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REVIEW ARTICLE

Research progresses of nanomaterials as lubricant additives

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Abstract: Friction and wear are unavoidable in mechanical movement. The use of lubricants with nano-additives can effectively reduce friction and wear, which is of great significance to saving energy and protecting the environment. At present, great progress has been made in the scientific research and industrial application of nano-additives for lubricants. This paper mainly introduces the types of nano-additives for lubricants (such as carbon nanomaterials, nano-metals, nano-oxides, sulfides, complexes, polymers, etc.), the tribological properties of lubricants with different components of nano-additives, and the lubrication mechanisms of the nano-additives (including tribofilm formation, rolling ball bearing effect, repairing effect, polishing effect, and synergistic effect). It also deals with the dispersion of nano-additives in lubricants and the influences of their particle size and microstructure on the tribological properties of lubricants. This review outlines the performance requirements of nano-additives in different lubrication states, discusses the use of nano-additives in challenging working conditions, and identifies various industrial oil nano-additives with reference to the appropriate options in diverse working environments. Furthermore, the existing problems of nano-additives and their application prospects are summarized. This review, hopefully, would help to shed light on the design and synthesis of novel high-performance nano-additives and promote their application in engineering.

Keywords: nanomaterials; lubricant additive; lubrication mechanism; research progress

1 Introduction

Since the industrial revolution, science and technology as well as industry of human society have developed rapidly. In today's world of energy shortage, friction and wear have always received great attention. Research published by Holmberg in 2017 shows that 23% of the world's energy losses are attributed to tribological contacts, and the vast majority of the energy losses is used to overcome friction [1]. For example, high friction and wear of engine components leads to a significant drop in the power of automobiles, and nearly one-fifth of the power generated by the engine is consumed to overcome friction [2, 3]. The friction and wear of mechanical parts are important causes of energy efficiency loss and failure of mechanical equipment, and they also cause extremely serious pollution to the environment as well as incalculable economic losses [4, 5], which needs to be solved urgently in today's world.

In order to overcome friction and wear, achieve energy saving and environmental protection, and improve the performance of machinery and equipment, we have lubrication as one of the effective strategies.

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In recent years, lubrication technology has developed rapidly worldwide. Unfortunately, traditional lubrication additives often cannot meet the needs of machinery and equipment in current era [6], not to mention that they produce toxic soot nanoparticles rich in sulfur and phosphorus harmful to environment and human health [7]. In this sense, nanoparticles whose sizes in all dimensions are less than 100 nm could be of particular significance in the field of lubrication, because they can easily enter the friction contact zone to keep off the rubbed metal surfaces and greatly improve the tribological properties of lubricants [8–10]. At present, water-based, bio-oil-based, vegetable oil-based, and diesel-based nanoscale lubrication additives are available; and they can function to reduce the friction and wear of frictional pairs via surface deposition and tribofilm formation [11–15], ball bearing effect [16–18], self-repairing effect [19, 20], polishing effect [21, 22], and synergistic effect [23]. In addition, the dispersion stability of nanoparticles in lubricating base oil and their microstructure (e.g., morphology and particle size) are important factors that affect the lubrication effect of nanoparticles as lubricant additives. Selecting nano-additives suitable for specific service environments (such as lubrication condition and harsh working condition) and specific basic lubricants can greatly improve the lubrication service efficiency of nano-additives.

2 Types of nanoscale lubricating additives

2.1 Carbon

Carbon materials have played a vital role in the course of human history. Since the twentieth century, researchers have discovered some nanoscale carbon materials (their size in at least one dimension is below 100 nm). After years of research and exploration, researchers have found that carbon nanomaterials have better physical, chemical, and mechanical properties, as well as a wide range of morphological characteristics, efficient resistance and thermal conductivity than the microscale counterparts. In fact, carbon nanomaterials have been widely studied in the fields of optics, mechanics, tribology, electrochemistry, and biology [24–26]. In terms of various challenges of today's world, such as the depletion of mineral resources, the indirect shortage of supply and the environmental pollution caused by metal minerals, abundant carbon elements could have great potential for replacing metal materials [27]. Particularly, carbon nanomaterials as sustainable solid lubrication additives have attracted great interest in the field of tribology, due to their stable tribological properties and promising applications. Differing from traditional lubricating oil additives rich in sulfur, phosphorus and zinc that are harmful to environment, carbon nanomaterials do not produce toxic emissions to pollute the environment and can maintain efficient long-term anti-wear performance [28, 29].

What should be noticed is that carbon nanomaterials as lubricating oil additives are liable to agglomerate, because of their high surface energy and surface area. This means that the stable dispersion of carbon nanomaterials in base stocks is essential for them to exert excellent tribological properties [30]; and it is often necessary to achieve the stable dispersion of carbon nanomaterials by making use of physical and/or mechanical stirring and surface modification as well as incorporating dispersant [31]. This section highlights several typical carbon nanomaterials with potential applications in lubricant, including fullerenes, carbon nanotubes, nanodiamonds, and graphene (Fig. 1).

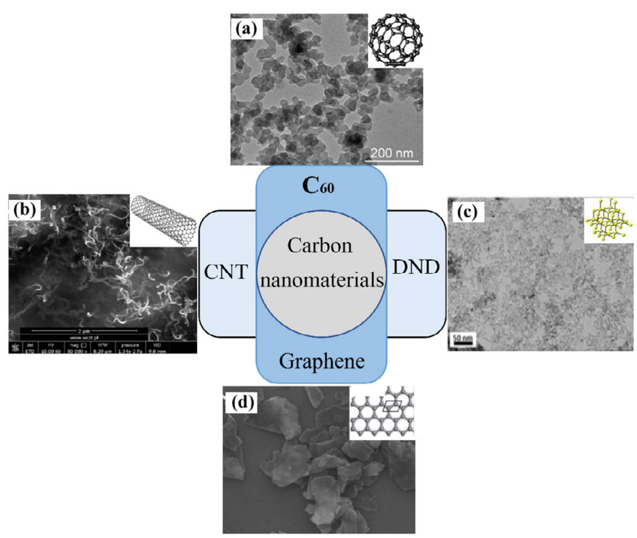

Fig. 1 Transmission electron microscopic (TEM) images of carbon nanomaterials such as (a) fullerenes C_{60} [32], (b) carbon nanotubes [33], (c) nanodiamonds [34], and (d) graphene [35]. Reproduced with permission from Ref. [32] for (a), © Springer 2017; Ref. [33] for (b), © Elsevier 2020; Ref. [34] for (c), © Elsevier 2017; Ref. [35] for (d), © Elsevier 2018.

In the mid-1980s, Harold Waterl Croto, a British doctor of chemistry, and Richard Smalley, an American chemist, prepared the world's first fullerene one-dimensional quantum dots. Since then, fullerenes have been widely used in materials science and tribology [30, 36] as well as photovoltaic field [37, 38], composite material field [39], medicine, sensor, and many other industrial fields. Currently available methods for preparing fullerene include combustion method, arc method, laser evaporation, laser combustion, microwave method, and so on. Among them, the microwave method (based on cable-type soot purification process) and arc method are facile and convenient for cost-effective industrial production of fullerene [40].

Song et al. [41] found that C_{60} fullerene with special spherical structure has good wear resistance as a lubricating material, and its unique onion-like crystal structure is favorable for increasing the load-bearing capacity of the lubricant. Yao et al. [42] prepared onion-like fullerene spheres (OLFs) with a diameter of about 25 nm and evaluated their tribological properties as the additive of Great Wall SE 15W/40 commercial lubricant; and they found that OLFs can improve the antiwear performance of base oil. Huang [30] et al. said that fullerene nanoparticle with a proper concentration (mass fraction 0.02%) in HM32 antiwear lubricant exhibits excellent friction-reducing, antiwear, and, extreme pressure properties. These researches demonstrate that the zero-dimensional quantum dots of C_{60} nanomaterials have excellent antiwear ability and load-bearing capacity in tribological applications. However, they have a high cost as well as poor dispersion stability in base oils and poor adhesion to rubbed metal surfaces, which is unfavorable for them to exert tribological effect. Besides, the antiwear mechanism of C_{60} nanomaterials awaits further studies; and it is worth developing other fullerene-like nanomaterials through controllable methods in order to broaden their application in tribology.

Similarly, zero-dimensional diamond quantum dots are of significance, due to their high hardness, non-toxicity, good chemical inertness and excellent optical and mechanical properties [43–45]. In fact, nanodiamond is an indispensable material in photonic devices [46], antiwear coatings [47], electrochemical coatings, and anti-wear agents [48], as well as drug delivery [49], auxiliary radiology, labeling, bioimaging, catalysis and other fields. As a lubricant additive, nano-diamond has excellent mechanical, thermal, anti-wear and dielectric properties. Ivanov and Shenderova [48] found that detonation nanodiamond (DND; Fig. 2(a)), a "green" all-carbon additive, can effectively improve the performance of lubricating oil and fuel economy and exhibits better lubricating performance when it is combined with conventional

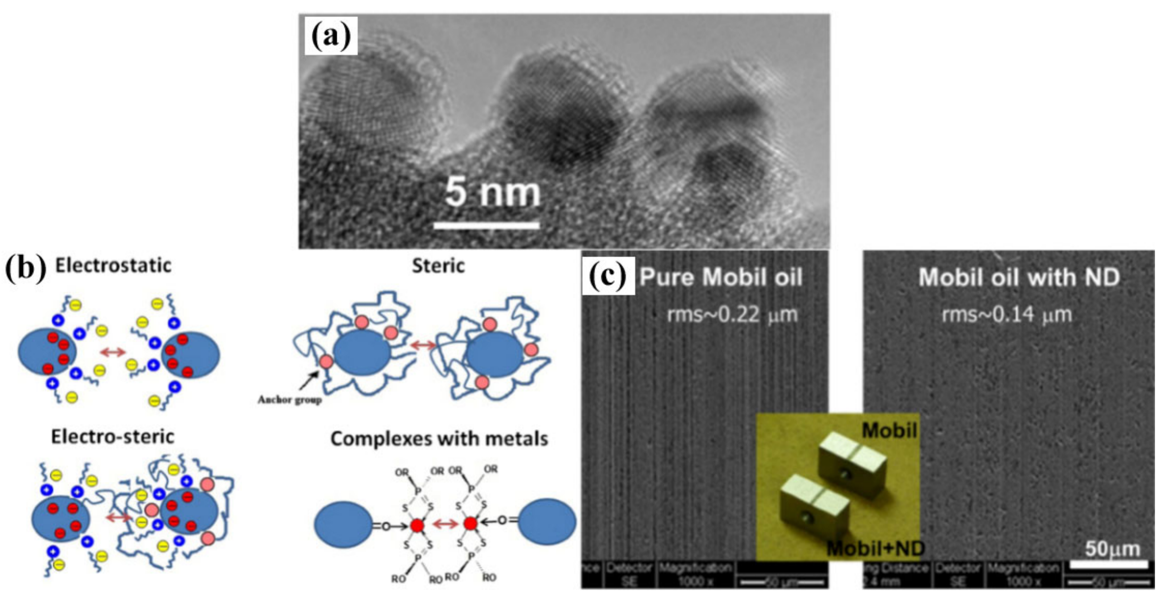

Fig. 2 (a) TEM micrograph, (b) method of maintaining stability of DND dispersion, and (c) SEM images of worn surfaces lubricated by base oil and base oil-DND. Reproduced with permission from Ref. [48], © Elsevier 2017.

engine oil additive zinc dialkyldithiophosphate (ZDDP), thanks to its stable dispersion therein with a concentration of 0.01%–0.10% (mass fraction; Figs. 2(b) and 2(c)).

Golchin et al. [50] found that nano-diamond in pure water can significantly enhance the tribological properties of ultrahigh molecular weight polyethylene composites. Lee et al. [34] modified the surface of nano-diamond with oleic acid to achieve the stable dispersion in the base oil and acquired excellent friction-reducing and antiwear performances. So far, nanodiamond compounds, DND lubricant films, and DND composites have been attracting much attention, especially due to their great potential for tribological applications. However, DND as lubrication additive still faces challenges with its extremely high cost and poor dispersion stability in lubricant as well as limited recognition of its tribo-mechanism.

Carbon nanotubes (CNTs) were first discovered by Professor Iijima in the 1990s; and they can be divided into single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) according to the number of the outer layers of the tube wall. As a kind of unique one-dimensional carbon nanomaterial, CNTs have attracted much attention from researchers, due to their high strength, light weight and excellent thermal conductivity. To date, CNTs are one of the hotspots in the field of lubrication, due to their excellent lubrication and anti-wear properties [51–54]. Nevertheless, CNTs with a high concentration in the base oil would tend to agglomerate spontaneously. Kałużny et al. [33] found that a trace of carbon nanotubes added in engine oil (mass fraction: 0.03%) is of good dispersion state and can significantly reduce engine friction and the vibration of engine cylinder block in the direction of piston transverse force. Bai et al. [55] said that the introduction of a small amount of CNTs in the lubricating base oil can significantly reduce the friction and wear of the sliding pair. Su et al. [56] tested the tribological properties of multi-walled carbon nanotubes (NWCNTs) with different sizes and volume fractions as the additives in LB2000 vegetable oil and found that thin-walled and short-walled carbon nanotubes have better friction-reducing and anti-wear properties than thick-walled and long-walled carbon nanotubes. A recent study reveals that NWCNTs as the additive in engine oil (SAE 10W-40) can reduce the friction coefficient under different loads by up to 34%, which is due to the peeling characteristics of the tested NWCNTs [57]; and relevant details about the characterization of the tested NWCNTs are presented in Fig. 3.

Carbon nanotubes have a unique one-dimensional tubular structure, efficient self-lubricating properties, and significant advantages as friction-reducing and anti-wear agents. In recent years, great progress has been made in the research on the preparation process and application of carbon nanotubes. However, the dispersion stability of carbon nanotubes, their competitive adsorption with lubricating base oil, their lubrication mechanism, and the peeling characteristics of MWCNTs still await further studies.

In the early 2000s, Andrey Gaim and Konstantin Novoselov from the University of Manchester of UK successfully separated graphene from graphite, making one of the major discoveries of the 21st century. As a representative of typical two-dimensional carbon nanomaterials, graphene is widely used in electronics, aerospace, biology, and many other fields, due to its unique two-dimensional layered structure, excellent thermodynamic properties and electrical properties, and a large specific surface [58]. As an excellent lubricating material, graphene has attracted extensive attention in the field of tribology in recent years. A large number of graphene family materials and their nanocomplexes have been prepared and studied as lubricant additives [59, 60].

Zhao et al. [35] studied the tribological properties of graphene with different layer number and layer spacing as the lubricant additive and found that graphene with a higher degree of peeling exhibits better lubrication performance. This means that the original peeling degree of graphene plays a crucial role in its structure evolution; and the graphene with ordered structure as well as its conversion to graphite contributes to enhancing the lubricity. Particularly, the tribofilm formed on the metal friction contact surface is parallel to the sliding direction, which indicates that the graphene with a high degree of peeling does slide thereon. Liang et al. [61] used jet method to prepare directly peeled graphene as a lubricating additive for pure water and tested its

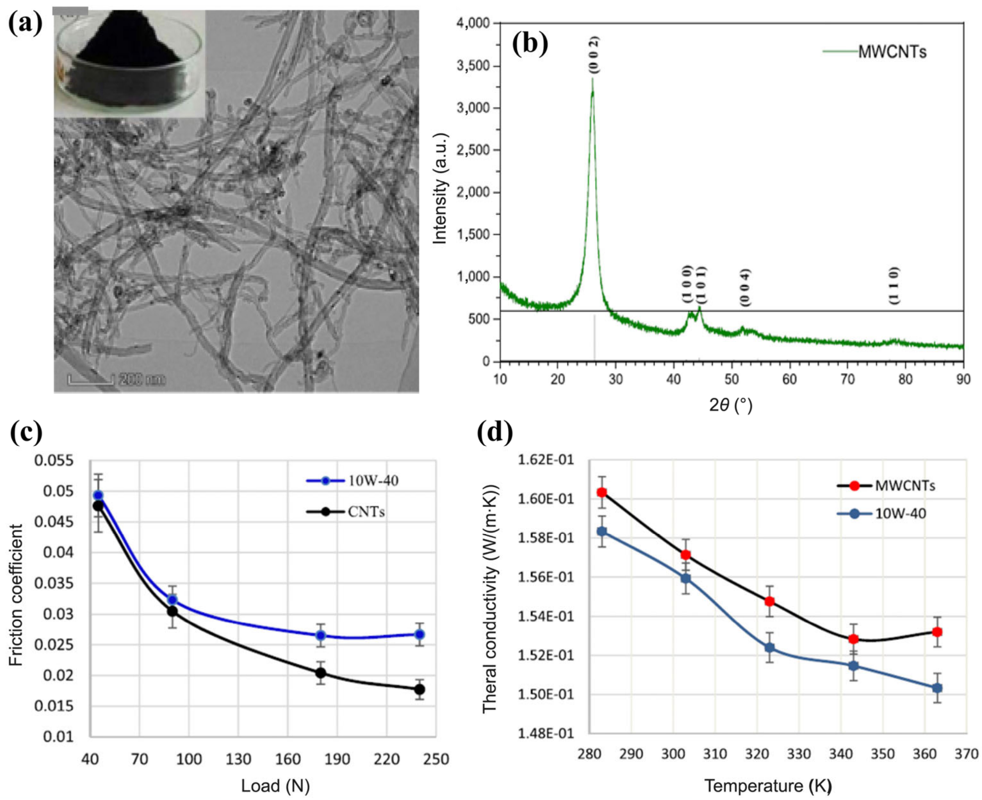

Fig. 3 (a) TEM microstructure and (b) XRD pattern of the tested NWCNTs as well as (c) Friction coefficient versus applied load under the lubrication of the engine oil containing NWCNTs and (d) thermal conductivity experimental results for the base oil and base oil-MWCNTs. Reproduced with permission from Ref. [57], © IOP Publishing Ltd. 2021.

tribological properties with a UMT friction and wear tester. They found that adding graphene nanosheets could reduce the friction coefficient by 22.8% and wear rate by 44.4%. Xie et al. [62] tested the tribological properties of multiple layer graphene as the lubricant additive in natural wax and found that the additive can improve the bearing capacity and high temperature lubrication performance of natural wax. Li et al. [63] reported that graphene with a mass fraction of 0.1% in lithium grease can significantly reduce the friction coefficient and wear scar diameter under different loads (Fig. 4).

