DEVELOPMENT OF ANTIWEAR AND EXTREME PRESSURE ADDITIVES FOR LUBRICANTS AND GREASES

There are a myriad of mechanical devices that are integral to societal development. From road vehicles to power plant machinery– these devices have intricate moving parts that depend on lubricants and greases, a special class of lubricants, to inhibit the wear that results from frictional interactions. The application of lubricants/greases is a fundamental element to any mechanical system that allows for its optimal functionality over extended periods. Greases themselves are sophisticated, and the fine-tuning of grease characteristics is pertinent for efficacy in specific mechanical operations. Coupling the proper lubricant/grease with the correct situation can save both money and resources by increasing the reliability of the tribological systems in question (where tribology is the study of friction, wear, and lubrication). One aspect of lubricants that can greatly enhance their characteristics is the additives that modulate their effectiveness in certain situations. Wear preventative additives are among the most important additions that increase the longevity of the machinery.

The most important attribute of a lubricant is its ability to prevent wear. The need for resources to replace and repair mechanical parts due to high friction and wear is more than 6% of the Gross National Product (GNP) [1]. These types of mechanical wear can take different forms in a lubricated system, including adhesive wear, abrasive wear, pitting, and spalling. The emphasis on wear preventative additives especially to greases cannot be overstated. Wear preventative additives become a pillar of how effectively a grease can perform and are an integral part of mechanical devices that are prone to damage from wear. There are two types of wear preventative additives employed depending on the situation: antiwear (AW) additives and extreme pressure (EP) additives.

AW and EP additives are tailored to operate best under certain conditions. Both additives function by depositing a protective barrier on the metal surface via a chemical reaction [2]. AW additives are best suited for lubricants that operate under mild conditions with low loads and high speeds and are made to reduce the rate of continuous and moderate wear [2]. This design to reduce wear from moderate stress over time is done by coating the application surface with the AW additive. EP additives are relegated for use under heavier loads at high temperatures and low speeds [2]. Lubricants that have EP additives are crucial for the prevention of catastrophic failure or seizing of the application [2]. Applications of AW additives are widespread and are often used with hydraulic oils, engine oils, gear oils, automatic transmission fluid, and some greases [3]. EP additives are used in more niche applications, which usually include transmission fluids and non-worm gear oils [3].

The method in which AW/EP additives operate is in the form of a consumable. To perform their function, these additives in the lubricant get used up over time, after which damage from adhesive wear will increase [4]. Chemically, AW/EP additives are polar and attach to frictional metal surfaces. They function by reacting with the metal surfaces when they make physical contact. The heat from metal-to-metal contact activates these additives to form a film that minimizes wear, which serves to also protect the base oil from oxidation and the metals from corrosive acids [4]. This film can withstand compression and prevents the metal surfaces from making contact because the film has lower shear strength than the metal [4,5]. The general criteria for the chemicals that are used in AW/EP additives is that they must use elements that can form iron compounds for proper reaction with the metal surface [5].

The main differences between AW and EP additives are in their physical and chemical characteristics. While they both serve the same purpose to reduce wear, they are each used in different scenarios. Notably, EP additives focus on particular attributes that do not pertain to AW additives. EP additives are built for more severe metal-to-metal interactions, and thus, the film coating is tougher and thicker than those of AW additives [5]. Chemically, EP additives act more aggressively with a higher rate of reaction with the metal as well as faster EP film formation [5]. In some cases, this high reactivity can result in certain EP additives being corrosive to particular metals, warranting careful application of these additives.

Another major distinction of EP additives is in their method of activation. Activation of EP additives can be done through means that are either temperature-dependent or temperature-independent. This differs from AW additives, which almost always form from a temperature increase created due to load [3]. Temperature-dependent EP additives activate and chemically react from the heat produced by the heightened levels of friction and pressure between metal surfaces [6]. Common EP additives that are temperature-dependent are compounds that are based on boron, chlorine, phosphorus, or sulfur [6]. These chemicals form iron compounds when they react with the metal surface, which produces a chemical film that serves as a barrier to reduce friction, wear, and metal scoring in addition to deterring welding [6]. Temperature-independent EP additives, like overbased sulfonate, rely on a different mechanism to function. The iron from the metal surfaces interacts with the colloidal carbonate salt that is dispersed within the sulfonate, creating a film barrier between the metal surfaces [6]. The resulting barrier from this method serves the same purpose as those from the temperature-dependent additive but does not require elevated temperatures to proceed with the reaction. Thus, temperature-independent EP additives provide value in applications where there are low temperature operating conditions.

