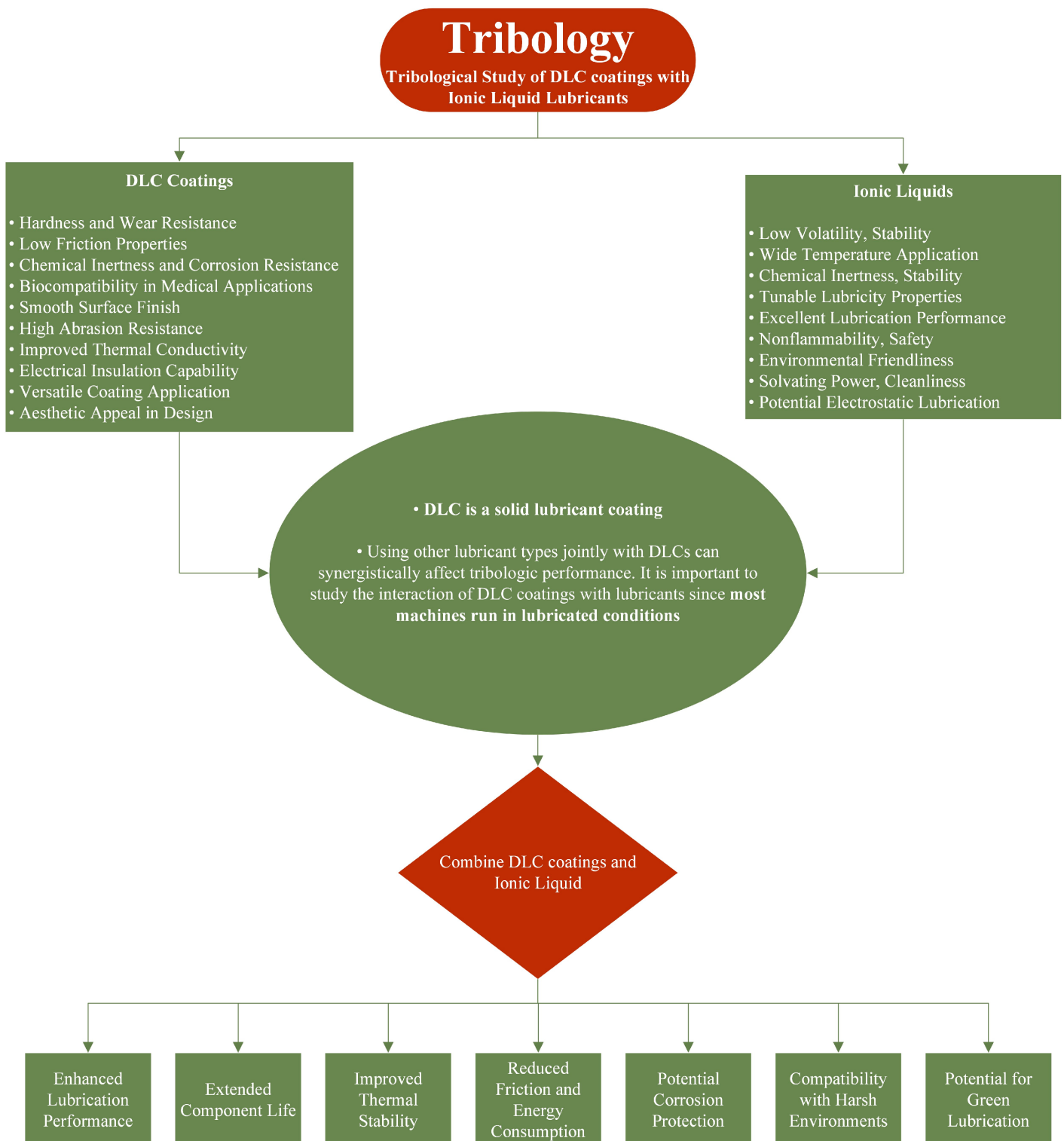
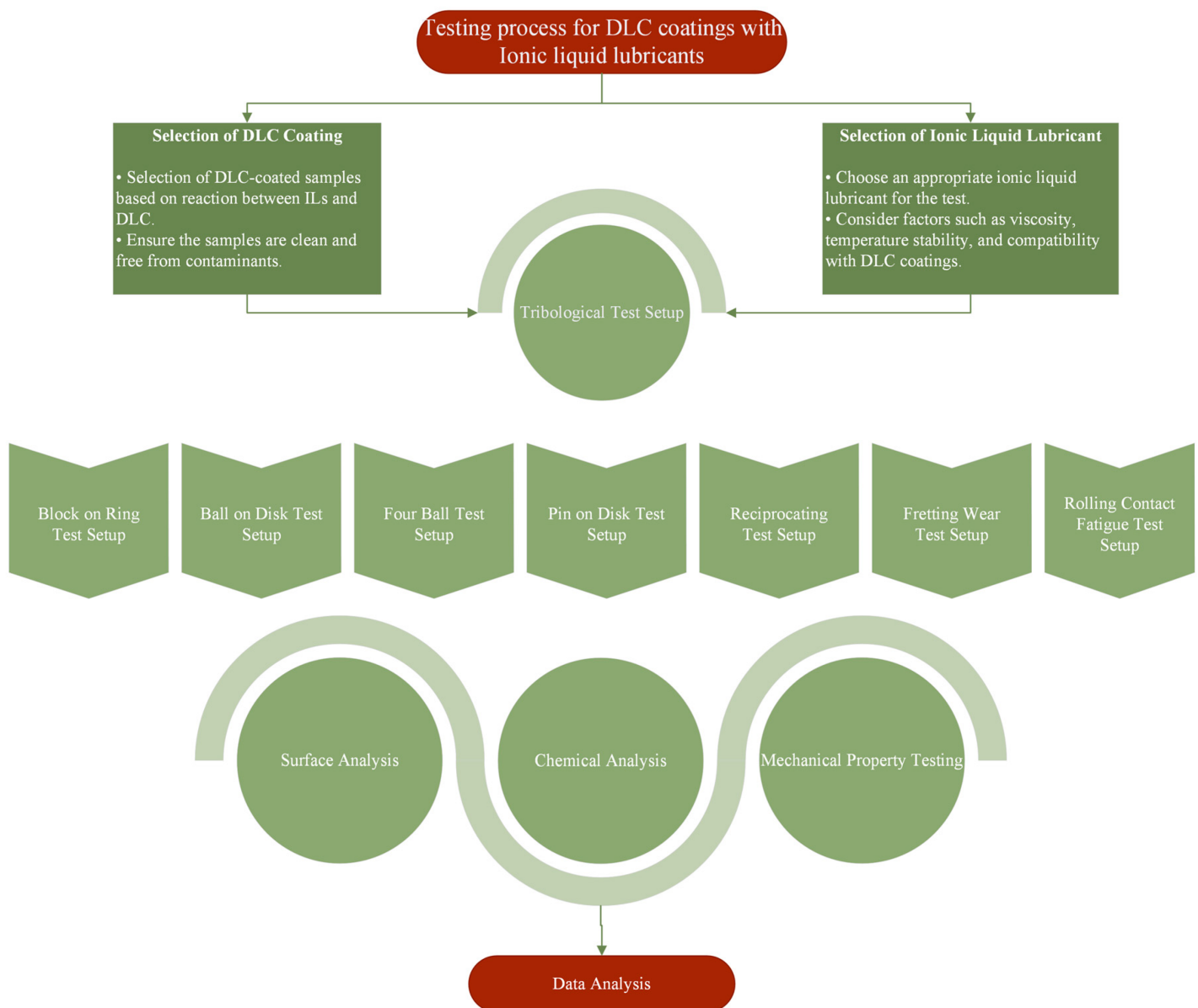