At present, graphene as an environmentally friendly lubricant additive with excellent performance is widely used in the field of nano-lubrication. Needless to say, the dispersion stability of graphene in lubricant base oil is always an important issue; and it could be imperative to achieve the self-dispersion of graphene in the lubricant base oil by properly tuning its morphology and size. Unfortunately, few are currently available about the large-scale industrial production of high-performance graphene and its compounds. In this sense, it is urgent to establish novel economical, green and efficient industrial preparation method of graphene in order to promote its commercialization; and it is also imperative to further study the microscopic lubrication mechanism of graphene-based nanomaterials thereby adding to tribological theory.

In summary, carbon nanomaterials have promising potential in the field of lubrication. Environmentally friendly carbon nanomaterials are in line with the concept of green development in today's world and will occupy a huge market in the future. However, with the development of mechanical systems, the requirements for friction contact surfaces and interface properties are more stringent, the working environment for lubrication additives is more demanding, and the application of carbon nanomaterials as novel lubricant additives still face great challenges. In terms of the future development of carbon nanomaterials, it is imperative to further study their dispersion stability in lubricant base oil and adsorption capacity on

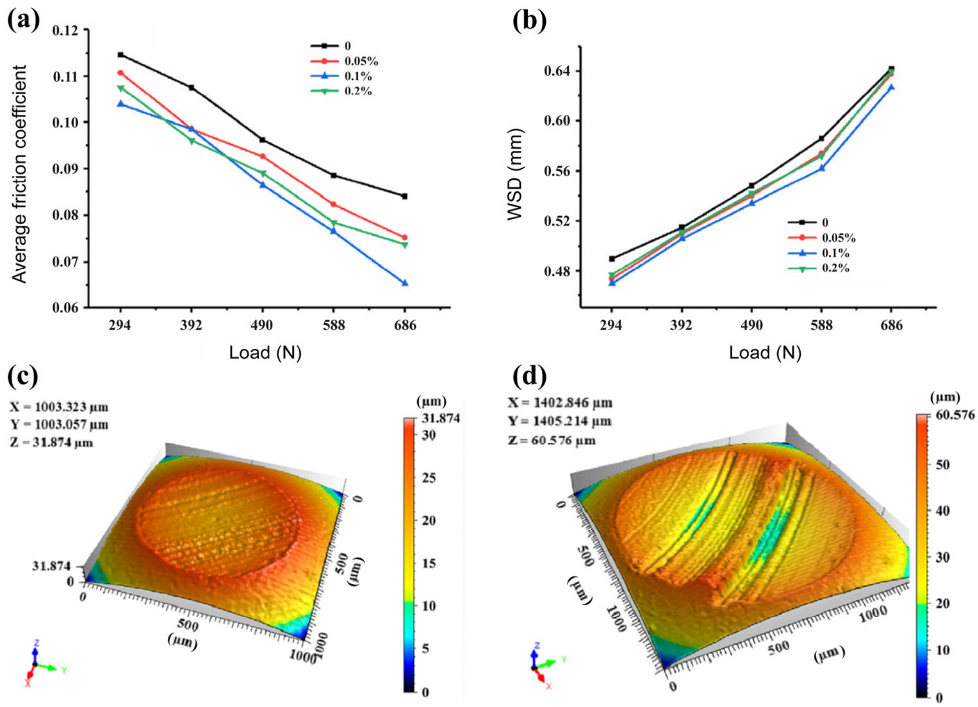

Fig. 4 Variation of (a) average friction coefficient and (b) wear scar diameter (WSD) under different loads as well as three-dimensional (3D) topography of worn metal surfaces lubricated by (c) base grease and (d) base grease containing 0.1% graphene. Reproduced with permission from Ref. [63], © Wiley 2020.

rubbed metal surface, their lubrication mechanism and large-scale production method as well, thereby promoting their application in industry.

2.2 Metal

The special physical and chemical properties of metal nanoparticles make them excellent lubricant additives [64–66]. Particularly, some metals (e.g., Ag and Cu) as lubricant additives exhibit excellent anti-wear and film formation abilities as well as desired self-repairing performance, due to the grain boundary slip effect ascribed to their relatively low shear strength and melting point as well as high elongation capacity. For example, silver nanoparticles have high chemical stability, rich synthesis methods, and excellent lubricating potential [72]. The commonality of metal nanoparticles is the premise of their use as lubricant additives, and their lubrication potential highly depends on their microstructure and characteristics. Table 1 gives some examples of recent studies on metal nanoparticles as lubricant additives.

We previously investigated the use of nano-sized copper, nickel and other metal nanoparticles as lubricating additives and realized the industrial production of oil-soluble nano-sized Cu as lubricating additive with excellent performance in 2012. The as-prepared nano-Cu has good compatibility with lubricant base oil as well as good comprehensive performance after combination with commercially available lubricants, due to synergistic tribo-effect. Besides, the as-prepared nano-Cu can be deposited on rubbed metal surfaces to form lubricating film and fill up the grooves and scratches thereon, thereby achieving self-repairing of the metal–metal sliding pair. Zhao et al. [10] prepared water-soluble copper nanoparticles (diameter 3 nm) by using an in situ surface modification technology. The as-prepared water-soluble copper nanoparticle with a concentration of 0.6% (mass fraction) in distilled water can significantly reduce the friction coefficient and wear rate (Figs. 5(a), 5(b) and 5(c)), which is related to its good ability to accelerate the heat transfer of the friction pair. Guo et al. [73] found that there is a synergistic effect between copper nanoparticles and conventional lubricant additives like diisooctyl sebacate (DIOS), which is favorable for improving the tribological properties of the lubricant base oil. Kumara et al.

Table 1 Some recent studies on metal nanoparticles.

Metallic nanoparticle	Published journal	Time	Research unit	Function
Cu [67]	*Tribology International*	2018	Federal University of Rio de Janeiro	Improve the friction-reducing and anti-wear performance of mineral oil
Cu [68]	*Lubrication Science*	2022	Lanzhou University	Improve the friction-reducing and anti-wear performance effect of PAO6 base oil and 10W\40CH-4 industrial lubricating oil
Ti [69]	*Applied Surface Science*	2022	Centre for Automotive Research & Tribology	Excellent anti-wear ability in base oils
Ni [70]	*ACS Applied Nano Materials*	2021	Henan University	In situ self-repair performance for steel sliding pair
Fe [71]	*Science Direct*	2018	Toyohashi University of Technology	The friction coefficient in ester oil is reduced by controlling the microstructure of Fe nanoparticles
Al [16]	*Applied Sciences*	2017	National Kaohsiung University of Applied Sciences	Reduce the friction and wear of the sliding pair lubricated by glycerol aqueous solution
Ag [72]	*ACS Applied Materials & Interfaces*	2017	Oak Ridge National Laboratory	The dispersion in PAO results in a very thick tribofilm, thus reducing the friction

[72, 74, 75] conducted a series of studies on silver nanoparticles and palladium nanoparticles. They said that silver nanoparticles modified by benzyl thiol and dodecanethiol can be stably dispersed in lubricant base oil to reduce wear by 85% under boundary lubrication condition; and dodecanethiol-modified Pd nanoparticles added in PAO oil are able to generate a thick protective film (2 ~ 3 mm) on the rubbed metal surfaces to reduce friction coefficient and wear rate by 40% and 97%. Le et al. [16] confirmed that aluminum nanoparticles added in glycerol aqueous solution (containing sodium aluminum ion with the best concentration) play an important role in the improvement of friction-reducing and anti-wear performance (Fig. 5; four-ball machine, load 90 N).

A large number of studies on metal nanoparticles have confirmed that single metal nanoparticle can indeed improve the tribological properties of base lubricants, and the research on metal nanoparticles as lubricant additives is relatively mature. There have been many reports on the improvement of the preparation methods of metal-based nanoparticles with respect to mass production and commercial use. In terms of the existing problems and future research directions of metal nanoparticles, it is suggested to focus on finding more cost-effective dispersion methods, studying the adaptability of metal nanoparticles to different basic lubricants and friction interfaces, developing novel high-performance metal-based composite nanomaterials, and exploring the self-dispersion methods for metal nanomaterials.

2.3 Sulfide

In recent years, sulfide has been widely used as a nano-lubricant additive. Transition metal dihalides (TMDC) such as WS_2 and MoS_2 are representative sulfides; and they have attracted great attention, due to their typical two-dimensional layered structure and good chemical stability [76–80]. Many studies show that nanoscale MoS_2 with various shapes (nanotubes, nano-onion-like, platelet-like, etc) can be used as lubricant additives [81, 82]; and the excellent tribological properties of WS_2 and MoS_2 nanoparticles are ascribed to their good ability to form protective film on rubbed metal surfaces [83–88].

Wu et al. [89] synthesized an organic-MoS_2 hybrid by surface modification and found that the as-prepared hybrid as the additive can significantly improve the load-bearing capacity and high temperature lubrication performance of PAO10 base oil. Wang et al. [87] said that thiol-modified functional MoS_2 nanosheets exhibit good dispersion stability in water as well as excellent

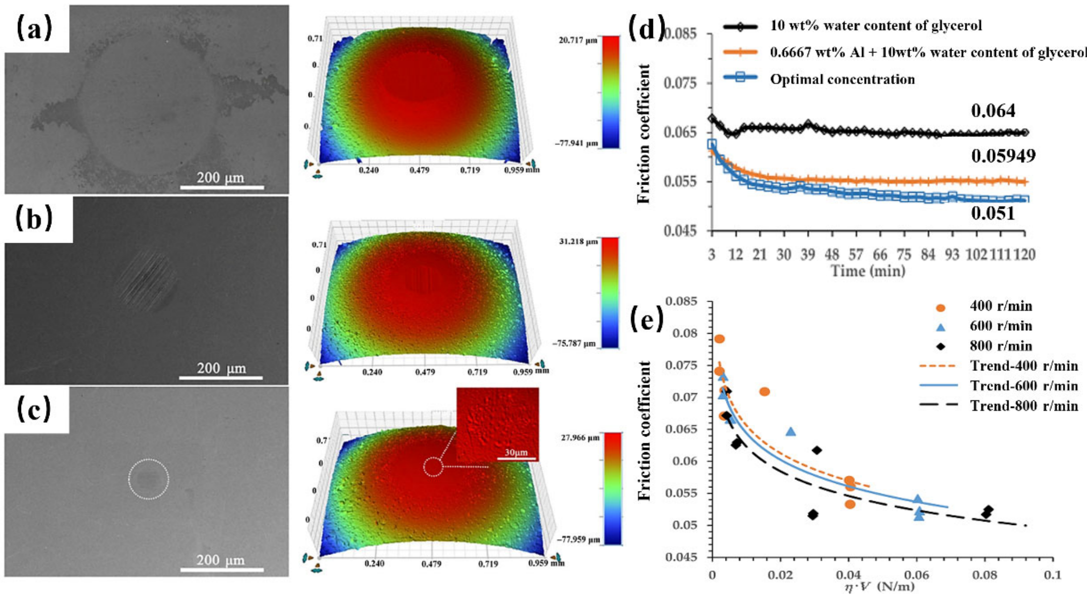

Fig. 5 SEM and 3D contour of worn surfaces under the lubrication of (a) distilled water, (b) commercial lubricant, and (c) distilled water containing 0.6% nano-Cu, as well as (d) variation of friction coefficient with Al concentration and (e) Stribeck curves at different speeds. Reproduced with permission from Ref. [10] for (a–c), © Tsinghua University Press 2019; Ref. [16] for (d, e), © Balkan Society of Geometers 2017.

tribological properties. Similar to MoS_2, nano-sized WS_2 also has great lubricating potential in harsh environment [90].

In our previous researches, we prepared WS_2 nanoparticles modified by oleylamine via liquid-phase in situ surface modification at elevated temperature and found that the surface-capped WS_2 nanoparticles exhibit good dispersion stability in lubricating oil and can greatly improve the tribological properties of the base stock [91–93]. Particularly, they can significantly improve the tribological properties of PAO6 base oil at elevated temperatures of up to 300 ºC (Fig. 6), being superior to traditional lubricating additives like zinc dialkyldithiophosphate (ZDDP). When WS_2 nanosheets are modified by oleamine containing carboxyl group, their surface polarity is increased, which is favorable for enhancing their adsorption on rubbed metal surfaces thereby exerting good friction-reducing and anti-wear abilities in ester oil. Similar to oleamine, maleic anhydride dodecyl ester as the modifier also can significantly improve the tribological properties of WS_2 nanoparticles as the additive in ester oil.

Liu et al. [94] said that CuS nanoparticles coated with surfactants can be stably dispersed in diethyl succinate lubricant and exhibit good friction-reducing effect, which is because they can be enriched on rubbed metal surfaces especially under externally applied electric field. Zhao et al. [95] prepared water-soluble CuS nanoparticles by surface modification and found that the as-prepared water-based additive can effectively improve the tribological properties and thermal conductivity of distilled water. Wang et al. [96] prepared nanoscale ZnS by simple precipitation method with $Zn(CH_3COO)_2·2H_2O$ as precursor and polyethylene glycol monomethyl ether disulfide as modifier and found that the as-prepared ZnS nanoparticle can significantly improve the anti-wear and friction-reducing properties of lubricant.

In terms of structure, WS_2 consists of layered S–W–S unit, and each unit layer contains a layer of tungsten atoms and two layers of sulfur atoms connected by covalent bonds. The unit layers are combined through van der Waals forces. Similarly, molybdenum disulfide crystals also have a layered structure, with a layer of molybdenum atomic planes sandwiched between two layers of sulfur atom planes; and the molybdenum disulfide planes are connected by van der Waals forces. CuS, on the other hand, crystallizes in a hexagonal crystal system with six CuS (12 atoms) in a unit cell, where disulfide bonds (S–S) are present. WS_2 and MoS_2 nanoparticles are believed to possess better lubricating performance than CuS, because

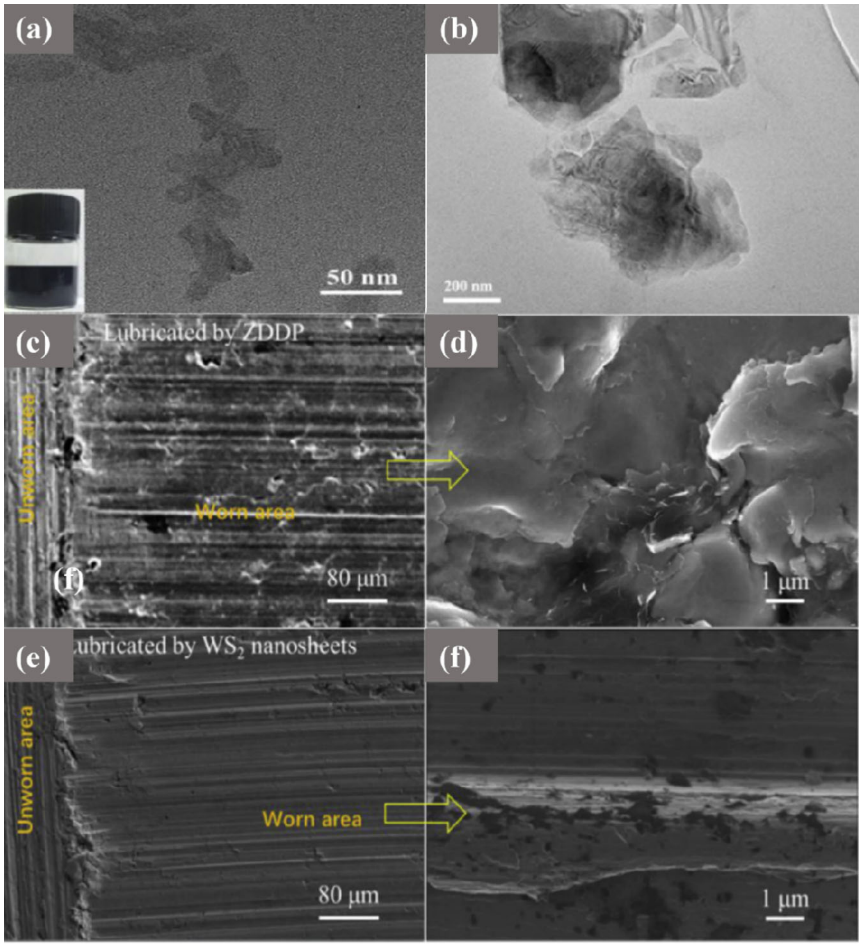

Fig. 6 TEM images of (a) WS_2 and (b) MoS_2 as well as SEM pictures of worn surfaces of cylinder liner lubricated by (c and d) PAO6 with 1.75% ZDDP and (e and f; OA refers to oleic acid) PAO6 with 1.00% OA-modified WS_2 nanosheets at 150 °C. Reproduced with permission from Ref. [89] for (b), © ACS 2018; Ref. [91] for (a, c–f), © Elsevier 2019.

their sheet structure is favorable for facilitating the sliding of adjacent layers under shear force to result in better anti-wear and friction reduction abilities as well as load-bearing capacity. Besides, WS_2 has very good stability at high temperatures, which is favorable for its use thereat [97]. Moreover, it often needs to exert surface modification of sulfide nanomaterials in order to improve their dispersion stability in lubricants; and in this respect various surface modifiers and bonding forms could be utilized.