There are several limitations to consider for the application of EP additives. Some EP additives such as those derived from sulfur and phosphorus can be very chemically reactive, resulting in polishing wear [7]. Polishing wear is the result of two interacting solid bodies that remove materials and produce a polished finish on at least one of the solids. In gear oil applications, polishing wear is detrimental to gear accuracy by wearing away the gear tooth profiles, which is most notable in slow-speed gear interactions (less than 10 feet per minute) [7]. To amend this issue, potassium-borate additives can be used to restrict the chemical reaction with the metal and deposit the EP film [7]. Another limitation is that sulfur-phosphorus EP additives are limited in their functional temperature range, with a high-temperature limit of 95°C [7]. These additives are also somewhat corrosive to yellow metals, such as brass and bronze, and can be fairly incompatible with zinc EP additives as well as zinc AW additives depending on the amount used [7]. Solid-suspension EP additives that are used at temperatures too extreme for oil are still limited by their wear lives and inability to carry the loads necessary to maintain long gear and bearing life [7]. It is important to keep these limitations in mind when applying EP additives to grease or lubricating oil.

To determine the performance of a particular AW/EP additive, there are testing methods that measure their tribological properties. The first approach is tribological testing by the 4-ball methods. The 4-Ball Wear test is a common technique that rotates a steel ball against three stationary lubricated balls positioned as a cradle [8]. This test determines the wear preventative attributes of a lubricant under a specified load, speed, temperature, and time, which are outlined by ASTM D-2266 (greases) or ASTM D-4172 (oils) [8,9,10]. Testing conditions are typically run at 1200 rpm and 40 kg of load at 75°C for 60 minutes [2]. Results of the 4-Ball Wear Test are presented as wear scars that appear on the stationary balls, which are subsequently measured in size and averaged [2]. The coefficient of friction (COF) is also calculated from the measured frictional torque throughout the duration of the test and is averaged as well [8]. A higher-performing lubricant would have a lower COF and smaller wear scars on the stationary balls at the end of the test, thus demonstrating its wear preventative capabilities. The data collected from this test can also distinguish important properties between different lubricating greases, which include load-bearing capabilities, wear protection, and friction reduction [8].

Another variation on the 4-ball testing methods focuses on the

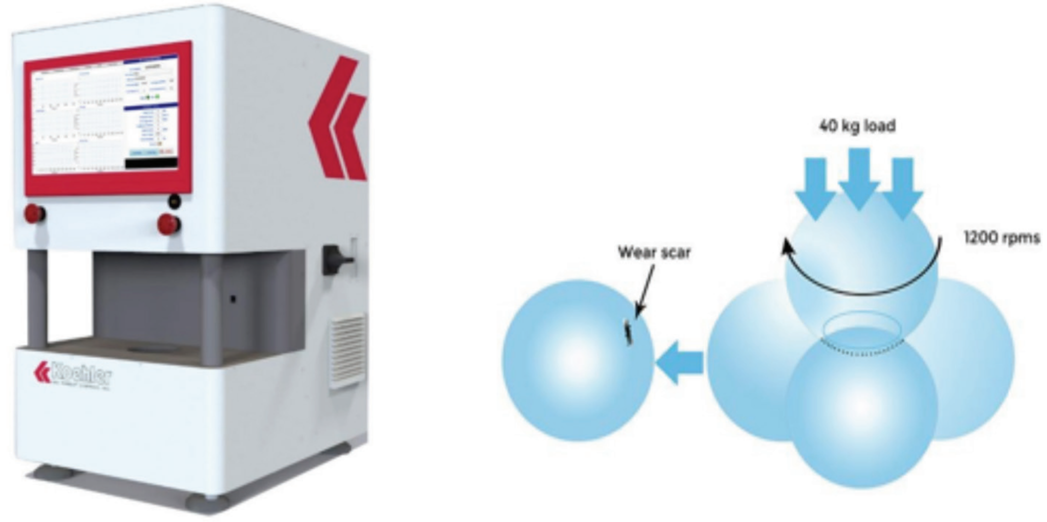

Figure 1: 4-ball wear testing method configuration [8]