Figure 1. Tribological study of DLC coatings with ionic liquids lubricants.

2. Tribological Testing of DLC Coatings with Ionic Liquid Lubricants

Tribological tests are designed to evaluate the friction, wear, and lubrication properties of materials in relative motion. **Figure 2** shows the setups that help researchers and engineers understand the tribological performance of materials and lubricants, guiding the selection and design of components for various applications.

**Figure 2.** Testing process for DLC coatings with ionic liquid lubricants.

2.1. Tribology of Pure Diamond-like Carbon Coatings and Ionic Liquids

Industry has long used solid lubricants to lessen wear and friction under varied circumstances. Advanced vacuum methods have been used to deposit materials like tungsten disulfide (WS_2), hexagonal boron nitride (HBN), borides (MgB_2 and ZnB_2), and soft metals like Cu, Ag, Sn, and Au as protective coatings or as lubricant additives. These substances provide a barrier that shields friction pairs from one another, reducing friction and enhancing wear resistance [27]. Because of its outstanding qualities, such as high hardness, excellent chemical stability, high thermal conductivity, low friction, and exceptional wear-resistance, carbon has attracted the most interest of these solid lubricants [20]. There are various forms of carbon, and each form's characteristics rely on its particular structure. Researchers have intensively investigated different carbon forms and their applications over many decades [27]. Two specific forms of carbon, namely sp^3 and sp^2 form diamond-like carbon (DLC) thin films [27].

While operating under boundary lubrication conditions, applying solid lubricant thin films (coatings) can improve the lubrication of moving pairs [20][28]. A solid-liquid composite lubricating system can have a synergistic impact by integrating the benefits of liquid and solid lubrication. Solid lubrication coating has favorable load-bearing capabilities and low volatility. By doing so, the system is able to retain the benefits of solid lubrication while simultaneously gaining the benefits of liquid lubrication [29]. When the thin layers of fluid that separate surfaces fail, solid coatings can bear the load and serve as a secondary form of lubrication. This provides a backup mechanism to prevent direct contact between surfaces and reduce friction [30]. The combination of solid and liquid lubrication systems has become a popular choice for dynamic equipment, including space and automotive mechanisms. This composite lubricating system offers an effective solution for reducing friction and preventing surface wear. Thus, recent research has explored the potential benefits of integrating ionic liquids (ILs) with solid lubricating films. This promising approach has the potential to further improve the performance and durability of the composite lubricating systems [20].