In summary, nano-WS_2 and nano-MoS_2 as lubrication additives have attracted great attention in the field of lubrication. At present, TMDC has been proven to have excellent friction-reducing, anti-wear, and extreme pressure properties. For example, MoS_2 and WS_2 nanosheets can adapt to different base lubricants (such as mineral oil, vegetable oil, synthetic oil, grease, water) and effectively improve the tribological properties of base oils under high temperature and direct current (or magnetic) field [92, 98]. Other metal sulfides such as CuS and ZnS as nano-lubricating additives also have excellent tribological properties and promising potential in lubrication engineering. In future studies on nanosulfides as lubricant additives, it is suggested to pay more attention to investigate their tribological properties and lubrication mechanism under harsh working conditions, their dispersion behavior upon changing morphological feature, and their combination with other nanoparticles for developing novel high-performance nano-additives.

2.4 Metal oxides

In recent years, the studies on metal oxide nanomaterials as lubricant additives have also attracted much attention. For example, nanoscale CuO [99–102], ZnO [17, 103, 104], CeO_2 [105, 106], SiO_2 [21, 107–109], and TiO_2 [110] have been extensively studied by scholars at home and abroad. The researches of metal oxide nanomaterials as lubricant additives mainly focus on their dispersion stability in lubricant base stocks, their thermophysical characteristics (thermal conductivity, kinematic viscosity, flash point, pour point), their best concentration for enhancing the friction-reducing and anti-wear effect, their load-bearing capacity, and other characteristics. At present, their poor dispersion stability in organic lubricants is one of the major disadvantages of metal oxide nanoparticles.

He et al. [106] added nano-CeO_2 to lithium base grease and evaluated the tribological performance. By observing the worn surface morphology with scanning electron microscope (SEM) and NANOVEA 3D profilometer, they concluded that the addition of 0.6% nano-CeO_2 in the lithium base grease can reduce friction coefficient and wear scar diameter to some extent. Wu et al. [111] used novel TiO_2 as an additive for water-based lubricants and studied its lubricating properties and mechanism. They found that TiO_2 nanoparticles can significantly reduce the friction coefficient of water-based lubricants. Rawat et al. [109] prepared SiO_2 nanoparticles by improved sol-gel synthesis method and evaluated the friction-reducing and anti-wear properties in a thickened paraffin oil. They said that adding SiO_2 nanoparticles in the paraffin lubricating oil can reduce friction coefficient and wear scar diameter by 20% and 42%, respectively. Zhang et al. [104] prepared nanometer ZnO modified with dialkyl dithiophosphate by one-step method; they found that the surface-capped ZnO nanoparticles not only can overcome the poor lubrication performance of ZDDP for aluminum frictional pair but also have less sulfur and phosphorus content (reduced by about 80%) than ZDDP. Similarly, ZnO nanoparticle modified with dialkyl dithiophosphate (ZODDP) as the lubricant additive of DIOS base oil exhibits better tribological properties than ZDDP (Fig. 7), showing promising prospect as the lubricant additives for aluminum-base materials. Wu et al. [112] prepared oleamine modified cerium oxide nanoparticle (OM-CeO_2) by one-pot pyrolysis method and tested its tribological properties with a four-ball machine. They said that OM-CeO_2 dispersed in PAO lubricating oil can significantly improve the anti-wear and extreme pressure properties, which is due to the formation of tribofilm.

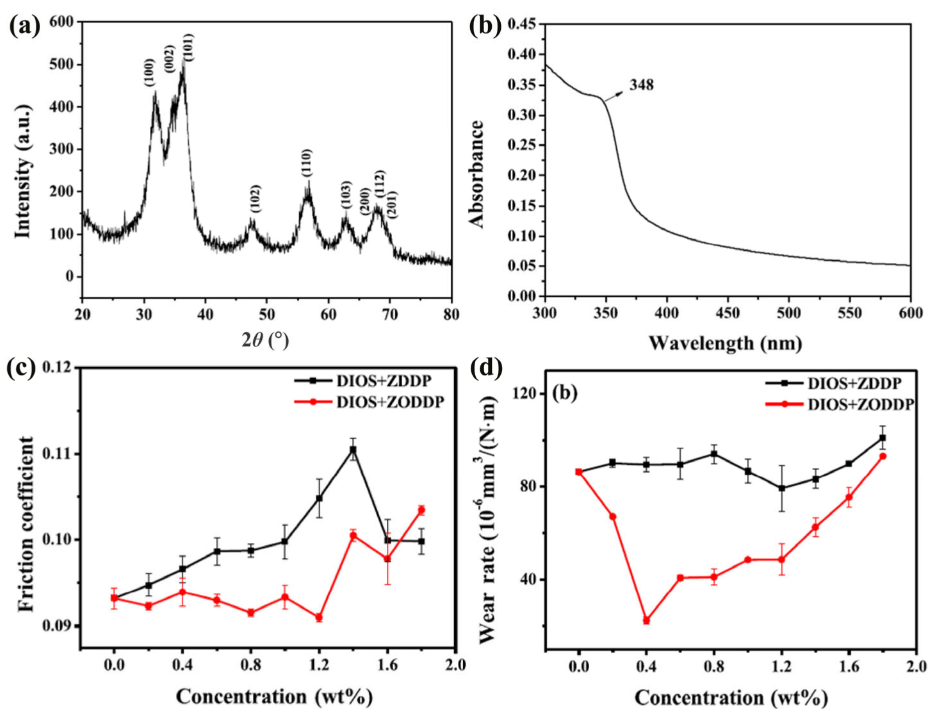

Fig. 7 (a) XRD pattern and (b) UV-vis absorbance spectrum of ZODDP as well as (c) variation of friction coefficient and (d) wear rate with concentration of ZDDP and ZODDP in DIOS. Reproduced with permission from Ref. [104], © Elsevier 2020.

Nano-oxides have highly efficient tribological properties and are well adaptable to pure water, oil-based lubricants, and grease-based lubricants. Changing the compatibility of nano-oxides with base lubricants by surface chemical modification is the key to improving their tribological properties; and the magnetism of nano-oxides themselves is favorable for their adsorption on the surface of frictional pair. Particularly, the combination of nano-oxides with other nanoparticles is of significance for the development of novel nanoscale composite lubricant additives possessing desired comprehensive properties.

2.5 Polymer

Polymers have excellent practical properties such as good corrosion resistance, low density, high strength, and high temperature resistance. In the meantime, polymers' lower weight, lower friction noise, and better self-lubricating performance in comparison to other nano-lubricant additives could be significant for getting rid of the seizure of metal-to-metal friction pairs at abnormally high loads and/or low viscosity oils in long periods of service [113]. In the field of tribology, researchers have been exploring and applying polymers for a long time. Current researches on polymers as nano-lubricating additives mainly focuses on the following aspects: the lubricating effect of polymer itself, the modification of polymer matrix by inorganic nanoparticles, the modification of lubricating base oil, the synergistic effect of polymer with other nanoparticles, and the preparation of polymer-based complexes.

Polytetrafluoroethylene (PTFE) as a typical polymer lubricant additive can significantly improve the wear resistance and load-bearing capacity of lubricating base oil. Li et al. [114] prepared poly(propyl 3-methacrylate sulfonate potassium salt-co-styrene) amphiphilic polymer nanosphere with soft/hard coupled structure and found that the as-prepared amphiphilic polymer nanosphere as a water-based additive exhibits excellent friction-reducing and anti-wear behavior as well as a high load-bearing capacity and a strong potential in biological lubrication. Murdoch et al. [115] demonstrated that an amphipathic functionally alkylated copolymer can reduces friction in the boundary region. Aharonovich et al. [116] developed a kind of single-chain polymer nanoparticle as a shear elastic viscosity regulator of lubricating oil; and they found that the product can make the viscosity of solution permanently increase with rising shear and greatly improve the performance of lubricating oil. Feng et al. [117] put forward that, as biological lubricating additive, polystyrene (PS) nano-ball with surface grafted hydrophilic polymer can form a thick layer of hydration, thereby reducing the friction and wear, and can withstand strong shear forces for a long period of time (Fig. 8).

Polymers usually increase the viscosity of base oil, but the effect of the same skeleton length on the rate of change in viscosity versus temperature can vary based on their composition and structure. The mechanism by which polymer works depends on both the chemistry and structure of the polymer itself and the base oil [118]. Under harsh operating conditions such as high pressure and high sliding speed, pure polymers tend to generate high wear and friction, due to their low load-bearing capacity ascribed to low viscosity and chemical inertness. To enhance the tribological properties of polymers under water lubrication, researchers often utilize two main methods: adding reinforcing agents to the polymer matrix and incorporating functional additives into water [119, 120].

Hydrogels are an important class of polymer materials consisting of a three-dimensional cross-linked polymer network with a high water content. Due to their good biocompatibility and biomimetic properties, hydrogels have garnered great attention in the field of biomedicine [121]. Improving mechanical properties and enhancing anti-wear and anti-friction effects are important research directions for the application of hydrogel materials in lubrication. Rudge et al. [122] showed how soft hydrogel nanoparticle suspensions can act as excellent lubricants via micro-bearing effect in friction pair clearances. Shoaib et al. [123] studied the friction characteristics of polypropylamide (PAAm) hydrogels of different concentrations and revealed two boundary lubrication mechanisms with different laws, which could be enlightening for use to better correlate the hydrogel lubrication mechanism and slip friction. Feng et al. [124] synthesized poly(NIPAAm-co-AA) microgel

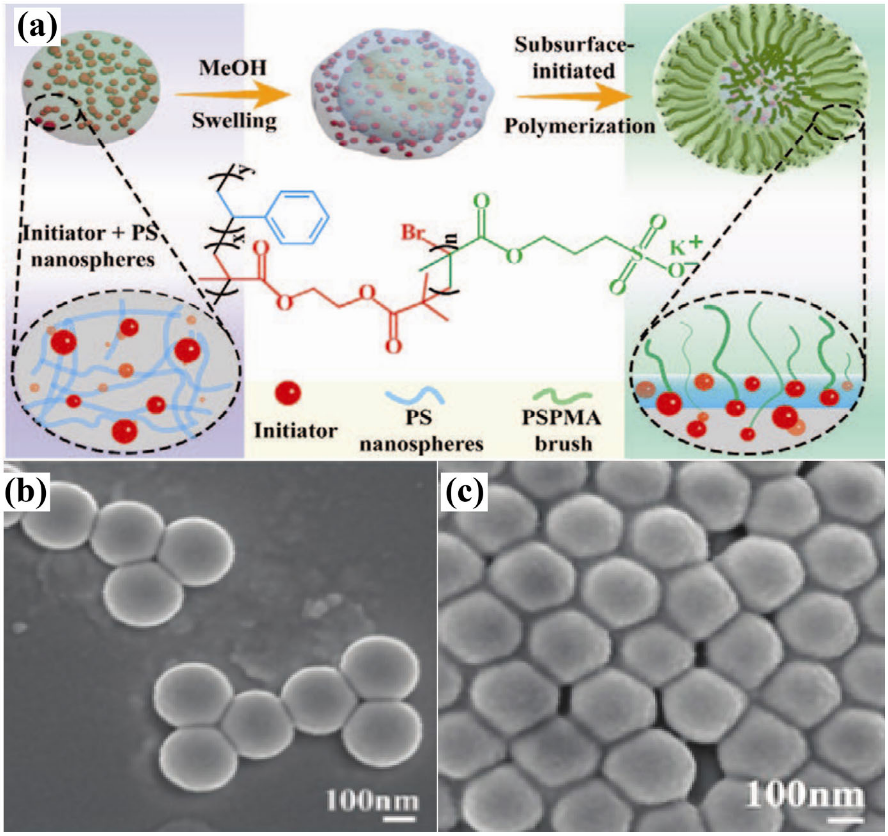

Fig. 8 (a) Schematic diagram of PS nanospheres prepared with a subsurface initiated graft polymer brush and (b, c) their SEM images. Reproduced with permission from Ref. [117], © Wiley, 2020.

via emulsion polymerization and used it as a water-based lubricating additive for Ti6Al4V titanium alloy contact, obtaining a 46% reduction in friction coefficient and a 45% reduction in wear volume compared to pure water; and they said that the hydration layer around the microgel, the micro-bearing effect, the interfacial adsorption, and the colloidal stability jointly determine the anti-wear and friction effect of the poly(NIPAAm-co-AA) microgel under water lubrication. Wang et al. [125] successfully synthesized a copolymer hydrogel composed of sultamine methacrylate (SBMA) and 2-methacryloyloxyethyl phosphocholine (MPC) polymers with ethylene glycol dimethacrylate (EGDMA) as the crosslinking agent. Connected by MPC and SBMA polymer chains, the as-prepared P(MPC-co-SBMA) copolymer hydrogel network is constructed via additional polymerization of alkane carbon–carbon double bonds. As a water-based lubricant additive, P(MPC-co-SBMA) copolymer hydrogel exhibits excellent load-carrying capacity and can significantly reduce the friction coefficient.

Polymer size greatly impacts performance; selecting appropriate preparation methods with a view to the application field and adjusting key experimental parameters can effectively control the particle size and distribution [126]. Rosiuk et al. [127] systematically studied the influence of polymer properties on nanoparticle formation by changing the hydrophobicity and charge of polymers over a wide range. Experimental results demonstrate that introducing hydrophilic and charged groups can reduce the size of nanopolymer particles (minimum 20 nm). Xu et al. [128] successfully prepared N-isoacrylamide (PNIPAM) microgels via surfactant-free emulsion polymerization to improve the lubricating properties of titanium alloys at different temperatures. They investigated the effects of initiator, SDS (sodium lauryl sulfate) concentration, and mixing process on the particle size of spherical PNIPAM microgel and found that potassium persulfate as the initiator, drop-by-drop mixing process (DM), and high SDS concentration contribute to the formation of microgel nanoparticles with small particle sizes. Particularly, PNIPAM microgel with a minimum particle size of 50 nm can be obtained by properly controlling the synthetic conditions.

Samanta et al. [129] prepared functionalized graphene oxide (GO) based polymer brushes by layer assembly and found that the as-prepared polymer brushes can reduce the friction and wear of steel–steel friction pair under different pressures. Agrawal et al. [130] used glass fiber reinforced polymer (GFRP) composite to fill carbon nanotubes. They found that the carbon nanotubes filled with GFRP can improve the friction-reducing and anti-wear effect of lubricating base oil, which is due to the synergistic effect of CNT and GFRP polymer composites. Lu et al. [131] prepared zwitterionic polymer functionalized nitrogen-rich porous carbon nanosheets (denoted as N@PCNS); and they said that N@PCNS exhibits excellent lubrication performance in water, due to the formation of a protective tribofilm via chemical reaction on the rubbed metal surfaces.

As lubricant additives, nanopolymers can improve the friction-reducing, anti-wear, and extreme pressure properties as well as oxidation resistance of the base lubricant, while they have excellent thermal stability and adaptability to harsh working conditions. Particularly, the combination of polymer composites with inorganic nanoparticle provides feasible pathways to prepare composite nano-lubricating additives with desired comprehensive properties.

2.6 Nanocomposites

In recent years, scholars have developed a variety of nanocomposites as lubricant additives. Thanks to the synergy among various components, nanocomposites often may exhibit better tribological properties than each single component as the lubricant additive. In other words, nanocomposites not only can combine the advantages of each single material (such as dispersion stability, tribological performance in high temperature environment, service life in special environment) but also can sometimes possess new properties that are not available in the raw materials. Common nanomaterials such as nano-sized carbon materials, sulfides, metals, and metal oxides can be used as lubricant additives on their own or as synthetic materials for composite nanoparticles. Table 2 lists various nanocomposites reported in recent studies.

Gong et al. [147] prepared nanocomposites MoS_2@CNT, MoS_2@Gr, and MoS_2@C_{60} from nano-sized MoS_2 particles and carbon materials by simple hot solvent method. The as-prepared nanocomposites added in polyalkyl diol (PAG) base oil not only show better dispersion stability than single MoS_2 nanoparticle but also can improve the friction-reducing and anti-wear performance of PAG at high friction temperature, which is attributed to the synergistic effect between carbon nanomaterial and MoS_2. Using laser in situ irradiation method, Luo et al. [133] prepared a layered nanoscale composite material (L-rGO/MoS_2) with multi-layer graphene inlaid with ultra-smooth MoS_2 microspheres. This novel layered lubricating additive combines the advantages of zero- and two-dimensional structural lubricants. Namely, the spherical MoS_2 can change sliding friction into rolling friction under strong shear while the 2D graphene layer can protect the surface of the friction pair from scratches, thereby cooperatively improving the friction-reducing and anti-wear performance (Fig. 9). Cu@graphene composites as lubricant additives

Table 2 Various nanocomposites available in recent publications.