EP properties of a lubricant. The 4-Ball EP Test uses the same ball position and technique described previously, but testing parameters are centered around wear produced from different levels of load, which is outlined by ASTM D-2596 [11]. As such, the purpose of this testing is to ascertain the proficiency of a grease's load carrying properties for high load applications like bearings [8]. The main parameter that is tested is the load wear index (LWI), which is a grease's ability to perform under extreme pressure conditions [8]. Throughout the duration of the test, three notable measurements are taken to calculate the LWI. The first measurement is the last non-seizure load (LNSL), which is taken at the highest applied load where lubrication is still present between the 4 balls [8]. Following this measurement is a continuous increase in load, where the point at which the lubricant film fully disappears is called the seizure region [8]. The final measurement is taken when catastrophic welding occurs. The presence of welding can be detected by the testing machine through the following behaviors [8]:

- The friction-measuring component experiences a sharp transverse movement
- A heightened level of motor noise
- Smoking from the ball pot
- A sudden drop of the lever arm
- An average ball scar of over 4 mm is recorded

The values from these three measurements can calculate the LWI, which itself is a numerical value that distinguishes a grease's ability to prevent wear at various applied loads. Both these 4-ball testing methods have become industry standards in testing EP, wear, and frictional performance.

A different methodology in tribological testing uses a device called the SRV instrument where SRV stands for Schwingung (Oscillating), Reibung (Friction), Verschleiž (Wear). The SRV operates either in a rotational or linear oscillatory motion by which it measures the physical interactions between a lubricant and two specimens in loaded contact. Throughout the test, the two specimens oscillate against each other at a specified frequency, stroke length, load, temperature, and time parameters [12]. The variable that the SRV measures is the frictional force, which then gets used to calculate the COF [12]. The specimens themselves can come in various materials, including metals, plastics, and ceramics. If both specimens are metallic, an additional attribute that can be measured is the electrical resistance [12]. Particular testing methods that use the SRV are outlined by ASTM D5706, ASTM D5707, and ASTM G99. ASTM D5706 focuses on the EP capabilities of a lubricant by increasing the test load every two minutes until the specimens weld together, thus indicating lubricant failure [13]. ASTM D5707 reports on the COF and wear scars produced after running the test for a specified duration at moderate loads [14]. Lastly, ASTM G99 concentrates on the friction and wear characteristics of lubricated subjects when the SRV is in rotational mode, in which a stationary upper specimen is in contact with a rotating disc [15]. The versatility in testing methods makes the SRV instrument an attractive option for customizing the test for certain needs.

Perhaps some of the earliest compounds used for EP additives were based on sulfur. Oil-soluble organic sulfur compounds commonly named sulfur carriers, with the general formula of $R–S_x–R$, provide better solubility and control over sulfur reactivity [16]. Sulfur-based EP additives can come in both active and non-active forms, depending on the configuration of the sulfur carriers. Sulfur carriers with predominantly disulfide bridges (x=2) are of the nonactive form, possessing relatively stable C–S bonds that only react at elevated temperatures [16]. Active sulfur carriers are configured with x between 3 and 5 and are much more reactive due to the easy availability of sulfur from the labile bonds of the polysulfide bridges at low temperatures [16]. The general mechanism by which sulfur-based EP additives operate is through physical adsorption, followed by chemisorption, cleavage of the sulfur, and its reaction to the metal surface [16]. Typically, this reaction operates at temperatures over 600°C [17]. Active sulfur EP additives are excellent at welding prevention by continuously sacrificing reaction layers under severe loads in a controlled manner [16]. Inactive sulfur is well suited for use with non-ferrous metals, as the high reactivity of active sulfur additives at lower temperatures can be corrosive to yellow metals and alloys [16,18]. A common EP agent used by the lubricant industry is sulfurized olefins. Sulfurized olefins come in two varieties: long-chain olefins that contain ~10-20% sulfur and short-chain olefins, such as isobutylene (~45% sulfur), dicyclopentadiene, and dipentene olefins (~35% sulfur) [5]. EP potency improves with higher sulfur content, making sulfurized isobutylene a very strong EP additive with exceptional scoring protection properties [5]. The downside of sulfurized olefins is their corrosive nature, warranting their replacement with other EP additives in applications like engine oils [18]. Other sulfur-based EP additives include sulfurized fats or esters, xanthates, thiocarbonates, dithiocarbonates, and molybdenum disulfide.