Zhao et al. [31] examined the adhesion and friction of DLC and DLC coating in the presence of 1-octyl-2,3-Dimethylimidazolium bis(trifluoromethyl)sulfonyl ionic liquid as lubricant. To do so, a colloidal probe mounted on an AFM cantilever was used in contact mode to evaluate adhesive and nanotribological behaviors, and a UMT-3 tribometer was used in a ball-on-plate reciprocating mode to evaluate their microtribological behaviors. The experimental findings indicated that the friction forces of the DLC films with micro-grooves were effectively reduced by introducing IL to the tribological pair. The reduced adhesion and friction forces were explained by lubricity property of ionic liquid to prevent direct contact between the DLC coatings and colloidal tip, which made the sliding of the colloidal tip on the DLC coating's surface easier. The reciprocating test setup involves applying back-and-forth sliding motion to examine the wear and friction characteristics of materials, particularly in reciprocating applications such as ball-on-plate reciprocating mode or a piston-cylinder system. This setup proves valuable in assessing the performance and durability of components subjected to reciprocating motion which is shown in the below **Figure 3**.

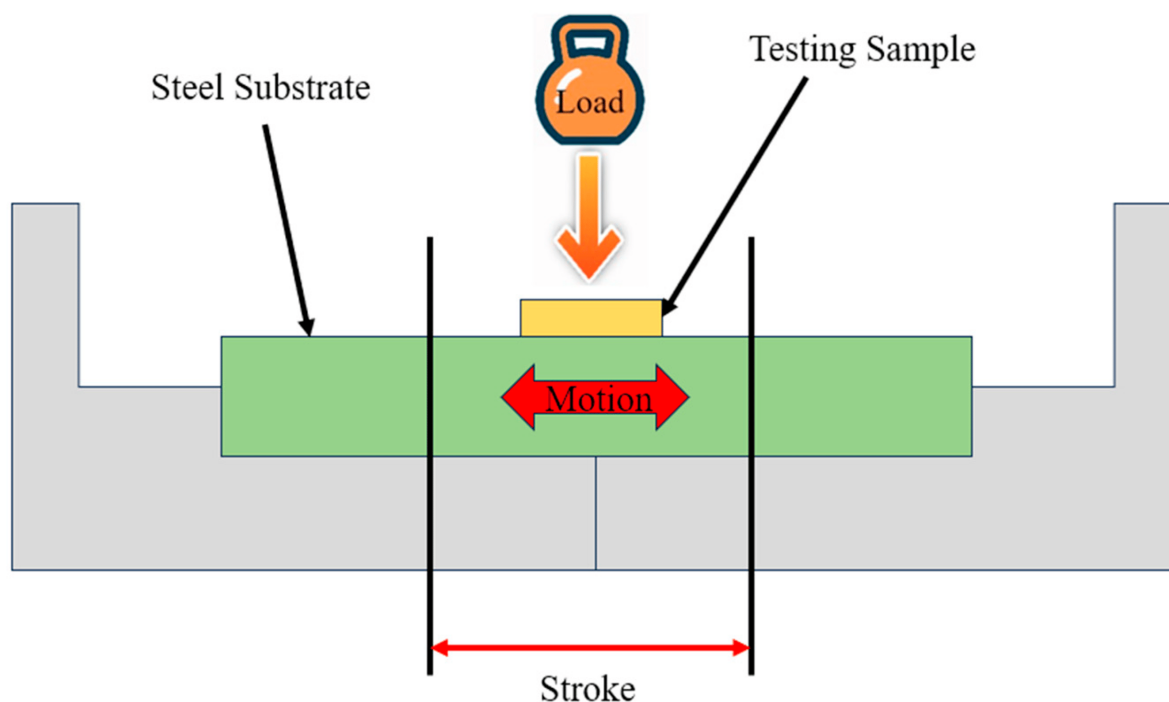


Figure 3. Pin-on-plate reciprocating tribological test setup.

The outcomes of the experiment that tested the friction coefficient of three PVD coatings using [BMP][FAP] lubricant demonstrated comparable values for TiN, CrN, and DLC, with CoF of DLC being the smallest, which suggests that the ionic liquid had a noticeable impact on the coatings regardless of their type [32]. To gain a more profound understanding of the interactions between the coatings and the ionic liquid at a chemical level, XPS analyses were conducted to examine the wear surfaces. The breakdown of ILs starts the process, and the dynamic components in the ILs may respond with the newly formed surface to create a reaction film, also known as a tribofilm. This tribofilm is capable of protecting the surface from significant wear. A layer is formed on the surface of DLC by the ionic liquid, where 40% of the fluorine is involved. When the experimental conditions became harsher, causing an increase in temperature and pressure, the interaction rate also raised. As a result, roughly 80% of the overall fluorine became connected to the surface within the wear scar, in contrast to 40% outside the scar [32]. Another study investigated the same coatings but in the presence of another ionic liquid described as [(NEMM)MOE][FAP]. When the films were lubricated with pure ionic liquid ([NEMM)MOE][FAP]), the lowest friction coefficient was achieved compared to when used as an additive to PAO 6 as base oil, and no noticeable wear scar was identified. The behavior of the ionic liquid and DLC interaction differed significantly from that observed in other films. The ionic liquid does not interact with the surface of DLC unless there is a significant increase in loading conditions. As a result of significant increase in loading conditions, approximately 77% of the fluorine (F) reacts with elements inside the wear region. When compared to previous research conducted under identical testing conditions, the [BMP][FAP] ionic liquid displayed marginally superior performance as a pure lubricant and as an additive in the lubrication of the three PVD coatings examined, relative to the [(NEMM)MOE][FAP] ionic liquid. The improved tribological behavior of [BMP][FAP] can be attributed to its higher viscosity and stronger interaction with these coatings, which encourage the growth of tribofilms [32][33].