Category	Case
Carbon nanocomposites	GNS@MoS_2 [132], rGO@ MoS_2 [133], Ag@rGO [20], GO@PTFE [134], CMS@PMMA [135], CNS@ ricinoleic acid [136]
Metal nanocomposites	Ag@rGO [20], Sc-Au@GO [137], BN@Cu [138], Cu@SiO_2 [14], Ag@BP [139]
Oxide nanocomposites	Al_2O_3@TiO_2 [140], TiO_2@BP [141], PTFE@$CaCO_3$ [142], Mn_3O_4@Gr [143], G@Fe_3O_4 [144], SiO_2@GO [145], Fe_3O_4 @h-BN [146]
Sulfide nanocomposites	GNS@MoS_2 [132], rGO@MoS_2 [133], ZB@MoS_2 [23], MoS_2@CNT/Gr/C_{60} [147]
Polymer nanocomposites	GO@PTFE [134], N@PCNS [131], GO-PEI-PSS [129]

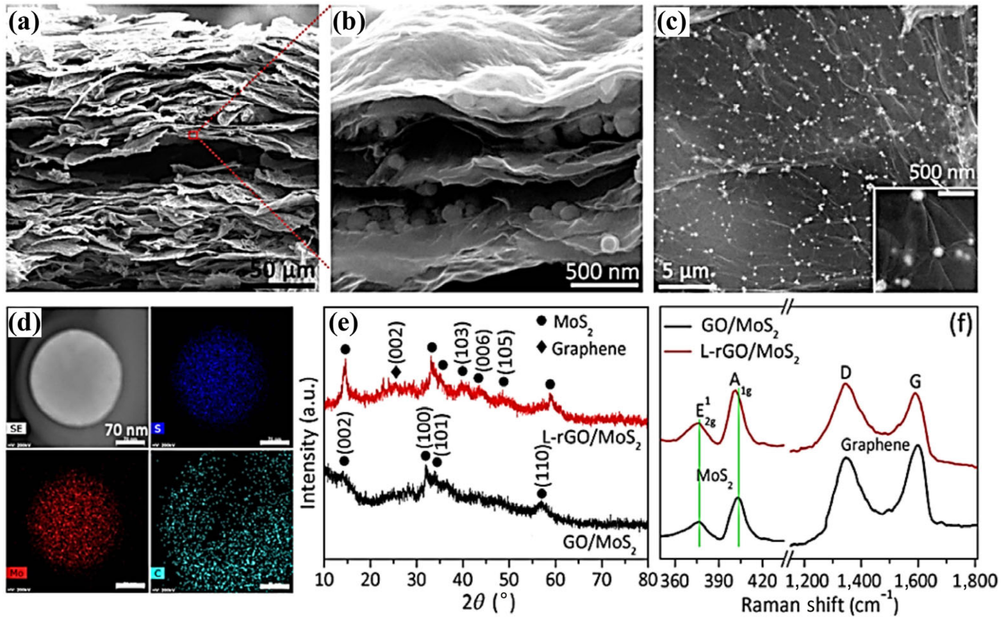

Fig. 9 SEM micrographs of L-rGO/MoS_2 composite structure ((a) front view, (b) section, and (c) top view); (d) element mapping of MoS_2 spheres in the composite structure, as well as (e) XRD patterns and (f) Raman spectra of the original GO/MoS_2 and L-rGO/MoS_2 composite structures. Reproduced with permission from Ref. [133], © IOP Publishing Ltd. 2018.

show excellent tribological properties, and the Cu and graphene interface is connected mainly by electrostatic bonding and chemical bonding [148, 149].

With the continuous efforts of scholars at home and abroad, many composite nanoparticles with excellent tribological properties have been studied and their preparation schemes have been continuously optimized. It has been found that nanocomposites lubricant additives often exhibit better comprehensive properties especially friction-reducing and anti-wear abilities than the single component. However, the preparation of nanocomposites requires more rigorous design scheme, greater economic investment and more complex research process; and their application potential still awaits further exploration.

Various types of nano-lubricating materials described in this paper, such as carbon nanomaterials, nanometals, nanooxides, sulfides and polymers, exhibit excellent tribological properties and dispersion stability under specific conditions. However, based on the properties of each nanomaterial, their mechanism of action in lubricating service is different. In addition, each material has a special tribochemical reaction, which gives them unique advantages in specific applications. For example, carbon nanotubes (CNTs, NWCNTs) as representative carbon nanomaterials have excellent mechanical properties, tensile/flexible and thermal conductivity, and can act as "roller bearings" when entering the friction pair gap, which contributes to improving the anti-wear and friction-reducing abilities as well as load-bearing capacity of the base lubricant. The layered structure of graphene leads to low shear stress, and the relative sliding between layers greatly improves its tribological properties. Due to their relatively low shear strength, melting point, and high elongation, nanometals exhibit excellent anti-wear and film-forming capabilities as well as ideal self-repairing properties. Nano oxide has high surface activity, strong load-bearing capacity, excellent thermophysical properties, and strong adsorption on the surface of friction pair, which is favorable for improving the anti-wear and friction-reducing abilities as well as extreme pressure properties and high temperature resistance of the base lubricant. The excellent tribological properties of nanosulfides are attributed to their good ability to form protective tribofilm. Polymers with light weight and little friction-induced noise as well as good lubricity are widely used in grease and water lubrication. Particularly, polymer microspheres with a spherical structure can function as rolling bearings to form protective tribofilm, which, in combination with their good synergy with inorganic nano-additives, contributed to improved tribological properties.

3 Dispersion of nanoparticles in lubricants

The dispersion stability of nanoparticles in lubricants has always been a very important issue, since the stable dispersion of the inorganic nanoparticles in base lubricant greatly affects the comprehensive properties. The reason why nanoparticles are difficult to disperse stably and uniformly in lubricants is their high surface energy and chemical activity that make them liable to agglomeration in the lubricant. The dispersion stability of nanoparticles in lubricants can be determined by standing precipitation, UV-Vis spectrophotometry, and small-angle X-ray scattering. Lubricants containing dispersed nanoparticles can be significantly different from base lubricants; and visual inspection is widely used to distinguish the aggregation and precipitation of nanoparticles in lubricants, thereby detecting their dispersion stability therein [150].

The addition of dispersants to the suspension often can improve the dispersion stability of nanoparticles. Common dispersants include surfactants, inorganic electrolytes, polymers with good adsorption capacity, etc. If there is no suitable dispersion method to stably disperse nanoparticles, aggregation effects will eventually occur, regardless of their material and shape. Fortunately, the use of dispersants combined with nanoparticles to change the formulation of the lubricant and the surface chemical modification of nanoparticles by organic modifiers often can help to achieve the stable dispersion of nanoparticles in lubricant media [151]. This is because surfactant molecules can be adsorbed on the surface of nanoparticles to make them oleophilic, hydrophilic or ester-friendly, while the surface modification of nanoparticles through chemical reactions can change the surface structure and characteristics of the inorganic nanoparticles thereby getting rid of agglomeration.

3.1 Influencing factors of stable dispersion of nanoparticles

The ratio of the number of surface atoms to the total number of atoms of nanomaterials increases rapidly with the change of particle size, resulting in changes in material properties, which is called the surface effect of nanomaterials. The large number of atoms on the surface of nano-sized particles results in high surface energy and surface area of nanoparticles. Due to the insufficient coordination number of surface atoms and high surface energy, these atoms are easily combined with other atoms to reach a stable state, which refers to high chemical activity of nanoparticles. Nanoparticles as lubricant additives would have quite high intermolecular forces, due to their large specific surface area and surface energy; and untreated nanoparticles would be prone to agglomeration upon dispersion in the base lubricant.

The Stokes equation is written as Ref. [152]:

$$v = \frac{2r^2 g(\rho_1 - \rho_2)}{9\eta} \tag{1}$$

where v is the sedimentation velocity, r is the radius of the nanoparticle, g is the acceleration of gravity, ρ_1 is the concentration of the nanomaterial, ρ_2 represents the density of the fluid medium, and η is the viscosity of the fluid medium. The sedimentation rate of nanoparticles is determined by factors such as the size and density of the nanoparticles, as well as the density and viscosity of the fluid medium. The sedimentation rate of nanoparticles is proportional to the square of their radius, and smaller nanoparticles can exhibit better dispersion stability. Alves et al. [153] prepared three kinds of spherical CuO nanoparticles of different sizes (average diameters 2.5 nm, 4.4 nm, and 8.7 nm) by alcohol thermal method. They found that the as-prepared CuO nanoparticles produce no visible precipitates after storage at ambient condition for 30 days; and the CuO nanoparticles with smaller average diameter exhibit better dispersion stability. Huo et al. [154] said that graphene nanosheets with smaller transverse dimensions exhibit better dispersion stability and anti-wear ability. Su et al. [56] mentioned that fibrous carbon nanotubes are more likely to agglomerate than spherical graphite. Chebattina et al. [155] ball-milled multi-walled carbon nanotubes to shorten their size and acquired significantly improved dispersion stability and tribological properties; they suggested that the concentration of nanoparticles in the base lubricant is an important factor affecting their dispersion stability, and higher concentrations refer to stronger intermolecular forces as well as more severe aggregation. Kuang et al. [156] observed similar concentration–aggregation relationship of CeO_2 and CeO_2 modified with oleic acid and stearic acid, and they claimed that the surface-capped CeO_2 and CeO_2 nanoparticles added in the base lubricating oil have better dispersion stability at lower concentrations.

The surface charge of nanoparticles highly depends on the pH, and a rule of thumb for stabilizing the suspension of nanoparticles is that the pH is one full pH unit away from the isoelectric point (IEP): when the IEP is close to pH, the nanoparticles will undergo severe agglomeration in the medium, thereby reducing the dispersion stability of the suspension. The pH value of the base lubricant also affects the dispersion stability of the nanoparticles therein. Kader et al. [157] found that the acidic functional graphene has a zeta potential of –48 mV as well as excellent dispersion stability at pH = 7. Mao et al. [158] proposed that the suspension stability of nanofluids is affected by pH: when the pH is lower than 7, the dispersion stability of the nanoparticles increases with the increase of pH; and when the pH is greater than 7, obvious precipitation will occur. Guedes et al. [159] synthesized superparamagnetic nanoparticles and found that the magnetic nanoparticles are uniformly and stably dispersed in the base oil under externally applied magnetic field, which is favorable for improving the tribological properties of the base oil.

3.2 Dispersion methods

Due to the large specific surface area and surface energy of nanoparticles, untreated nanoparticles are very easy to agglomerate in the lubricating medium, and most nanoparticles, especially nanometals, nanooxides and sulfides, require physical or chemical dispersion methods to achieve stable dispersion in base lubricant. This section summarizes the dispersion techniques used in most studies: physical dispersion, chemical dispersion, and other dispersion methods. Due to their low density and special structure (such as paper lumps), some carbon nanomaterials and their composites have been shown to have self-dispersing capabilities [160]. The treatment of valence nanoparticles by surface modification is important to enhance their dispersion stability. Different types of nanomaterials are combined with the functional groups of the decorative agent in different ways, which leads to changes in the surface chemical structure of the modified nanomaterials and adds to their compatibility with lubricant base oils. The dispersion methods of nanoparticles and the efficiency for them to form stable solutions are summarized in Table 3.

3.2.1 Physical dispersion methods

The agglomerated nanoparticles can be uniformly dispersed in lubricant medium with the assistance of physical dispersion methods including mechanical method, magnetic method, shear homogenization, and ultrasonic method. The mechanical dispersion method is simple and effective and does not cause chemical reactions of the nanoparticles, which makes it suitable for maintaining good dispersion stability of nanoparticles in a short term without changing their molecular properties.

Liñeira et al. [12] added graphene nanosheets with different sizes (GnP7 and GnP40 with sizes of 7 nm and 40 nm) into a biodegradable base oil and observed their dispersion stability after ultrasonic treatment by standing precipitation. They found that GnP7 with a mass fraction of 0.55% and GnP40 with a mass fraction of 0.75% did not precipitate within four weeks. Elagouz et al. [4] added ZnO nanoparticles to commercial motor oil (10W-40) and mixed the nanolubricant for 4 h with a mechanical stirring device; and the as-obtained suspension is uniform and stable while the ZnO nanoparticles show good dispersion stability in up to 336 h. Usually, surfactant is adsorbed or bound to the surface of nanoparticles by van der Waals forces to change their surface properties. Besides, nanoparticles can be coated with surfactant under high temperature condition, thereby significantly reducing the tendency of nanoparticles to agglomerate and maintaining their stable dispersion state in different lubricant media.

Oleic acid, a monounsaturated fatty acid, can act as a surfactant to improve the dispersion stability of nanoparticles. Mariño et al. [17] successfully prepared ZnO nanoparticles coated with oleic acid; after ultrasonic treatment, the OA-capped ZnO nanoparticles can be uniformly and stably dispersed in PAO40 oil

Table 3 Dispersion methods and efficiency of various nanoparticles.

Nanoparticles	Size (nm)	Media	Concentration	Dispersion method	Stability
Physical dispersion method					
Graphene [12]	7 × 3, 40 × 10	BIOE	0.055 wt%, 0.075 wt%	Ultrasonic bath (180 W) 4 h	28 days
ZnO [161]	30	Diesel	0.1 wt%	Ultrasonic bath 45 min	5 days
ZnO [4]	20	10W-40	0.6 wt%	Magnetic stirring 4 h	14 days
C_{60} [30]	N/A	HM32	300 ppm	Electromagnetic stirring and ultrasonic oscillation	20 days
Graphite [162]	50	Palm oil	0.05 wt%	Magnetic stirring, overhead stirring and high shear homogenization	3 days
GN, CNT, GO [163]	N/A	Vegetable oil	N/A	High speed homogenization for 1 h	45 days
CuO [150]	N/A	PAO	0.1 wt%	Toluene is added as a dispersant	30 days
PTFE [164]	30–50	OP	6.0 wt%	Add 1.0 wt% PIBSI dispersant	63 days
TiO_2 [165]	30	Water	3.0 wt%	Add SDBS surfactant and PAAS thickener	5 days
Chemical dispersion method					
Graphene oxide [166]	N/A	Base oil	0.01 wt%	The alkyl straight chains of octadecylamine, tetradecylamine and capylamine form covalent connections with graphene oxide	30 days
Graphene [167]	2000–5000 × 3.1	PAO6	0.5 wt%	Modified by octadecylamine and dicyclohexyl-carbodiimide, long chain alkyl grafted on the surface of graphene	30 days
Al_2O_3 [168]	78	Naphthenic oil	N/A	The silane coupling agent KH-560 is directly on the surface of Al_2O_3 by polar bonds of hydrogen-oxygen groups	20 days
CeO_2 [156]	8	Shell SN/CF 5W-30	10 ppm	The modifier oleic acid and stearic acid are grafted onto CeO_2 by carboxyl–COO–grafting	7 days
ZnO [17]	10	PAO40	0.25 wt%	Use oleic acid as a surface modifier (modifier by carboxyl–COO–grafted ZnO)	29 days
ZnO [104]	2.4–5.2	DIOS	N/A	Dialkyl dithiophosphoric acid (DDP) is used as the modifier, and ZnO nuclei is bound to the DDP modified layer by covalent bonding	10 days
ZnS [169]	4	PAO4	0.5 wt%	Nano-ZnS is modified by dodecanethiol (modifiers form a protective inorganic–organic interface via S–C bonds)	30 days
WS_2 [91]	N/A	PAO6	1.0 wt%	Oleamine is modified on the WS_2 surface by amino-coordination	3 days
Other dispersion methods					
Crumpled graphene [160]	500	PAO4	0.1 wt%	Self-dispersion capacity due to morphological features	20 h
Graphene [61]	500 × 1	Water	7.3, 12.3, 27.4 μg/mL	The transverse size of the graphene sheet is controlled at about 500 nm, which is more conducive to the stable dispersion of graphene	15 days
GO@PTFE [134]	N/A	Water	0.6 wt%	By compounding with graphene oxide nanosheets, the problem of poor dispersion stability and wettability of PTFE is solved	28 days
Cu@SiO_2 [14]	230	Water	6.48 mg/mL	Cu nanoparticles are incorporated into SiO_2 nanospheres to solve the problem that CuDDP cannot be directly soluble in water	30 days
Fe_3O_4@h-BN [146]	2–7	PAO6	0.5 wt%	Fe_3O_4@h-BN nanocomposites are stable in dispersion, while monomaterials have extremely poor dispersion stability	20 h

for one month. Das et al. [171] studied the effects of various surfactants (cetyltrimethyl ammonium bromide (CTAB), sodium dodecyl sulfate (SDS), sodium dodecyl benzene sulfonate, acetic acid) on the dispersion stability and thermophysical properties of TiO_2-based nanofluids. Relevant zeta potential data indicate that CTAB and SDS can effectively improve the dispersion stability of the nanofluids. Mello et al. [150] used toluene, hexane, and ethylene glycol as dispersants to inhibit the agglomeration effect of CuO nanoparticles in PAO base oil. They found that the CuO nanoparticles can be dispersed more evenly in the base oil with toluene as the dispersant. In addition, the lubricant with toluene as the dispersant can significantly reduce the friction coefficient and wear scar.

3.2.2 *Chemical dispersion methods*

Physical dispersion method can be used to achieve uniform dispersion of nanoparticles in lubricant medium, but it is limited by the insufficient long-term dispersion stability of the dispersed nanoparticles. When the external force disappears, the as-dispersed nanoparticles would tend to re-aggregate under the action of van der Waals forces. Therefore, chemical dispersion is required to achieve long-term and stable dispersion of nanoparticles in the medium. Chemical surface modification method for that purpose often uses surface modifier containing surface functional group to react with nanoparticles, thereby changing the surface structure and properties of the nanoparticles. Commonly used surface modifiers include various organic compounds such as OA, oleyamine, silane coupling agent, etc., and they usually have polar groups and non-polar groups or long alkyl chains in the molecular structure. The non-polar groups can chemically react with the surface of nanoparticles while the polar groups at the other end can be adsorbed onto the surface of nanoparticles to acquire hydrophilicity or lipophilicity, thereby adding to the dispersion stability of the nanoparticles in lubricant base oil. The chemical modification method is advantageous in that it can chemically tune the surface structure of the nanoparticles in order to achieve and maintain their stable dispersion in the medium for a long period of time.