Figure 2: SRV Instrument [12]

On the AW additive side, phosphorus-based compounds provide the best attributes for wear prevention on steel-based machinery in medium stress conditions with high torque and low-speed operations. These additives perform particularly well in the presence of ridging or rippling due to metal flow under extreme stress and high loads [5]. Additionally, these additives are usually neutral or acidic phosphoric acid ester derivatives, and their metal, amides, or amine salts [16]. The reactivity of phosphorus-based additives increases with higher acidic levels and decreases with more neutral forms. Initially developed for aircraft engines as antioxidants, phosphate esters were found to be effective AW additives to lubricants for automobile engines [19] and refrigeration compressors [20]. Neutral phosphoric acid esters include trialkyl and triaryl phosphates, the most popular being tricresyl phosphate, which has been used for friction and wear reduction since the 1940s [16,21]. Tricresyl phosphate operates through the formation of a multilayer film on steel surfaces that acts as a lubricious polymer [22]. The lubricious coating is maintained under wear from the diffusion of iron through the phosphate film, which is the rate-determining step in the film formation [23].

The best AW agents are those that combine the properties found in both sulfur-based compounds and phosphorus-based compounds. The most important and well known additive agents are the zinc dialkyldithiophosphates (ZDDPs), which are sulfur-phosphorus compounds. ZDDPs are perhaps the most effective multifunctional AW agents and can satisfy EP needs as well. Extra functions of ZDDPs include the ability to impede corrosion of the metal surface and trapping of free radicals and peroxides that cause lubricant oils to oxidize [18]. These additives are synthesized from the reaction of phosphorus(V)sulfide with either primary and secondary alcohols (C_3–C_{12}) or alkylated phenols [16]. This is followed by a neutralization reaction between the resulting dialkyldithiophosphoric acid with zinc oxide [16]. The AW/EP performance of ZDDPs, along with thermal and hydrolytic stability, are influenced by the structure of the alkyl groups. These versatile attributes make ZDDPs one of the most cost-effective and widespread additives, finding integral applications in engine oils, shock absorber oils, and hydraulic fluids [16]. However, ZDDPs also have several hindrances that require the mindful implementation of this additive. For one, ZDDPs can interfere with the performance of other antioxidant additives, which limits additive compatibility [18]. They also can form soluble and insoluble degradation products [18]. ZDDP additives can interfere with the performance of catalytic converters, warranting limited use in automotive lubricants [18]. A final hindrance comes from regulatory measures limiting sulfated ash and sulfur-phosphorus compound emissions in automobile applications [18].

A different approach with AW/EP additives uses nanomaterials to reinforce the lubricant. Nanolubricant additives can come in three different flavors: nanometal-based, nanocarbon-based, and nanocomposite-based additives. A promising candidate for a nanometal-based additive is Cu and CuO nanoparticles. In particular, CuO nanosheets serve as an excellent additive to self-lubricating carbon fibers reinforced polytetrafluoroethylene (PTFE), enhancing the AW performance [24]. Additionally, metal hydroxides like La(OH)3 nanoparticles, when added to a base oil, have proven to strengthen friction reduction and AW properties [25]. Nanocarbon-based additives provide excellent tribological properties in conjunction with being environmentally friendly, making these additives an integral component of a sustainable future. The addition of PTFE nanoparticles to lithium greases can enhance the friction reduction and AW properties [26]. The AW and EP attributes of lithium greases can also be enhanced from a combination of MoS_2 and graphite nanoparticles (40:60 ratio) at a 5% additive level [27]. Graphene and graphite-based additives continue to be investigated and have shown promising performance as lubricant additives. Deriving ultrathin graphene from graphite oxide has shown an improvement in AW performance of 33% and EP performance of 40% [28]. The final style of nanolubricant additive, nanocomposite, benefits from synergistic lubricating effects on the composite tribofilm. Nanocomposites can pull the best attributes from different source materials to create a multifaceted additive. Carbon nanotubes are one source material often used in nanocomposites. Developments such as poly(vinyl alcohol)-carbon nanotube composites (PVA-CNTs) and room temperature ionic liquid (RTIL)/multi-walled