Jia et al. examined the effectiveness of synthetic ionic liquid functionalized borate esters as additives in PAO as the base lubricant in terms of friction and wear properties for DALC films compared to ZDDP [34]. According to the results obtained, the addition of borate esters in PAO provided improved friction and wear performance for DLC films in comparison to ZDDP. The authors hypothesized that a triboplasma forms during the process, particularly on DLC films, and that the borate esters modified with ionic liquid might adhere to the worn surface of the pair because of the triboplasma. Furthermore, the decomposition of the borate esters during the process leads to the B atoms infiltration into the flaws and sublayer of the worn surface. Consequently, a tribofilm containing B, N, and F was formed to lower friction and reduce wear [34]. **Table 1** shows the brief summary of the undoped diamond-like carbon coatings and ionic liquids.

Table 1. Summary of the available literature investigating tribological performance of undoped diamond-like carbon coatings and ionic liquids.

Name of ILs	Tribometer	Additive or Main Lubricant?	Ref.
1-octyl-2,3-Dimethylimidazolium bis(trifluoromethyl)sulfonyl	UMT-3 tribometer (ball-on-plate)	Used as main lubricant (100 wt.%)	[31]
1-butyl-1-methylpyrrolidinium tris(pentafluoroethyl)trifluorophosphate	CETR-UMT-3 micro-tribometer (ball-on-plate)	Used as both additive in PAO 6 (1 wt.% additive) and main lubricant (for comparison)	[32]
ethyl-dimethyl-2-methoxyethylammonium tris(pentafluoroethyl)trifluorophosphate	UMT-3 microtribometer (ball-on-plate)	Used as both additive in PAO 6 (1 wt.%) and main lubricant (for comparison)	[33]
The synthesized ionic liquids of functionalized borate esters (The specific names are not mentioned but molecular structure is explained in the article)	Reciprocating friction and wear tester (ball-on-disk)	Used as 2 wt.% additives to polyalphaolefin (PAO 6) and compared with 2 wt.% ZDDP as a reference additive	[34]
(1) 1-butyl-3-methylimidazolium tetrafluoroborate And (2) trihexyltetradecylphosphonium bis(trifluoromethylsulphonyl) amide	T-01M tester (ball-on-disc configuration)	Used as main lubricant (100 wt.%)	[35]

comprised of a steel ball and a steel disc coated with DLC. In this comparison, the 1-butyl-3-methylimidazolium tetrafluoroborate ionic liquid outshone its counterpart, the trihexyltetradecylphosphonium bis(trifluoromethylsulphonyl) amide ionic liquid [35]. Notably, the former demonstrated superior efficacy in lubricated friction and exhibited compatibility with a-C:H type DLC coatings. The friction pair, consisting of a steel ball and a steel disc with DLC coating, played a crucial role in the evaluation. The steel ball, a standard material for friction testing, offered durability and widespread applicability in industrial settings. Meanwhile, the steel disc's DLC coating contributed to enhanced wear resistance and served as a protective layer, prolonging the material's lifespan. Exploring possibilities for synthesizing or mixing these ionic liquids could further optimize their performance. Such endeavours may lead to improved lubrication properties, enhanced compatibility with DLC

coatings, and potentially cost-effective solutions. The ball-on-disk test setup, depicted in **Figure 4**, served as a valuable tool by allowing the examination of contact between the ball and rotating disk, providing insights into friction and wear properties. This testing methodology finds extensive applications in both sliding and rolling conditions, offering a comprehensive assessment of material performance and durability in various industrial contexts.