Casado et al. [172] proposed a chemical method to cover the surface of ZrO_2 particles with long hydrocarbon chains. The principle is that the –OH group on the surface of ZrO_2 nanoparticles can covalently bond with hydrophobic groups (such as carboxylic acid, phosphonic acid, and alkyl silane) through condensation reaction. As schematically shown in Fig. 10, the nucleophilic substitution of acyl chloride is used for the surface modification, with which octanoyl chloride, decanoyl chloride and palmitoyl chloride are used to connect C–8, C–10 and C–16 saturated flexible chains onto the surface of ZrO_2 nanoparticles. The suspensions of the as-modified of ZrO_2 nanoparticles exhibit good dispersion stability in the base lubricating oil and can well reduce the friction coefficient. Wang et al. [87] obtained functional MoS_2 sheets modified by thiol molecules through

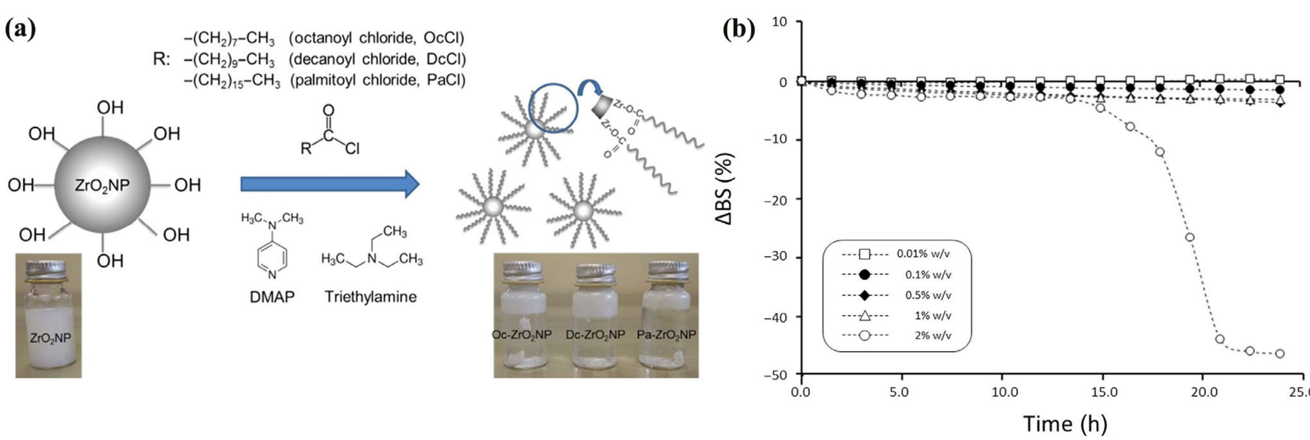

Fig. 10 Schematic modification principle of ZrO_2 NP and sample of original and modified ZrO_2 NPs suspensions in water (a) as well as variation of backscatter (BS) of different concentration of DC-ZrO_2 NPs with time (b). Reproduced with permission from Ref. [172], © IOP Publishing Ltd. 2017.

liquid stripping and thiol chemical collection; and the surface-modified MoS_2 sheets can even be stably dispersed in water for a long time, while they can significantly improve the tribological properties of water.

Kumara et al. [72, 75] modified palladium (Pd) nanoparticle and silver (Ag) nanoparticle with dodecanthiol to obtain PD-C12 and Ag-C12 nanoparticles. After thiolation, the two kinds of nanoparticles exhibit good dispersion stability in lubricating oil and show better lubrication performance, while they have synergistic tribo-effect as compared with single nanoparticle. Liu et al. [173] prepared oleic acid modified RGO@MoS_2 composite material to support oilophilic groups on its surface, achieving better dispersion stability in 10# white oil. Compared with unmodified RGO-MoS_2 composite nanoparticle, the OA-modified counterpart has better lubrication performance. This is due to the insertion of O atoms into MoS_2 skeleton leading to the increase of the layer spacing of the composite material, as evidenced by relevant HRTEM images, XRD data, and infrared spectra (Fig. 11). Particularly, the new absorbance bands of OA-RGO-MoS_2 at 3,452 cm^{-1} and its strengthened absorbance peak at 1,717 cm^{-1} (assigned to the –OH and –COOH functional groups in oleic acid) directly confirm that oleic acid is successfully incorporated onto the surface of RGO@MoS_2 composite.

3.2.3 *Other dispersion methods*

Physical dispersion method is green, safe, and simple in operation. However, nanoparticles treated by physical dispersion method are very easy to re-agglomerate and cannot maintain long-term dispersion stability. The nanoparticles treated by chemical method can be stably dispersed in the base lubricant for a long time, but the surface-modified nanoparticles may undergo chemical reaction, which could lead to changes in their original chemical characteristics to some extent thereby affecting their comprehensive performance as a lubricant additive. It has been found that various nanoparticles can achieve self-dispersion in lubricants via changing the microscopic morphological characteristics or in combination with other nanoparticles to form nanocomposites.

Dou et al. [160] prepared a kind of crumpled graphene ball as a lubricant additive with good

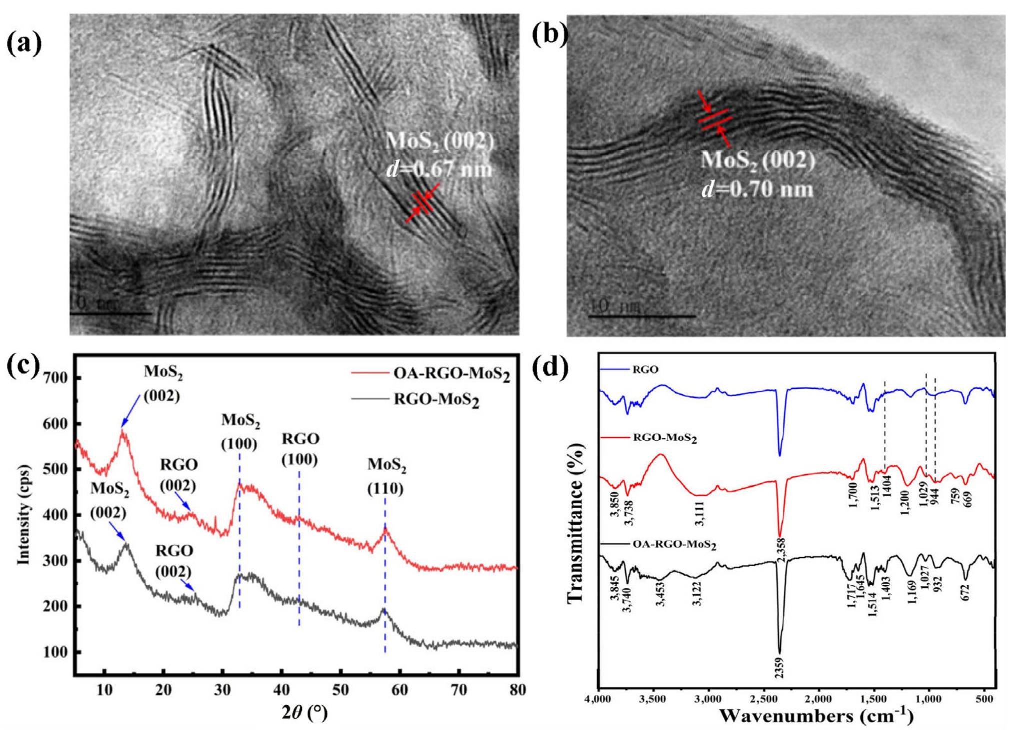

Fig. 11 HRTEM images of (a) RGO-MoS_2 and (b) OA-RGO-MoS_2, (c) XRD patterns of RGO-MoS_2 and OA-RGO-MoS_2, and (d) infrared spectra of RGO, RGO-MoS_2 and OA-RGO-MoS_2. Reproduced with permission from Ref. [173], © Springer 2022.

dispersion stability in PAO4. They said that the crumpled graphene sphere with wrinkled spherical shape can inhibit the agglomeration behavior caused by the force between graphene spheres, which contributes to the good self-dispersion performance. He et al. [174] used a one-step method to rapidly prepare high-purity onion-like carbon nanomaterials (OLC) with an average diameter of 51.3 nm. The as-obtained OLC nanoparticles composed of spherical and multilayer concentric graphene carbon shells exhibit good dispersion stability in mineral oil, although they are not modified by any functional groups. Liang et al. [61] prepared directly stripped graphene as an additive of pure water by jet cavitation and conducted tribological tests. They found that the as-prepared graphene monolayer has excellent dispersion stability in water and exhibits friction-reducing and anti-wear properties upon suspending for 15 days (reducing friction coefficient and wear rate by about 23% and 44%).

Some nanoparticles with good lubricating properties may possess poor dispersibility in lubricating media. This bottleneck could be broken by integrating various nanoparticles to afford nanocomposites. The as-prepared nanocomposites often have good dispersion stability in lubricant medium as well as better tribological properties than those single nanomaterial, which is favorable for their application as lubricant additives. Yang et al. [134] used GO nanosheets to electrostatically coat PTFE and obtained GO@PTFE composite, a new water-based lubricant additive (Fig. 12(a)). The GO@PTFE composite can be stably dispersed in water for 28 days (Fig. 12(d)), getting rid of the poor stability and wettability of PTFE dispersion. As a lubricant additive, it has better tribological properties than GO and PTFE alone, due to the synergistic effect of graphene nanosheet and PTFE. Liu et al. [14] used water-in-oil reverse microemulsion method to compound copper nanoparticles with silica microspheres; and the as-obtained Cu@SiO_2 nanoparticles can be stably dispersed in distilled water without agglomeration precipitation after 30 days. Luo et al. [141] prepared TiO_2@BP composite nanoparticles and added them to PAO6 base oil. After standing for 48 h, the suspension did not precipitate, showing good dispersion stability.

The stable dispersion of nanoparticles in lubricants is always a key issue in the field of lubrication, and the currently available dispersion technology and surface modification technology still face challenges in reducing cost and increasing efficiency. It is urgent

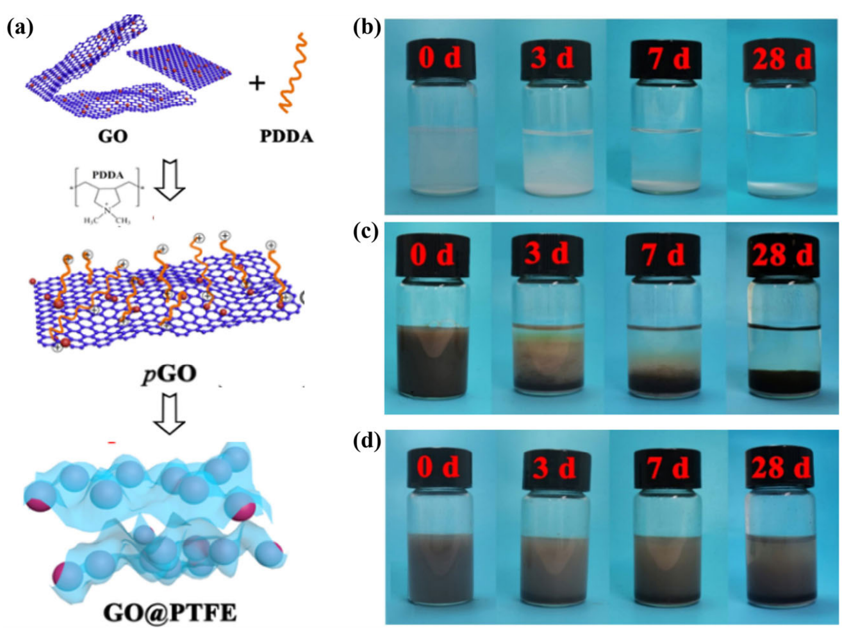

Fig. 12 Schematic diagram showing (a) the preparation principle of GO@PTFE; Optical photos of (b) PTFE, (c) mixed GO/PTFE, and (d) GO@PTFE water dispersions after different standing times (0, 3, 7, and 28 days). Reproduced with permission from Ref. [134], © Elsevier 2022.

to develop low-cost, high-efficiency surface modification technology for nanoparticles as lubricant additives in order to promote their large-scale industrial production; and the research and development of new lubricant additives with self-dispersing ability is the key to breaking through the bottleneck for the development of nano-lubrication.

4 Morphology and particle size of nanoparticles

According to the size of nanoparticles in each dimension of space, they can be divided into zero dimensional, one dimensional, two dimensional and three-dimensional nanoparticles. Now spherical, tubular, onion-shaped, sheet-like, and rod-like nanoparticles are available. Researchers have achieved the purpose of controlling the morphology and size of various nanoparticles by optimizing the preparation procedures. Aside from morphology, the size of nanoparticles is also the key factor that influences their excellent tribological effect. The size and morphology of nanoparticles determine whether they can enter the contact zone of the friction pair and fully contact its surface through adsorption during friction, thereby affecting the tribological properties.

4.1 Zero-dimensional nanoparticles

One-dimensional nanoparticles refer to the particulates with sizes in all dimensions less than 100 nm, while quantum dots are usually between 2–20 nm in diameter. One-dimensional nanoparticles and quantum dots often exhibit spherical shape and extremely small volume, being easy to enter the friction pair gap and function tribologically (Fig. 13).

Alves et al. [153] added spherical CuO nanoparticles into the synthetic oil and studied the influence of the size and concentration of CuO nanoparticles on the tribological properties of the base oil. By comparing the friction test data of three kinds of CuO nanoparticles (2.5 nm, 4.4 nm and 8.7 nm in diameter) dispersed in the base lubricant, they concluded that the friction coefficient tends to decrease with decreasing size of CuO nanoparticles. Lin et al. [175] synthesized two kinds of lanthanum borate nanospheres with a diameter of 50 nm and 105 nm, respectively, and studied their tribological properties in soybean oil with a four-ball machine. Their test results show that the two kinds of lanthanum borate nanospheres can significantly improve the tribological properties of soybean oil, and the lanthanum borate nanospheres with a diameter of 50 nm exhibits better lubrication effect. Many studies have demonstrated that the particle

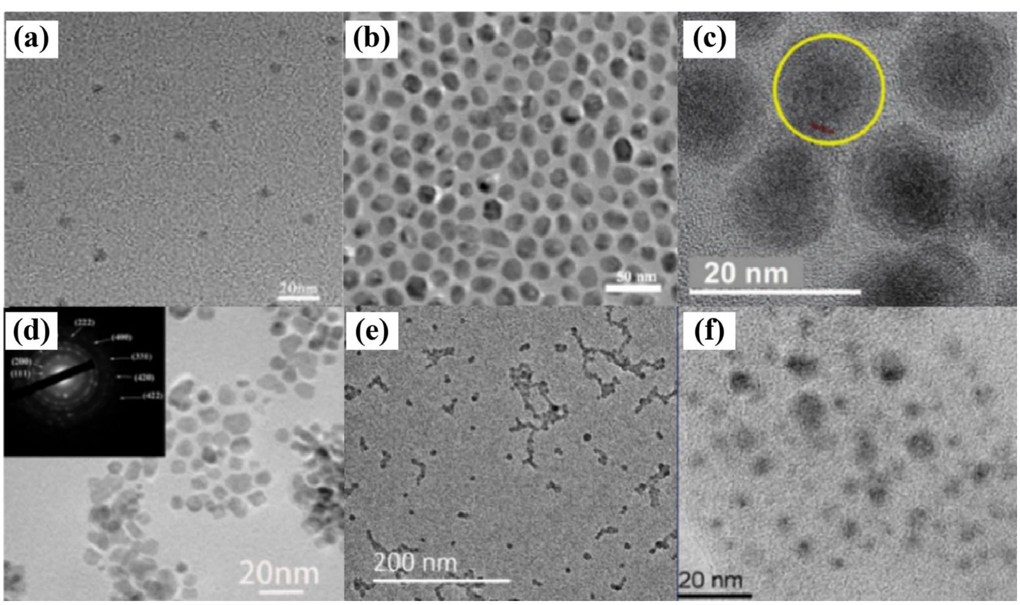

Fig. 13 TEM images of typical zero-dimensional quantum dots of (a) Cu, (b) Ni, (c) Fe, (d) CeO_2, (e) RGO, and (f) WS_2. Reproduced with permission from Ref. [10] for (a), © Tsinghua University Press 2019; Ref. [70] for (b), © ACS 2021; Ref. [176] for (c), © Wiley 2019; Ref. [112] for (d), © Springer 2020; Ref. [177] for (e), © Elsevier 2022; Ref. [76] for (f), © ACS 2020.

size of one-dimensional spherical nanoparticles has an important impact on their tribological properties, and the one-dimensional nanomaterials with smaller size often exhibit better tribological performance. This could be because the spherical nanoparticles with smaller diameters are more likely to enter the friction contact area form a more uniform friction protective film, thereby well protecting the worn surface and more fully exerting tribological properties.

4.2 One-dimensional nanomaterials

One-dimensional nanomaterials are linear, tubular or filamentous in shape; and carbon nanotubes are representative ones. In recent years, multi-walled carbon nanotubes [57, 178, 179] and their composites filled with other nanomaterials have been developed as lubricant additives [180–183]. Some scholars transformed traditional nano-additive materials into one-dimensional shape (e.g., filamentous CuO) by tuning the key parameters in the preparation process to manipulate the morphology of nanoparticles [99]. The lubricating effect of one-dimensional lubricant additives has been proven by research; and they may have great potential in improving the tribological properties of lubricants. Figure 14 presents the TEM images of several kinds of one-dimensional nanomaterials.