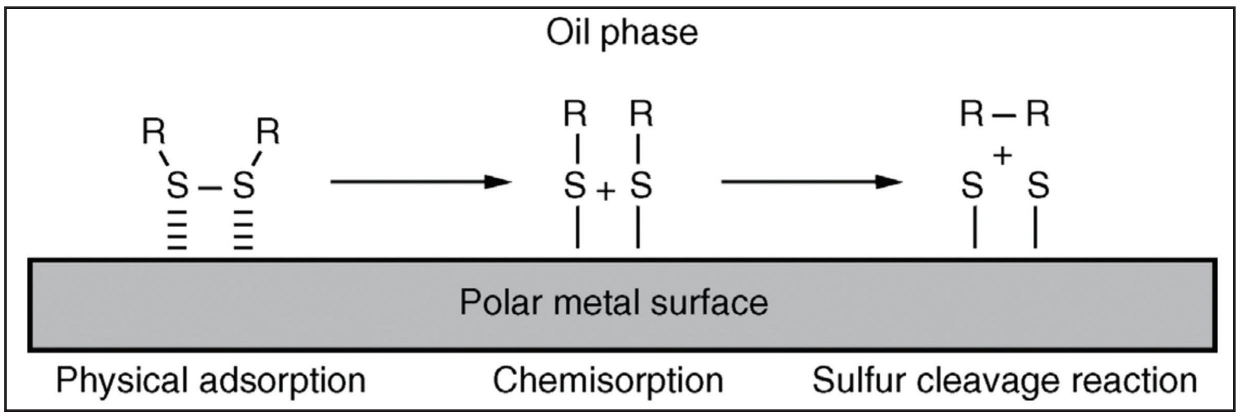

Figure 3: Sulfur carrier mechanism under EP conditions [16]

carbon nanotubes (MWNTs) composites have both shown improved AW properties such as better load-carrying capacity and wear resistance [29,30].

Several other miscellaneous types of AW lubricant additive chemistry have been investigated. The first of these is boron-based additive compounds. Boron additives work well as an adjunct to phosphorus additives for increasing AW performance [5]. However, common limitations hold back boron, including issues stemming from its compounds hydrolyzing in water and incompatibility with some other AW/EP additives [5]. Halogen-based additives were some of the earliest AW/EP agents used in the lubricant industry, particularly chlorine. Chlorine additives used in conjunction with sulfur demonstrated good AW and EP properties and were commonly used in the cutting oil industry [5,31,32]. Ecological shortcomings of chlorine-based AW/EP additives include the high reactivity to form compounds that pollute water and harm animals has prompted legislation to limit chlorine content in lubricants to parts per million, making their application in modern lubricants impractical. Iodine and fluorine-based additives have also shown some AW properties in albeit niche applications such as aluminum processing. Nitrogen is another element that has shown good AW properties when used with sulfur, such as in N-heterocyclic compounds [33,34]. A final compound that shows good EP properties are naphthenic acid salts of metals like lead and tin, especially when used with sulfur [5]. However, these compounds are held back by ecological issues, as well as poor thermal and oxidative stability with regards to lead naphthenate specifically [5].

It is easy to see the complexity and multitude of different AW and EP additives that are used every day. Additive development for lubricants and greases continues to be an important aspect for maintaining tribological systems through the reduction of wear. Research into additive development aims to increase performance and efficiency sustainably. Of course, additive development represents just one aspect of a bigger system of tribological measures taken to enhance machinery performance. However, the unique ability to modulate the characteristics of lubricants and fine-tune their properties makes the pursuit of perfecting these additives a worthwhile endeavor. The sheer number of applications of wear preventative additives, each with their own operating conditions, requires these additives to be custom-made fit for the role. The many nuances and cost-saving potential of EP/AW additives make their development forever relevant.

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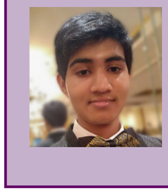

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