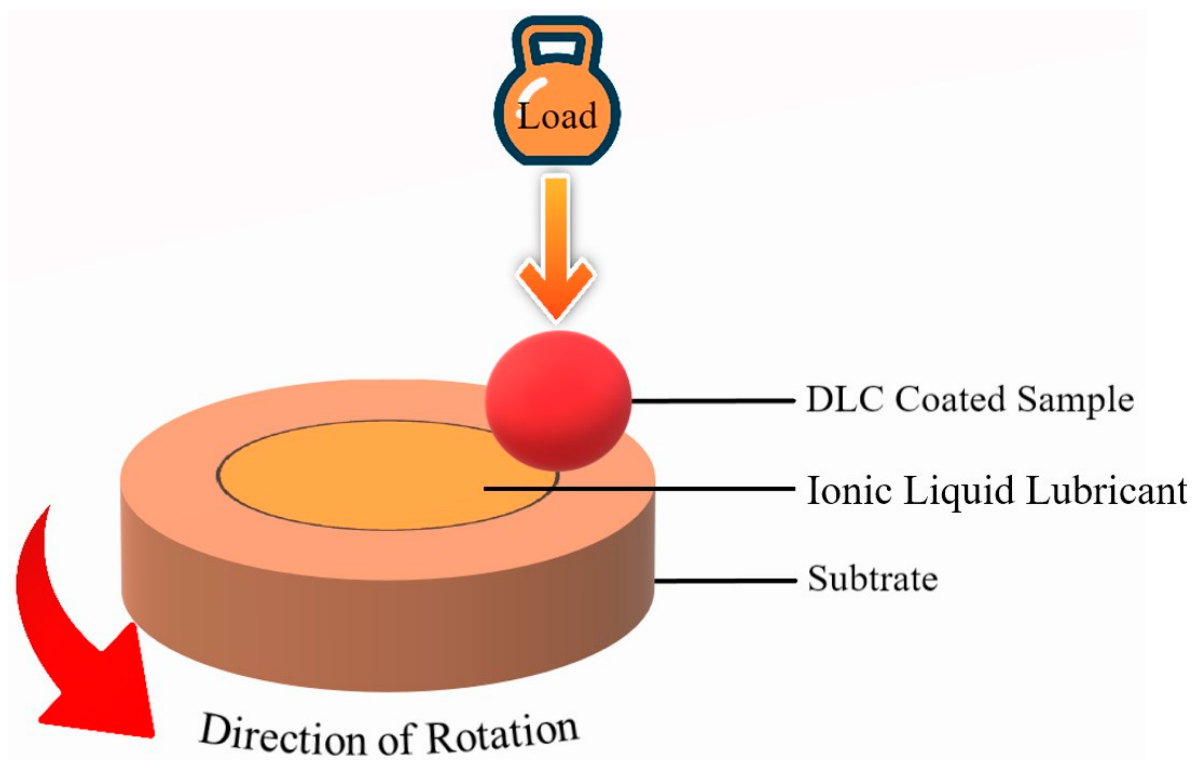


Figure 4. Ball-on-disk tribological test setup.

2.2. Tribology of Doped Diamond-like Carbon Coatings and Ionic Liquids

In their investigation, Madej et al. [36] explored the tribological properties of diamond-like carbon (DLC) coatings, specifically a-C:H and a-C:H:W, fabricated using plasma-assisted chemical vapor deposition (PACVD) and physical vapor deposition (PVD) methods. These superhard anti-wear coatings were applied to steel elements and subjected to scrutiny under both dry and ionic liquid lubricated conditions. The study utilized atomic force microscopy (AFM) and scanning electron microscopy (SEM) for comprehensive surface analysis, examining topography and cross-sections. Tribological assessments, employing a ball-on-disc tribometer and a pin-on-plate tribometer, revealed that the DLC coatings outperformed the steel substrate, displaying lower wear, reduced friction, and higher hardness [36]. To delve further into the wear resistance and tribological performance, there is potential in exploring synthesis possibilities such as optimizing blending ratios or combining PACVD and PVD methods. The pin-on-disk test setup, elucidated in **Figure 5**, emerged as a pivotal tool in assessing friction and wear characteristics in sliding contact applications. This comprehensive study contributes valuable insights into the advanced properties of DLC coatings. **Table 2** shows the brief summary of the doped diamond-like carbon coatings and ionic liquids.