The diameter of carbon nanotubes (carbon fiber) nanomaterials can be controlled by adjusting preparation conditions, and their morphology and size affect their tribological properties [57]. Kamel et al. [184] prepared multi-walled carbon nanotubes with a length of 5 mm and diameter of 10 nm as nano-lubricating additives. They said that MWCNTs as the lubricant additive can improve the load-bearing capacity and extreme pressure properties of calcium grease. Ye et al. [185] added modified MWCNTs into water and liquid paraffin wax and studies the effects of their length and diameter on tribological properties. They concluded that the diameter of MWCNTs have significant effects on their tribological properties, and the MWCNTs with proper length and diameter can

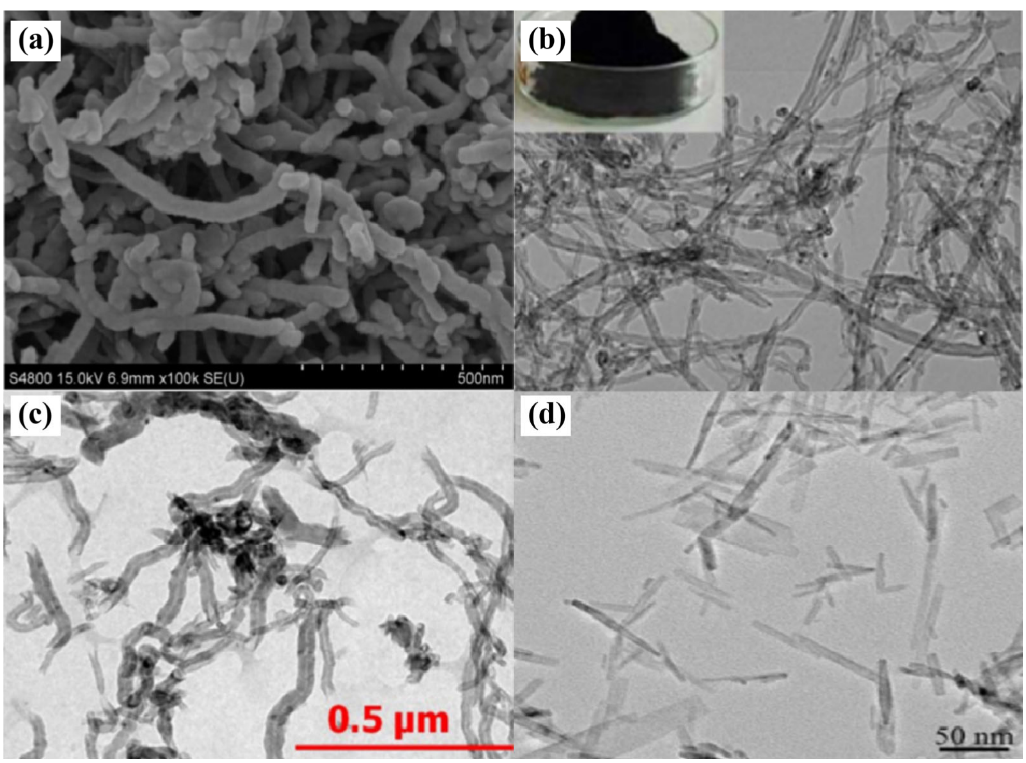

Fig. 14 Microscopic images of various one-dimensional nanomaterials: (a) SEM image of carbon nanotubes, (b) SEM image of multi-walled carbon nanotubes, (c) HR-TEM image of COOH functionalized multi-walled carbon nanotubes, and (d) TEM image of CuO nanoribbon. Reproduced with permission from Ref. [56] for (a), © SAGE Publications Inc. 2018; Ref. [57] for (b), © IOP Publishing Ltd. 2021; Ref. [179] for (c), © Springer 2021; Ref. [99] for (d), © Tsinghua University Press 2021.

easily enter the friction contact zone to reduce friction and wear of the sliding pair (Fig. 15).

The length and diameter of one-dimensional nanomaterials are not independent of each other, which affects their tribological properties. The diameter at the nanometer size is a prerequisite for ensuring that carbon nanotubes can easily enter the friction pair gap; and the smaller the diameter is, the better the tribological properties are. We suspect that the diameter and length of carbon nanotubes might determine their wandering speed and adsorption-filling efficiency at the friction interface. However, the tribomechanism of one-dimensional nanomaterials like carbon nanotubes in relation to their size still awaits further study.

4.3 Two-dimensional nanomaterials

Two-dimensional nanomaterials have been widely concerned, because of their unique molecular structure; and their synthesis, peeling, and tribological applications are constantly developing [79, 186, 187]. Two-dimensional nanomaterials are nano-sized materials that exist in two-dimensional space. They have a distinct sheet-like shape and mostly consist of several layers or multilayer structures, with adjacent layers held together by van der Waals forces. Single layer thickness of such materials is generally under 100 nm, with many only being 1–5 nm thick (some may reach tens of nanometers). Atoms within the same layer of two-dimensional nanomaterials are bonded by covalent bonding, resulting in high modulus and strength for the monolayer structure. Meanwhile, the van der Waals forces between adjacent layers provide lower shear strength, facilitating relative sliding of adjacent layers under shear forces. These properties give two-dimensional nanomaterials excellent tribological properties [77, 79].

Graphene and transition metal dihalides are typical representatives of two-dimensional sheet nanomaterials. The high strength and low shear force between atoms in the same layer make them suitable for the lubrication of double-friction surfaces [78]. As typical two-dimensional material, MoS_2 nanosheet added in lubricating oil and grease has excellent lubrication performance and extreme pressure properties, being suitable for heavy-duty working environment. During

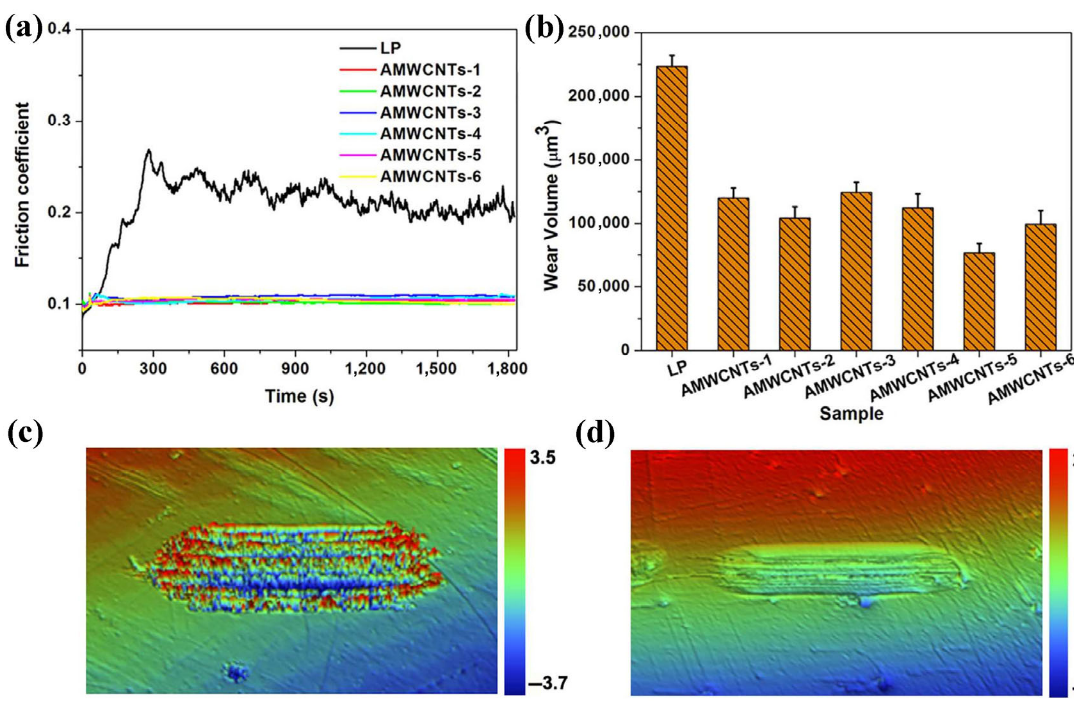

Fig. 15 Curves of (a) friction coefficient and (b) wear volume under the lubrication of liquid paraffin containing MWNCTs with different sizes, as well as optical microscopic images of worn surfaces lubricated by (c) liquid paraffin alone and (d) liquid paraffin containing MWCNT-5 with a diameter of 10–20 nm). Reproduced with permission from Ref. [185], © Elsevier 2019.

lubrication, the two-dimensional nanomaterials slide on the two contact surfaces to form tribofilm with good load-bearing capacity via adsorption and tribochemical reactions, thereby well preventing the direct contact between the two friction surfaces and filling up the grooves thereon [161, 188–190].

The size of two-dimensional materials affects their tribological properties. Liñeira et al. [12] studied graphene nanosheets with maximum lengths of 7 nm and 40 nm and thicknesses of 3 nm and 10 nm (denoted as GnP7 and GnP40; Figs. 16(a) and 16(b)); and they found that the two kinds of graphene nanosheets as the lubricant additives in biodegradable polymer ester can well improve the load-carrying capacity and anti-wear ability of the base oil while the one with a smaller lateral size exhibits better anti-wear ability. Kumar et al. [191] also confirmed the conclusion that the smaller the graphite size is, the better the tribological effect will be. Dong et al. [192] compared the lubrication effects of black phosphorus (BP) nanosheets of different sizes as water-based lubricant additives for titanium alloy-based friction pairs, and they found that the nanoscale (6–10 nm) black phosphorus quantum dots (BPQDS) exhibit better friction-reducing and anti-wear abilities than BP sheets with sizes of 2–4 μm and 300–500 nm (Figs. 16(c) and 16(d)).

A large number of studies demonstrates that the tribological properties of nanoparticles as lubricant additives highly depend on their morphology and size. Under the premise of stable dispersion of nanoparticles in lubricant base oil and/or grease, the shape and size of nanomaterials inevitably influence their tribological properties; and their size control in relation to the size optimization is an important issue of future researches. Besides, it is essential to adjust key preparation parameters or establish novel methods in order to tune the microstructure of nanoparticles and improve their adaptability to complicated friction environment, thereby promoting their application in engineering [71, 159, 193].

4.4 Three-dimensional nanomaterials

Structurally designing and assembling low-dimensional

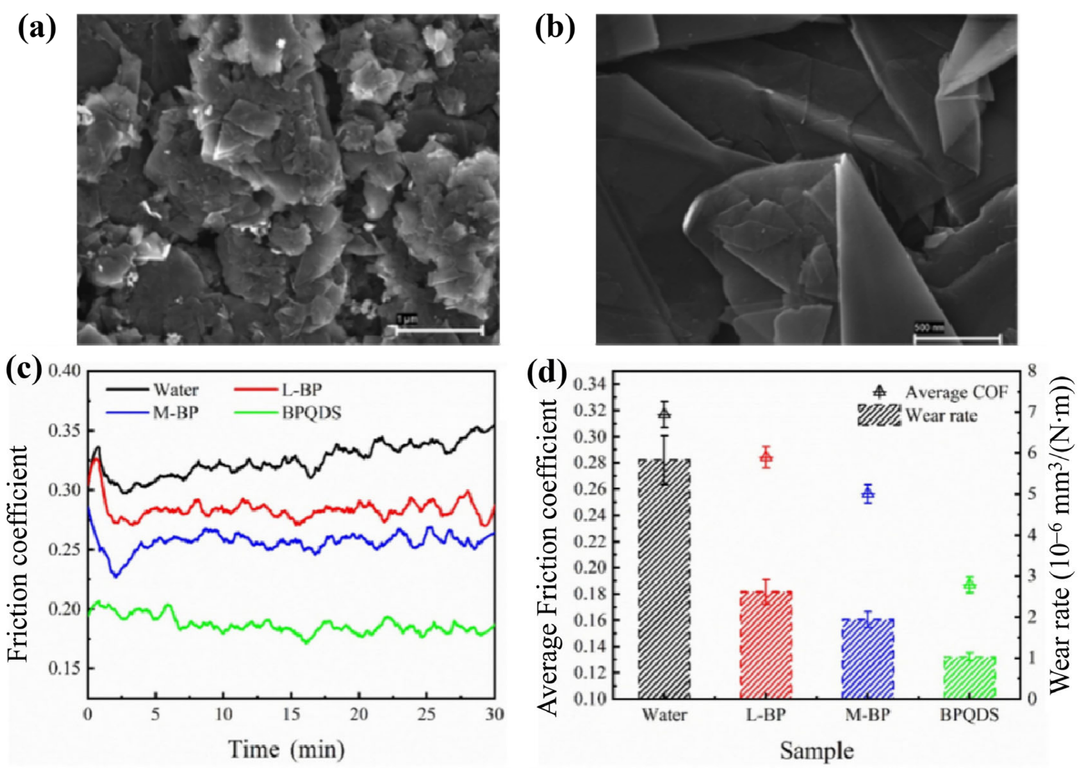

Fig. 16 SEM images of (a) GnP7 and (b) GnP40; (c) Friction coefficient versus sliding time. (d) Average Friction coefficient and wear rate for the ultrapure water and different-sized BP-NS lubricant. Reproduced with permission from Ref. [12] for (a, b) © Elsevier 2022; Ref [192] for (c, d) © MDPI 2022.

nanomaterials into more complex and hierarchical three-dimensional structures can enhance their functional properties. The three-dimensional structural modification strategy has been applied to the fields of energy, sensing, and electronics [194]; and three-dimensional nanomaterials have promising prospects in the field of lubrication and faces some challenges as well. Usually, three-dimensional micro-nano materials as lubricant additives are mainly designed from the structure or shape in low dimensions, exhibiting their unique three-dimensional morphology in space (e.g., paper lumps, multilayer graphene, flower-like, and spindle-like nanomaterials). Three-dimensional nanomaterials as lubricant additives are designed and prepared on the basis of classical lubricating nanomaterials; and as an extension of the original lubricant additives, they could contribute to improve the tribological properties of conventional lubricant additives.

Dou et al. [160] said that a ruffle graphene ball as a high performance lubricant additive exhibits significantly increased dispersion stability in poly-alpha olefin base oil and can effectively improve the tribological properties of the base oil. Xie et al. [62] used a kind of multilayer graphene as a lubricating additive for natural wax, and found that it could significantly improve the load-bearing capacity and high-temperature performance of natural paraffin. Zhao et al. [99] prepared a kind of fusiform CuO nanoparticles as a water-based lubrication additive and found that the friction film generated during friction contact can prevent the direct contact of the friction pairs, thereby improving the tribological properties of distilled water (adding fusiform CuO to distilled water can reduce the friction coefficient and wear scar diameter by 65% and 62%).

Pleated graphene as a kind of typical three-dimensional nanomaterial can be stably dispersed in base lubricant, which is favorable for their use in engineering. In the meantime, some nanomaterials can be endowed with self-dispersibility in lubricant media by tuning the microstructure and shape, which could be of significance for the development of novel high-performance nanomaterials as lubricant additives.

5 Nano-additives suitable for different usage conditions and industrial lubricants

5.1 Nano-additives suitable for different service conditions

Commonly seen lubrication states include boundary lubrication, mixed lubrication, and elastohydrodynamic lubrication. Nano-lubricant additives are effective in improving lubrication performance across various states, especially in the boundary lubrication and mixed lubrication states.

In the presence of liquid lubricants, liquid friction occurs before the onset of dry friction process, and such a liquid friction process refers to boundary lubrication state. Under the boundary lubrication state, the boundary lubrication film with a thickness of 1–50 nm accounts for the desired friction and wear behavior of the frictional pair. The selection of nano-lubricating materials involves the consideration of frictional pair material, mechanical structure, usage, friction environment (including factors such as temperature, load, and frequency), and other conditions. The formation of dense and firm protective films on the friction contact surfaces is a key factor for the selection of nanomaterials, with which nano-soft metals and sulfides are often potential candidates [72, 75, 90, 195]. In case of high temperature requirement, nanomaterials such as nano sulfides and metal oxides with excellent high-temperature resistance should be selected [91, 93, 196, 197]. Mello et al. [198] compared the tribological performance of conventional extreme pressure (EP) additives and oxide nanoparticles (CuO and ZnO) under boundary lubrication conditions. They found that oxide nanoparticles have superior friction reduction ability and environmental acceptance. Jazaa et al. [199] investigated the effects of CuO, WS_2, WC nanoparticles on the tribological properties of PAO base oil and found that under different dispersion conditions (oleic acid or polyisobutylsuccinimide as a surfactant), the nanoparticles can improve the lubricating performance to varying degrees (10%–20%) during boundary lubrication friction.

The mixed lubrication state is characterized by both hydrodynamic and boundary lubrication. Although hydrodynamic lubrication is dominant in most cases, local boundary lubrication can occur at the peak position of the contact surfaces, due to the inability of the friction pair surface to be entirely smoothed [200]. Wen and Huang [201] proposed that elastohydrodynamic lubrication state starts to transfer to mixed lubrication state when the lubrication film thickness reaches 25 nm. Overall, it is recommended to use nano-additives with strong film-forming ability in mixed lubrication state to handle partial boundary lubrication. Kerni et al. [202] examined the friction and wear characteristics of olive oil containing nanoparticles under boundary lubrication and mixed lubrication conditions. They found that the nanoparticles of Cu and h-BN with a mass fraction of 0.5% in the base oil lead to significant enhancement of the lubricating performance in association with their accumulation in the valley of the rubbed surfaces to form a thin film at the interface.

Elastohydrodynamic lubrication, also known as full film lubrication, involves the use of relative speed between frictional surfaces to create a fluid lubrication layer that fully separates them, with typical film thicknesses ranging in 0.1–1.0 mm. Elastohydrodynamic lubrication is considered as the best lubrication state for providing effective protection to frictional surfaces and supporting the rolling effect between them; and it requires greater pressure to maintain the lubrication films produced thereunder [203–205]. Besides, the flowing characteristics of nanofluids are critical to maintaining elastohydrodynamic lubrication state. Since friction contact surfaces are actually rough during movement, nanoparticles may be trapped in narrow areas within the friction pair gap, due to excessive particle size or agglomeration, which can seriously affect lubrication performance. Therefore, it is recommended to use nanomaterials exhibiting superior dispersion stability, small particle size, strong load-bearing capacity, and good film-formation ability in order to maintain elastohydrodynamic lubrication regime [74, 75, 206]. Due to the complexity of friction process, further researches are necessary for better understanding the impact of nanoparticles on the tribological properties of lubricating oils.

Currently, lubrication systems increasingly need to meet higher demands, such as bearing high speeds and loads in large mechanical equipment, long-term high-temperature operation above 100 °C for engine lubricating oil, and stable performance in vacuum environments for aerospace and other industries. As a result, conventional nano-lubricant additives have become increasingly inadequate for meeting application requirements. In response, scholars have conducted research and developed numerous high-performance nano-lubricant additives capable of operating effectively in harsh service environments. Table 4 summarizes the nano-additives that are used in such environments (e.g., extremely high/low temperatures, high contact pressures, vacuum); and it also presents corresponding tribological test conditions and experimental results.