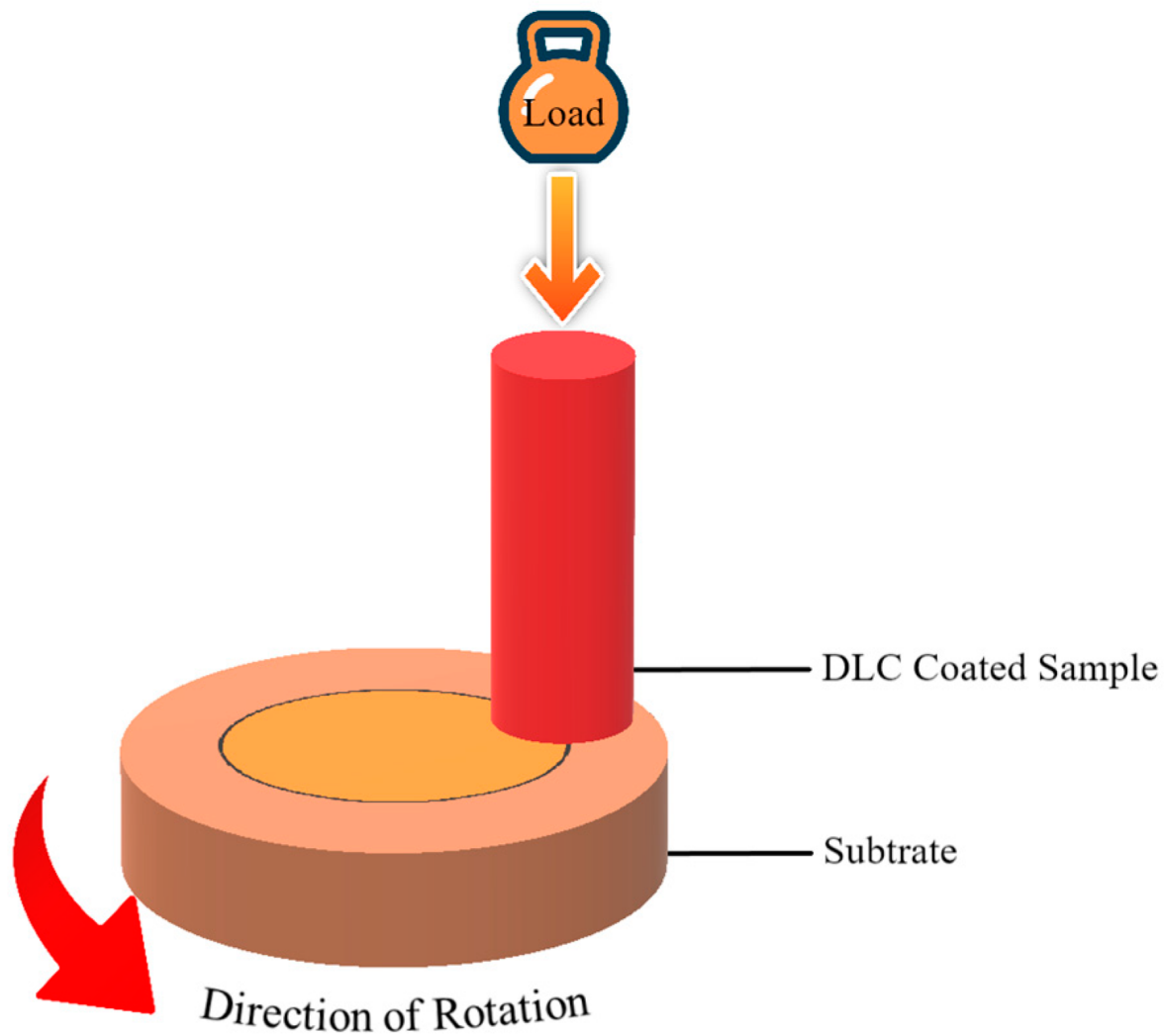


Figure 5. Pin-on-disk tribological test setup.

Table 2. Summary of the available literature investigating tribological performance of doped diamond-like carbon coatings and ionic liquids.

Name of Ionic Liquids	Tribometer	Doped DLC Type	Ref.
dialkylimidazolium tetrafluoroborates (Used as main lubricant, i.e., 100 wt.%)	T-01M tribometer (ball-on-disc configuration)	W-doped DLC	[36]
tributylmethylphosphonium dimethylphosphate (PP) (used as additive with 1 wt.%) and 1,3-dimethylimidazolium dimethylphosphate (IM) (used as additive with 1 wt.%) and 1-butyl-1-methylpyrrolidinium tris(pentafluoroethyl)trifluorophosphate (BMP) (used as additive with 1 wt.%)	TE 38-Phoenix Tribometer (reciprocating pin-on-disc tribometer)	W-doped DLC, and Ag-doped DLC	[23]

Name of Ionic Liquids	Tribometer	Doped DLC Type	Ref.
1,3-dimethylimidazolium dimethylphosphate (used as additive with 1 wt.%)	UMT-2 tribometer with (ball-on-flat-disc geometry)	W-doped DLC	[37]
(1) 1-butyl-1-methylpyrrolidinium tris(pentafluoroethyl) trifluorophosphate ([BMP][FAP]) (used as additive with 1 wt.%) and (2) tributyl-methyl-phosphonium dimethylphosphate (PP) (used as additive with 1 wt.%) and (3) (2-hydroxyethyl) trimethylammonium dimethylphosphate (AM) (used as additive with 1 wt.%)	UMT-2 tribometer (reciprocating motion with a ball-on-flat-disc geometry)	W-doped DLC	[38]
(1) tetrafluoroborate (LB104) or [BF ₄] (LB104) (used as main lubricant, i.e., 100 wt.%) And (2) tetrafluoroborate (LAB103) or [BF ₄] (LAB103) (used as main lubricant, i.e., 100 wt.%)	CSM Switzerland tribometer (ball-on-disc)	Cr-doped GLC and Cr-doped DLC	[26]
trihexyltetradecylphosphonium bis (2-ethylhexyl) phosphate [P ₆₆₆₁₄] [DEHP] (used as additive with 1 wt.%)	Block-on-Ring configuration	Gd-DLC and Eu-DLC	[39]
trihexyltetradecylphosphonium bis(trifluoromethylsulfonyl) amide (used as main lubricant, i.e., 100 wt.%)	Anton Paar TRB tribometer (Ball-on-disc configuration)	Si-DLC	[40]
1-alkyl-3-octylimidazolium hexafluorophosphate (L-P801) (used as main lubricant, i.e., 100 wt.%) And 1-alkyl-3-octylimidazolium hexafluorophosphate (L-P804) (used as main lubricant, i.e., 100 wt.%)	UMT-2MT (reciprocating ball-on-disk)	Ti-DLC	[41]

Tungsten carbide precipitates were formed by W in the DLC coating. Friction was controlled by two different additive-adsorption mechanisms: a triboelectrochemical activation mechanism for Ag-DLC and an electron-transfer mechanism for W-DLC, resulting in the largest friction reduction [23]. The friction of DLC (diamond-like carbon) is influenced by the electrical charge of the surface and the lubricant's ability to adsorb to the surface. The electrical conductivity of the lubricant (ionic liquid) drives the transport of additives to the surface and impacts friction. The higher the lubricant's electrical conductivity, the lower the friction. Additionally, the Zeta-potential of DLC surfaces in aqueous solutions is negative, resulting in no change in friction with the addition of negatively charged molecules [42][43]. The higher electrical conductivity in IL-additivated lubricants leads to faster transport kinetics of the cation and anion moieties to the surface, resulting in lower friction [23].

Arshad M. et al. explored the potential of enhancing the lubrication properties of tungsten-doped diamond-like carbon coatings by incorporating a 1,3-dimethylimidazolium dimethylphosphate ionic liquid into glycerol base oil. The researchers conducted tribological tests under varying loads (5 N, 10 N, 20 N) and elevated temperature (100 °C). They found that the friction coefficient was significantly reduced by approximately 50% when 1 wt% IL was included. They also discovered that the formation of a thin tribofilm on the surface of the coatings played a crucial

role in friction reduction. The study provides insights into the lubrication mechanism and offers implications for the development of improved lubricants for tribological systems [37].

Another investigation was conducted by Arshad M. et al. to examine the interaction between coatings of tungsten-doped diamond-like carbon (WDLC) and three phosphate-based ionic liquid (IL) additives. Among these additives, two contained the anion dimethylphosphate, while the third contained the hydrolytic trifluorophosphate anion. The tests were carried out under boundary-lubrication conditions. The findings indicated that IL additives containing dimethylphosphate anions exhibited reduced friction on the surface of WDLC, whereas the IL with trifluorophosphate anion displayed poor performance. Analysis of the surface revealed the formation of a tribofilm based on phosphate on the WDLC surface when dimethylphosphate additives were present, resulting in friction reduction. The WDLC coatings demonstrated remarkable resistance to wear [38].

Researchers conducted a study on Chromium doped diamond-like carbon coatings (Cr-DLC) using PVD and PECVD methods [26]. The lubrication performance of solid-liquid composite lubrication systems was studied using two ionic liquids (ILs) as lubricants. The findings indicated that the friction coefficient was reduced by about 40% when compared to dry conditions, and the composite system displayed an effective synergistic lubrication effect. The Cr-DLC coating showed superior tribological, mainly because of its improved physicochemical film formation during friction and the dense microstructure of the Cr-DLC coating. The ILs' viscosity, corrosiveness, and coating microstructure influenced the composite systems' synergistic effect [26].

Rare earth metals, such as Gd and Eu, present an intriguing avenue for enhancing the reactivity of diamond-like carbon (DLC) when incorporated into its composition. Studies indicate that DLC films doped with low atomic concentrations (1–3 at.%) of Eu or Gd, particularly in dry contact conditions without lubrication, exhibit a marginally higher coefficient of friction (CoF). However, these films demonstrate commendably low wear rates and boast high hardness, rendering them well-suited for diverse applications [39]. Furthermore, investigations by Shaikh et al. [44] and Sadeghi et al. [45] highlight that DLC coatings doped with gadolinium can yield an even lower coefficient of friction compared to pure DLC when subjected to trihexyltetradecylphosphonium bis(2-ethylhexyl) phosphate [P₆₆₆₁₄] [DEHP] ionic liquid (1 wt.%) as an additive to polyalphaolefin (PAO) 8. This is due to the reactivity of the ILs and dopants, to explore the possibilities and advantages of synthesizing or mixing these materials, attention should be given to optimizing the concentration of rare earth metal dopants for optimal tribological performance. The Block on Ring test setup, illustrated in **Figure 6**, played a central role in these investigations. This setup involves pressing a stationary block against a rotating ring, enabling the measurement of friction and wear characteristics between the two components. Widely applied in assessing materials for brake linings and clutch facings, the Block on Ring test provides valuable insights into the durability and tribological properties of materials under conditions resembling real-world applications. After the tests, the results were confirmed using the calculations in the stribeck curve and SEM/EDS analysis which showed presence of phosphorus from ionic liquid on the surface of the doped DLC coating.