5.2 Application of nanoparticles in industrial oils

Industrial lubricants may meet higher performance requirements, due to their frequent use in larger machinery and harsher service environments. Nano-additives can supplement and enhance base oil performance, introducing novel features that can significantly improve the overall performance of industrial lubricants under specific working conditions. This section examines several nano-additives for industrial oils.

Gear lubrication conditions are more intricate than those of bearings, as the contact pressure from gear meshing is extremely high and the load change amplitude is both large and discontinuous. Moreover, with the gear machine's wide output power range and high transmission efficiency, it frequently operates at excessive temperatures. In conjunction with environmental factors affecting gear lubricant service, nano-additives used in gear oil must provide good resistance to wear, extreme pressure, load impact, and high temperature.

Kiotia et al. [215] discovered that Al_2O_3 gear oil containing 0.3 wt% of nano-additives enhances anti-wear performance by 127% and reduces friction by 25%, whereas SiO_2 gear oil containing 0.3 wt% (SAE EP-90) increases anti-wear performance by 88% and reduces friction by 22%. Han et al. [216] observed that diamond and ZnO nanoparticles, when combined

Table 4 Researches on the application of nanoparticles under harsh working conditions.

Nanoparticles	Base oil	Concentration (wt%)	Performance testing	Lubrication effects
Al_2O_3 [196]	Silicone oil	0.5–1.0	UMT, RT–250 °C, 5 N	At 250 °C, the wear rate is reduced by 91%.
WS_2 [91]	PAO6	1.0, 1.8	UMT, 25–300 °C, 300 N	75–200 °C, the anti-wear and friction-reducing performance is improved. RT-300 °C, anti-wear performance is better than that of ZDDP.
Multilayer $Ti_3C_2T_x$ MXene nanosheets [207]	PAO8	3.0	MTM2, 25–100 °C, 5–28 N	100 °C and 28 N, 30% reduction in friction coefficient.
MoDDP + PB (potassium borate) [208]	Polyurea grease	3.0	SRV-4, 130 °C, 50 N	The average friction coefficient is reduced by 25% and the wear rate is reduced by 31%.
Mn_3O_4@G [143]	Lithium-based grease	0.03	SRV-4, 150 N, 100 °C	The friction coefficient and the depth of grinding spots are decreased by 35% and 75%, respectively. Stable lubrication at 120 °C.
g-C@Fe_3O_4 [209]	Rapeseed oil	0.3	Four-ball, 155 °C, 804 N	The friction coefficient is significantly reduced (0.029). At 980 N, the four-ball experiment runs smoothly.
MoS_2@Gr [210]	Polyalkyl alcohols	0.5	SRV-4, 50–100 °C, 25–100 N	Tribological performance is improved at different temperatures and loads.
$CaCO_3$ [197]	Pure jojoba oil	0.3–0.4	Four-ball, 75 °C, 392–618 N	There are significant improvements in final failure-free load (618 N), initial trouble-free load (784 N), weld load (1,569 N) and load-wear index (253 N).
Calcium borate/cellulose acetate-laurate [211]	PAO	0.6	Four-ball, 75 °C, 490 N	The average spot diameter and average friction coefficient are decreased by 26% and 49%, respectively, and maximum bite-free load (P_B) is increased by 80%.
BHD [212]	PFPE	1.0–2.0	SRV-IV, −20–100 °C, 5 N, vacuum (3.1×10^{-4} Pa)	It exhibits excellent tribological properties and good radiation resistance.
MoS_2 [213]	PFPE	1.0	Four-ball, RT, 200 N, vacuum (5.0×10^{-3} Pa)	The average friction coefficient and the diameter of the grinding spot are decreased by 45% and 40%, respectively.
Mo, W [214]	Polyalkyl-cycl opentane oil	0.5	Four-ball, RT, 294 N, vacuum (< 5.0×10^{-4} Pa)	Improved anti-wear, friction-reducing and extreme pressure properties.

with phosphorus ionic liquids as surfactants, lead to significantly enhanced anti-wear ability of gear oils. Rajendhran et al. [217] said that Ni@MoS_2 composite nanomaterials as the lubricant additive for gear oil (SAE75W) exhibit excellent anti-wear ability and load-bearing capacity under extreme pressure and uniform loading conditions and can enhance the thermophysical properties of the gear oil as well. Wang et al. [218] reported that the addition of 0.6 wt% nano-Cu to heavy-duty gear oil (SJ15W/40) significantly reduces the friction coefficient by 71% and wear by 25% under an applied load of 500 N. Fan et al. [219] found that rGO@MoS_2 heterostructured nanosheets with exceptional dispersion stability in industrial gear oils can significantly decrease the friction coefficient (up to 16.7%) and wear rate (up to 80%) under sliding contact condition.

The primary objective of bearing lubrication is to minimize the friction and wear of the bearing and lengthen its lifespan. Both oils and greases can fulfill the lubrication requirements of ball bearings theoretically. Grease has excellent sealing ability and can effectively reduce vibration and noise. Lubricating oil should be chosen for its good heat dissipation effect through oil circulation under high temperature and high speed conditions. Depending on the bearing's specific working conditions, such as low-speed heavy loads, variable loads impacted by shock, high-speed light loads, and large environmental temperature fluctuations, appropriate lubricating greases and nano-additives should be carefully selected.

Zhang et al. [220] found that graphene, when used as a lubricant additive, can reduce the friction coefficient in high-speed ceramic bearings and enhance the thermal conductivity of the lubricating oil and aid in heat dissipation from the bearing. Wu et al. [221] reported an increase of 27% and 43% in the anti-wear and friction-reducing abilities of an aviation lubricating oil containing 0.075 wt% graphene for the Si_3N_4/GCr15 steel sliding pair. Regarding bearing lubrication experiments, Nassef et al. [170] added reduced graphene oxide (rGO) as an additive to lithium-based grease and demonstrated significant improvements in load-carrying capacity (up to 100%), wear resistance (50%), noise reduction (50%), and cushioning capacity (20%) of rolling bearings. These findings can provide critical insights for the practical application of graphene in the hybrid bearing industry. Suryawanshi et al. [15] investigated the tribological properties of TiO_2 as a lubricant additive for commercial Mobil-class journal bearing lubricants. The four-ball friction test revealed a reduction of 2%–26% and 2%–7% in the friction coefficient and grinding spot diameter after the addition of TiO_2. Utilizing four-ball friction and bearing vibration tests, Wu et al. [222] examined the tribological and vibration-damping properties of calcium sulfonate composite grease containing 0.75 wt% h-BN and 0.25 wt% Al_2O_3; and they found that the average friction coefficient for calcium sulfonate composite grease is decreased by 11.3%, the wear mark diameter is decreased by 6.1%, and high-frequency, medium-frequency, and low-frequency vibrations are reduced by 19%, 11.7%, and 16.4%, respectively, after the addition of the nano-lubricant additives.

Steam turbine oil encompasses steam, gas and hydraulic turbine oils which are typically used in large industrial equipment. It serves as a lubricant for rolling bearings, reduction gears, governors, and hydraulic control systems in coupling groups. The primary functions of turbine oil include cooling, speed regulation, and lubrication.

Shen et al. [223] investigated the impact of carbon nanoparticles with different dimensions (zero-dimensional fullerene C_{60}, one-dimensional carbon nanotube CNTs, two-dimensional reduced graphene oxide (RGO) on the tribological properties of L-TSA-45B turbine oil. Experimental outcomes revealed that RGO exhibits better dispersion stability in turbine oil; and the introduction of 0.015 wt% graphene into the turbine oil leads to an increase in the P_B value by 52% as well as decreases in the friction coefficient, grinding spot diameter, and surface area of the grinding spot by 25%, 49%, and 74%, respectively. Heris et al. [224] reported that nanoparticles (CuO, TiO_2, and Al_2O_3) can increase the heat transfer coefficient of turbine oil. Saidi et al. [225] synthesized octadecyl modified flower-like MoS_2 nanoparticles and found that the target product as lubricant additive in wind turbine oil exhibits excellent long-term dispersion stability and can effectively reduce the friction coefficient (by more than 30%) and wear depth (up to 97%).

6 Lubrication mechanism of nano-lubricating additives

The lubrication mechanism of nanomaterials as lubricant additives is one of the key issues to study their friction-reducing and anti-wear properties. In recent years, many new nano-lubricating additives emerged and many researchers are continuing to explore and reveal their lubrication mechanism, hoping to acquire more insights into their tribo-function and the development of novel high-performance nano-lubricant additives. At present, the lubrication mechanism of nanoparticles refers to the formation of tribofilm, the ball-bearing effect, the self-repairing and polishing effect, and synergistic effect. Table 5 summarizes the lubrication mechanisms of various nanoparticles as lubricant additives.

6.1 Formation of tribofilm

Nanoparticles usually exhibit high specific surface area, surface energy and surface activity, which is favorable for them to form protective tribofilm on the friction contact surface through adsorption-deposition and/or tribochemical reaction. The formation of the protective tribofilm is closely related to the molecular polarity of the nano-additives. The molecular polarity of nanoparticles and the polarity of the base lubricant

Table 5 Lubrication mechanisms of nano-lubricant additives.

Lubrication mechanism	Nanomaterials
Formation of tribofilm	Cu [13, 67, 68], Ag [72], Pd [75], Ni [70], CeO_2 [105, 106], TiO_2 [15], Cu@SiO_2 [14], BN@Cu [138].
Roll-bearing effect	Al [16], ZnO [4, 17], Al_2O_3 [226].
Self-repairing effect	Ni [70], Cu [19, 227], CuO [100], SiO_2 [21, 22, 109], Al_2O_3 [226].
Polishing effect	SiO_2 [21, 22], Nanodiamond [228, 229], MWCNT [178].
Synergistic effect	Fe [71], Ag–Pd [74], CuO [102], $CuZnFe_2O_4$ [230], ZrO_2 [231], graphene@Cu[149], Ag–WS_2–MoS_2 [232], TiO_2@BP [141], Fe_3O_4@MoS_2 [233], Mn_3O_4@rGO [234].

jointly affect the formation of the lubricating film. When the nano-additives have polarity similar to that of the base lubricant, they could be uniformly distributed on the friction surface, thereby favoring the formation of a uniform tribofilm [235]. However, the spillover electrons generated on the contact surface of the friction pair would form an enhanced interfacial electric field thereon, thereby promoting the surface adsorption of polar molecules, especially magnetic nanoparticles. Therefore, polar compounds as lubricant additives with good adsorption capacity on metal surfaces often can form an oil film thereon to prevent metal-to-metal contact, and some nanoparticles can exacerbate their adsorption on the contact surface [67].

Either nanoparticles or nano quantum dots, their small size allows them to be carried into the gap between frictional pairs alongside the base lubricant. This, aided by the surface effect, enables adsorption and deposition of the nano-additives onto the contact surface, creating a physical adsorption film that avoids direct contact of the frictional pair. In the meantime, some nanomaterials as the lubricant additives may interact chemically with the base lubricant and frictional contact surfaces, thereby yielding tribochemical reaction films that exhibit superior performance. The chemical properties of nanomaterials, lubricating media, frictional pair materials, and experimental conditions are critical to the formation of the tribochemical reaction film. Usually, relatively thicker tribofilm can provide better protection for the friction contact area, and the film thickness is dependent on the film-formation rate and wear resistance. Most nanoparticles can form tribofilm approximately 20–200 nm thick, and most nano quantum dots as lubricant additives are capable of generating ultra-thick lubricating-protective films.

Kumara et al. [75] investigated the tribological properties of dodecanethiol-modified nanopalladium (nuclear size: 2–4 nm) as a lubricant additive and found that the lubricant additive forms a tribofilm (2–3 μm) on the surface of the friction pair. Lei et al. [206] discovered that ZDDP combined with oleamine-modified cerium dioxide (OM-CeO_2) as a lubricant additive (average diameter 4–6 nm) quickly forms a tribofilm of 2 μm thick, due to the ultra-high adsorption rate of the composite additive and the generation of cerium phosphate via tribochemical reactions. The as-formed ultra-thick tribofilm is dense and stable and exhibits superb load-carrying and anti-wear capabilities, as evidenced by the HRTEM images of the cross-sections of the tribofilm extracted from the worn surface of the steel ball lubricated by PAO6 + CeO_2 + ZDDP (Fig. 17).

Copper nanoparticle as a lubricant additive can well improve the friction-reducing and anti-wear abilities of lubricants, thanks to its face-centered cubic structure as well as low shear force, excellent ductility, and low melting point. Copper nanoparticle as lubricant additive can be well deposited on the rubbed surface of friction pair to form a protective lubricating film, thereby exerting good friction-reducing and anti-wear properties [236]. Singh et al. [13] added Cu nanoparticles into plant-based modified desert date oil and found that the bio-based lubricant containing copper nanoparticles exhibits increased viscosity and can form a dense protective film on the friction contact surfaces.

Wu et al. [112] synthesized oleylamine-modified CeO_2 nanoparticles (OM-CeO_2) by one-pot pyrolysis and investigated their effect on the tribological properties of PAO. They said that the PAO base oil containing 0.2% OM-CeO_2 exhibits desired long-term anti-wear ability. This because the low concentration

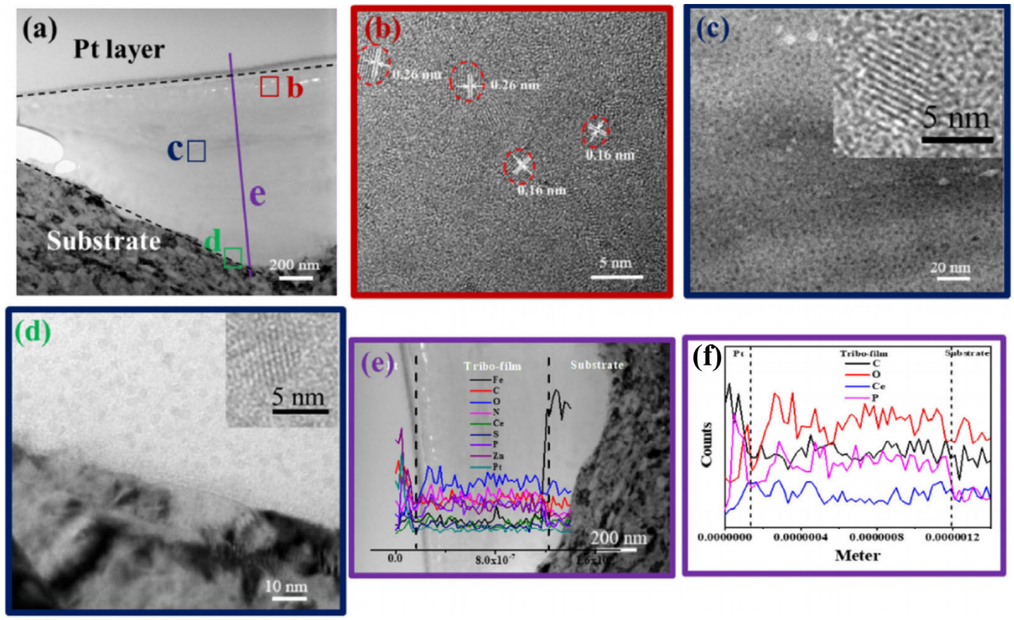

Fig. 17 TEM and EDS images of wear marks of steel ball lubricated by PAO6 + CeO_2 + ZDDP: (a) cross-sectional TEM image of tribofilm, (b) HRTEM image of tribofilm in zone b in (a), (c) HRTEM image of tribofilm in zone c in (a), (d) HRTEM image of tribofilm in zone d in (a), (e) line scan of wear surface elements, and (f) line scan of key elements in tribofilm (four-ball machine; 1,200 r/min, 392 N, 75 °C, 60 min). Reproduced with permission from Ref. [206], © Springer 2023.

of OM-CeO_2 can catalyze the oxidation of Fe to produce Fe_2O_3 or FeO on the worn surface while CeO_2 is transformed into Ce_2O_3, which gives rise to a compact tribofilm thereby reducing the friction and wear (Figs. 18(a) and 18(b)). High concentration of OM-CeO_2 is not preferred, because it generates a loose lubricating protective film on the worn surface (Fig. 18(c)). Zhao et al. [99] prepared water-dispersible CuO nanoribbons and nanorods as well as spindle-shaped nanostructures by controlling the types of surface modifiers, the reaction time, and reaction temperature. They found that the as-prepared nano-CuO with different structures could significantly improve the tribological properties of distilled water, due to the formation of protective tribofilm especially in the presence of the CuO nanoribbon and nanorods.

6.2 Roll-bearing effect

Spherical nanoparticles dispersed in lubricant can roll on the friction surface within a certain load range, thereby converting sliding friction into rolling friction at the micro-interface. For that purpose, the spherical nanoparticles need to be rigid enough and can easily enter the contact area of the frictional pair. This requires that the nanoparticles exhibit a large enough particle size and are well accessible to the contact area of the frictional pair under applied normal load, thereby converting sliding friction into rolling friction and well exerting tribological effect. In this sense, nanoparticles with too small sizes are liable to be embedded in the grooves and pits on rubbed metal surfaces, which is unfavorable for them to exert friction-reducing and anti-wear effect.

Spherical nano-diamond particles have a solid surface, which is favorable for them to exert roll-bearing effect. Kim et al. [237] said that nano-diamond added in engine oil can significantly reduce friction coefficient, which is because the spherically shape nano-diamond particles can play the roll-bearing (ball-bearing) effect between two sliding surfaces in contact. Gu et al. [238] prepared monodisperse carbon microspheres by hydrothermal method and found that the as-prepared carbon microspheres dispersed in paraffin wax can significantly reduce the friction and wear of aluminum alloy–steel contact. This is mainly because the carbon microspheres can enter the friction contact zone during sliding friction and plays the role of ball bearing. Radhika et al. [239] synthesized graphene nanosheet-coated functionalized

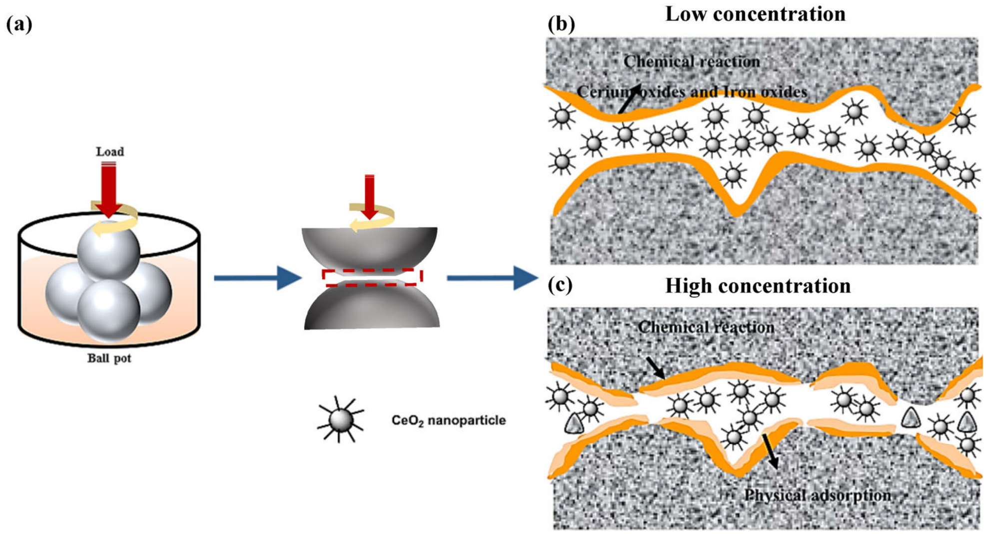

Fig. 18 (a) Schematic diagram of four-ball friction and wear tester as well as tribo-mechanism of OM-CeO_2 at (b) a low concentration and (c) a high concentration. Reproduced with permission from Ref. [112], © Springer 2020.

carbon spheres (GNP@FCS) by ultrasonic technology and found that the GNP@FCS dispersed in engine oil exhibit good tribological properties, which is also because GNP@FCS play the role of ball bearing on the contact surface of the frictional pair (Fig. 19(a)). Rawat et al. [109] investigated the tribological properties of silica nanoparticles dispersed in paraffin wax. They said that SiO_2 nanoparticles added in paraffin wax can significantly improve the tribological properties, since SiO_2 nanoparticles can act as rolling bearing and repair the worn surface (Figs. 19(b) and 19(c)).

6.3 Self-repairing effect

The self-repairing effect involves nanoparticles deposition onto the friction pair surface and their filling into the trenches created by friction-induced damage in conjunction with derivatives generated by tribochemical reaction, and both processes contribute to reducing the surface roughness of the frictional pair. Unlike the mechanism behind friction film formation, nanoparticles added in the base lubricant can enter the gap between the frictional pair to promote the development of a deposited and/or adsorbed film on the rubbed surfaces, thereby effectively preventing the direct contact of the sliding pair.

Shen et al. [19] studied the tribological properties of Cu nanoparticles on the interface of a steel–steel sliding pair. They said that the high contact pressure and temperature can promote the deposition and sintering of Cu nanoparticles on rubbed steel surface to form self-repairing film that can fill up grooves and pits thereon; and the Cu nanoparticles with a strong reducibility can be oxidized into CuO and Cu_2O during the friction process. As a result, the tribofilm mainly composed of CuO and Cu_2O exerts self-repairing effect to significantly reduce the friction and wear of the steel–steel contact (Fig. 20). Wang et al. [20] prepared nanocomposite Ag@GO by achieving uniform growth of scattered silver (Ag) nano-ball in graphene sheets and found that the as-prepared Ag@GO nanocomposite can significantly reduce the friction and wear, due to its repair ability for the worn metal surface via deposition thereon.

6.4 Polishing effect

The polishing effect involves flattening the large convex peaks formed by friction damage on the worn surface and filling of a small number of nanoparticles onto the rubbed surfaces, which leads to decrease in the roughness of the worn surface and ultimately produces a smoother and flatter surface. Usually, nanoparticles responsible for the polishing effect possess high levels of hardness and can act as abrasives that roll and micro-cut worn surfaces, thereby resulting in more smooth and flat surface.

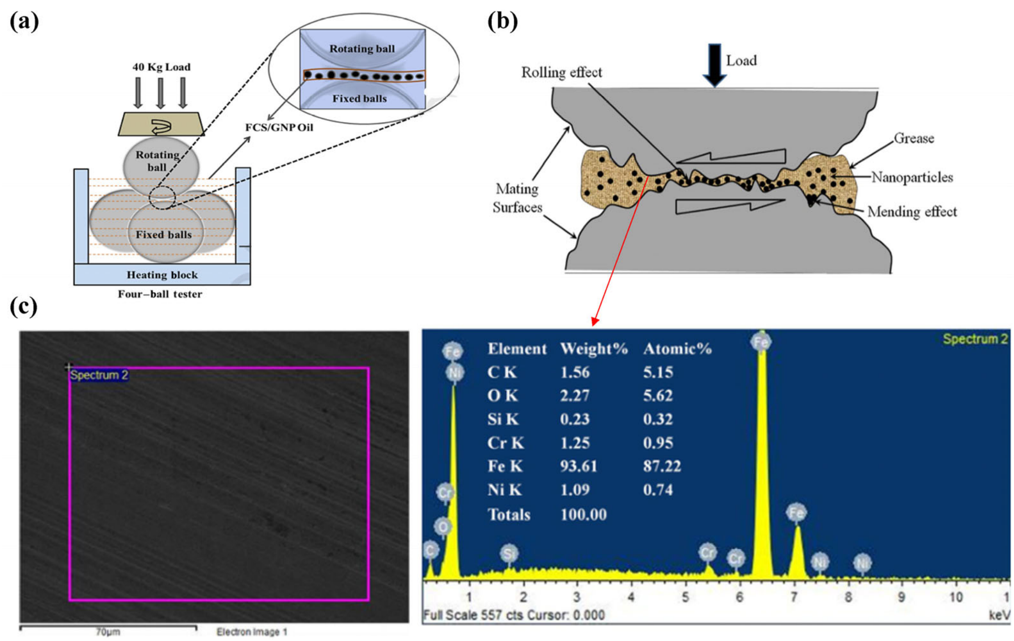

Fig. 19 (a) Schematic diagrams of four-ball test rig and (b) ball bearing effect of SiO_2 nanoparticles as well as (c) repair of worn surface by SiO_2 nanoparticles verified by EDS analysis. Reproduced with permission from Ref. [239] for (a), © Elsevier 2021; Ref. [109] for (b–d), © Springer 2019.

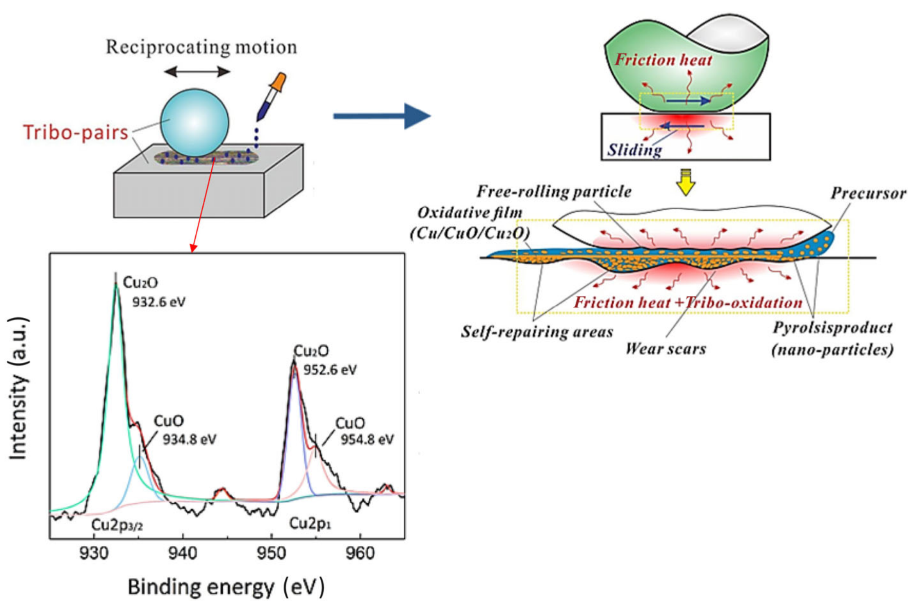

Fig. 20 Schematic diagram of friction-reducing and anti-wear mechanism of nano-Cu as well as its surface-repair effect verified by XPS analysis of worn surface. Reproduced with permission from Ref. [19], © Springer 2020.

He et al. [21] added nano-SiO_2 in lithium-based grease and observed obvious polishing effect: SiO_2 transfers readily to metal surfaces or even to worn regions of the friction pair under mixed or boundary lubrication conditions where nano-SiO_2 shares some compressive stress and forms a self-laminating protective film in a solid-state. Hao et al. [22] synthesized SiO_2 nanoscale ionic liquid (NIL) by using sulfuric acid-terminated organosilane as the corona and polyetheramine as the canopy. The as-synthesized product is an amphiphilic lubricant additive and functions by means of polishing, ball bearing, and repair mechanisms. Raina et al. [229] researched the application of diamond nanoparticles as secondary lubrication additives to steel–aluminum friction pairs in oil and found that the enhanced lubrication effect of nanodiamonds is due to their ball bearing effect and polishing action.

The study of Nunn et al. [228] confirmed that nanodiamond can significantly improve the lubricating ability of PAO oil via polishing effect. Wang et al. [178] produced ionic liquid-modified multi-walled carbon nanotubes (MWCNTs-IL) through the amide method, and discussed their lubrication mechanism as a water-based additive using a four-ball machine. They said that MWCNTs-IL exhibits excellent intercalation stability between friction surfaces by way of electrostatic interaction, while MWCNTs play roles in polishing, repairing and rolling within the frictional pair gap to improve the friction-reducing and anti-wear properties of the base lubricant (Fig. 21).

6.5 Synergistic effect

Nano-lubricant additives with excellent tribological properties are often combined in order to acquire better comprehensive properties as well as friction-reducing and anti-wear abilities via synergistic effect. For example, copper nanoparticles with low shear strength and grain boundary slip effect may exhibit further improved tribological properties as they are combined with other nano-additives such as graphene and carbon nanotubes, due to synergistic tribo-effect [19, 64, 227, 236, 240, 241]

Wu et al. [23] prepared zinc borate and disulfide composite (ZB@MoS_2) by liquid phase ultrasonic treatment. The four-ball friction test proved that ZB@MoS_2 could improve the friction-reducing, anti-wear, and extreme pressure properties of base grease; and zinc borate and molybdenum disulfide exhibit synergistic to form a protective film with good anti-wear ability and load-bearing capacity. Guo et al. [18] combined a zero-dimensional hydrophilic silica nanoparticle with two-dimensional layered nano-graphene oxide (GO) and proposed a new water-based nano-additive, amine-modified graphene oxide (SAG). The schematic diagram for the synthesis of SAG is shown in Fig. 22(a); and Fig. 22(b) shows the synergistic effect between the patching effect of graphene nanosheets and the microscopic ball bearing effect of SiO_2 nanoparticles. Under high contact pressure, SAG can produce very low friction coefficient and reduce wear rate in deionized water.

Wu et al. [195] found that polyisobutylamine succinimide (PIBS) as the modifier can help to achieve stable dispersion of MoS_2 nanosheets in paraffin oil; and graphene oxide and nano-diamond as lubricant additives in water exhibit synergistic tribo-effect and can significantly improve the tribological properties

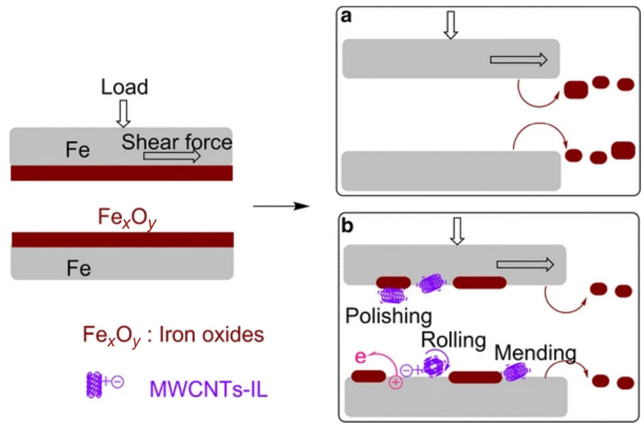

Fig. 21 Schematic diagram of the lubrication mechanism of MWCNTs-IL. Reproduced with permission from Ref. [178], © Springer 2017.

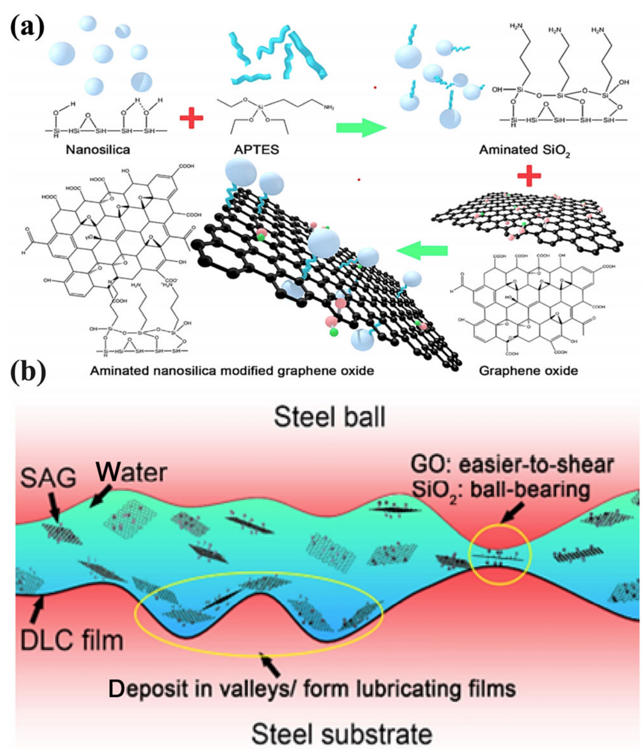

Fig. 22 (a) Schematic diagram of SAG synthesis and its friction-reducing and (b) anti-wear mechanism as a water-based additive. Reproduced with permission from Ref. [18], © ACS 2018.

of water. Gong et al. [142] pointed out that nano-PTFE and nano-$CaCO_3$ added in aluminum lubricating grease show excellent tribological properties, which is due to their synergistic tribo-effect. Zang et al. [138] prepared a hexagonal boron nitride and nano-copper composite (BN@Cu) and studied its tribological properties in paraffin wax after modification with oleic acid. They said that BN@Cu as the additive can reduce the friction coefficient and wear scar diameter by 27% and 25%, respectively, which is due to the formation of a tribofilm via the synergistic action of BN and Cu species.

Many scholars have devoted themselves to the study of the lubrication mechanism of nano-lubricating materials [242, 243]. We recommend to use modern methods such as molecular simulation and/or molecular in-situ tracking to analyze in detail how nanoparticles as lubricant additives play a role in tribological service and to design and prepare novel high-performance nanocomposites as lubricant additives, thereby promoting their application in engineering and benefiting social and economic development.

7 Conclusion and prospect

Nanomaterials play key roles in promoting people's production and life. As lubricant additives, they are advantageous over traditional lubricant additives in that they exhibit better comprehensive properties and can better prolong the service life of mechanical parts, save energy and resources, protect environment, and improve fuel efficiency.

This paper highlights the recent progresses in the researches on nanomaterials as lubricant additives at home and abroad. It summarizes commonly used nanomaterials as lubricant additives in relation to their chemical composition, including carbon nanomaterials, nano-metals, nano-oxides, nano-sulfides, nano-polymers, and nano-composites. Then it focuses on the methods for achieving the stable dispersion of nanomaterials in lubricant mediums, mainly covering physical dispersion method and chemical dispersion method. Furthermore, it briefs the effects of the size and morphology of nanomaterials on their tribological properties; and it also deals with the tribo-mechanisms of nanomaterials as lubricant additives, mainly referring to the formation of protective tribofilm, ball bearing effect, repair and polishing effect, and synergistic effect. In the future study of nano-lubricating additives, we suggest to pay close attention to the following aspects:

1) It needs to conduct further researches to improve long-term dispersion stability of nanomaterials in lubricant base oil/grease. Physical and chemical dispersion methods are currently available for dispersing nanomaterials as the additives in lubricants. However, physical dispersion method cannot guarantee the long-term dispersion stability, and chemical method often causes changes in the molecular structure and properties of nanomaterials. For solving this problem, it might be feasible to tune the microstructure of nanomaterials and fabricate nanocomposites in order to achieve the purpose of self-dispersion of nanomaterials with good long-term dispersion stability.

2) It needs to further study how to tune the shape and microstructure of nanomaterials by adjusting the key parameters of the preparation process and the synthesis method in order to realize the real controllable synthesis of target nanomaterials. This could be essential for the application of nanomaterials as lubricant additives in engineering, because the control of the morphology, microstructure, and size of nanomaterials often refers to their manipulable dispersion stability as lubricating additives and comprehensive properties as well.

3) It needs to adopt and/or establish new methods to analyze the tribo-mechanism of nanomaterials as novel lubricant additives. At present, the judgment of the tribo-mechanism usually relies on the analysis of the worn surface after the completion of the friction and wear test, which can hardly reveal the tribo-effect of nanomaterials in the real state. In this sense, it requires to further explore the friction gap of nanomaterials upon micro-movement in friction process, the tribochemical reactions occurring upon sliding, and the changes in the lubrication state of nanomaterials upon varying sliding parameters. It also requires to adopt advanced research methods such as molecular simulation and atomic directional tracking to investigate the lubrication mechanisms of nanomaterials at depth.

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Declaration of competing interest

The authors have no competing interests to declare that are relevant to the content of this article. The author Zhengquan JIANG is the Youth Editorial Board Member of this journal.

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