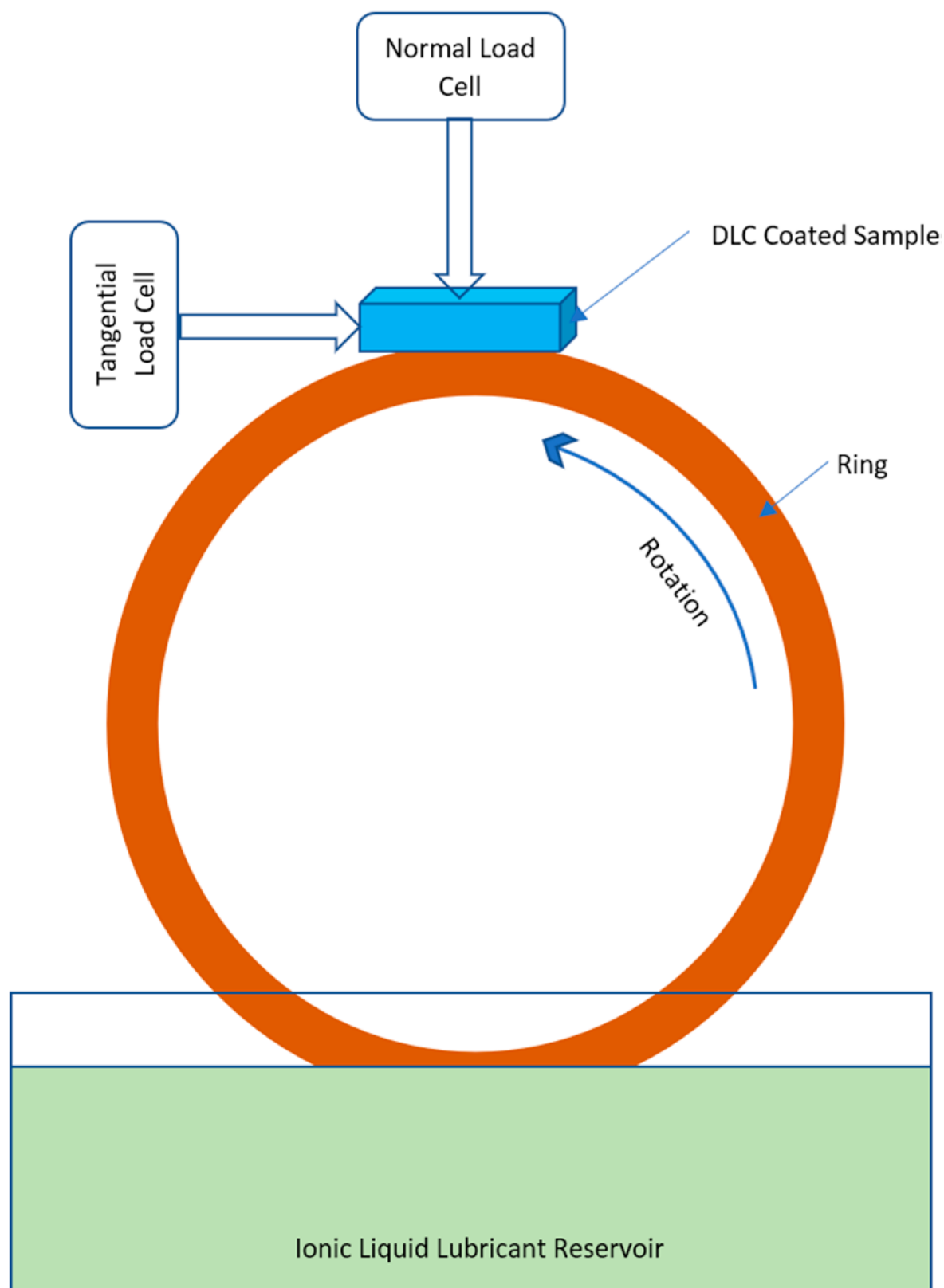


Figure 6. Block-on-ring tribological test setup.

Similar study examined the effect of a silicon doping on tribological behavior of diamond-like carbon coating in IL-lubricated friction pairs ^[40]. Tests showed that the Si-DLC coating, when used with the ionic liquid, reduced both

coefficient of friction and wear. The study concluded that using Si-DLC coatings lubricated with the ionic liquid helps to improve tribological properties of sliding surfaces under friction [40].

In another study, Ti-doped DLC coatings were analyzed using Raman and TEM. Two types of 1-alkyl-3-octylimidazolium hexafluorophosphate ILs (L-P801 and L-P804) were synthesized and evaluated as lubricants for Ti-DLC/steel contacts, with excellent friction-reducing properties. To investigate the chemical components of tribofilm formed on the contacting surface for Ti-DLC films in the presence of L-P801 ionic liquid, XPS characterization method was applied which confirmed the existence of PF₆ anion, amine and/or nitrogen oxide, and fluoride on the surface. These coatings with ILs lubricating systems have potential as lubricants in vacuum and space moving friction pairs [41].

Limited research has been conducted on the tribological properties of doped DLC coatings with ionic liquids (ILs), and their combined effect has been found to be highly beneficial. The addition of doping elements has been demonstrated to improve the tribological properties of DLC coatings significantly. ILs, with their exceptional characteristics such as excellent lubricity and high thermal stability, are deemed ideal for various tribological applications. Through the formation of a protective film or tribofilm on the surface of the coating, the combination of doped DLC coatings and ILs has been reported to elevate their tribological properties further.

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