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Interaction effects of hexagonal boron nitride, zinc dialkyldithiophosphate and sulfurized isobutylene on tribological performance and stability of lithium grease

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Abstract. The development of advanced lubricating materials with tailored functional properties is an important area of research in materials science and nanotechnology. The present study investigates the interactions of nanoparticles and microparticles of hexagonal boron nitride (h-BN), zinc dialkyldithiophosphate (ZDDP) and sulfurized isobutylene (SIB) in lithium grease, focusing on their influence on tribological properties and thermal and mechanical stability. The base grease was formulated from a mineral base oil and lithium 12-hydroxystearate. ZDDP, SIB, and h-BN were used as additives, with h-BN introduced at mass concentrations of 3% and 5%. The dropping point and mechanical stability of greases were determined. Tribological performance was assessed using a four-ball apparatus under a constant load of 300 N and different test durations. The tests were complemented by analysis of wear scar morphology using scanning electron microscopy and chemical composition using energy-dispersive spectroscopy. The most favorable anti-wear performance was obtained for the grease containing ZDDP, SIB, and 5% h-BN, for which the mean wear scar diameter decreased by 27.2–39.8%, the dropping point increased by 9.6%, and mechanical stability improved by 55.2% compared with the base grease. The improved performance is attributed to the formation of a tribochemical layer generated by ZDDP and SIB and the participation of h-BN particles in the formation and stabilization of the lubricating film. The results demonstrate that the synergistic action of conventional additives and nanoparticles and microparticles of h-BN enables the design of lithium greases with enhanced functional performance and improved structural stability.

Key words: hexagonal boron nitride; ZDDP; SIB; greases; tribological performance.

1. INTRODUCTION

The operational durability and reliability of engineering systems are strongly related to the effectiveness of lubrication in frictional contacts. Greases represent an important class of lubricants applied in numerous tribological systems operating under different motion kinematics and contact configurations. In technical practice, many types of greases are used, and continuous research is conducted to improve their performance. In this process, the selection of appropriate additives is of key importance.

Although additives are typically present in relatively small amounts, they play a decisive role in determining the functional properties of greases. Among the additives used in grease formulations, solid lubricants constitute a particularly important group. These materials are introduced in the form of solid particles and display natural lubricating ability associated with their specific structural features and advantageous physicochemical characteristics [1, 2]. One representative of this group is hexagonal boron nitride (h-BN).

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Relatively little attention has been devoted so far to the use of h-BN as an additive in greases. Previous studies report beneficial tribological effects resulting from the incorporation of h-BN into grease formulations, including reduced wear of elements [3-16], decreased friction resistance [3, 4, 8-12, 14-17], and improved vibration suppression in bearings [14, 15]. Additionally, the influence of this material on other performance properties of greases has also been investigated [4, 5, 8, 12, 13, 18].

The performance of greases containing hexagonal boron nitride is governed by several factors, as shown in previous studies [19-21]. The most important include the physicochemical properties of h-BN, particularly its concentration, the proportion of nanoparticle and microparticle fractions, and particle porosity. The type of base grease and its properties are also of significant importance. The interaction between these factors affects both the tribological characteristics of greases and their other performance properties.

In practice, greases are usually supplemented with packages of additives. In several previous studies, the effectiveness of combinations of solid lubricants has been evaluated, in which

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h-BN was mixed with aluminium oxide [14], calcium carbonate [15], or molybdenum disulfide [16]. However, the interaction between h-BN and widely used additives, which are often applied together with solid lubricants, remains insufficiently investigated. The lack of such studies limits the assessment of potential synergistic or antagonistic effects between h-BN and other additives used to improve tribological performance of greases. This can be attributed to the different mechanisms of action of solid lubricants and commonly used anti-wear additives. The simultaneous presence of these components in the friction zone may significantly influence the formation of the lubricating film.

Among the most widely used additives in greases are zinc dialkyldithiophosphate (ZDDP) and sulfurized isobutylene (SIB). These substances are characterized by high thermal stability and good solubility in the liquid phase of grease. Zinc dialkyldithiophosphate is a multifunctional additive acting as an antioxidant, corrosion inhibitor, and anti-wear protectant. Under frictional conditions, its action is associated with tribochemically initiated reactions with the metal surface, leading to the formation of protective phosphate–sulfide layers that limit direct contact between friction elements and reduce wear intensity. Sulfurized isobutylene exhibits similar anti-wear properties, and its effectiveness results from reactions with the metal surface and the formation of sulfide layers under conditions of elevated temperature and high contact pressures. Then, the mechanism of action of both compounds involves adsorption processes and chemical reactions leading to the formation of a protective lubricating film [22, 23]. The operating mechanism of these compounds differs substantially from that characteristic of solid lubricants, which may lead to complex interactions during their simultaneous operation in the friction zone. Therefore, investigating the interaction of h-BN, ZDDP, and SIB in greases is justified.

The objective of the present study was to examine the effect of hexagonal boron nitride, zinc dialkyldithiophosphate and sulfurized isobutylene, applied individually and in combination, on the properties of lithium grease.

2. EXPERIMENTAL METHODS

In the present study, a lithium grease prepared from mineral base oil and thickened with lithium 12-hydroxystearate was used as the base sample. This composition, denoted as Li, did not contain any additives. A modified grease labelled Li-Add was formulated by introducing zinc dialkyldithiophosphate at a concentration below 1 wt.% and sulfurized isobutylene at 0.3 wt.%. Hexagonal boron nitride was subsequently added into both greases at concentrations of 3 wt.% and 5 wt.%. The physicochemical characteristics of the h-BN used in the study were described in detail in [19] and summarized in Table 1.

The preparation of grease samples containing hexagonal boron nitride involved weighing the appropriate amounts of individual components followed by their dispersion in the base grease. Mechanical mixing was carried out in a rotary bowl with a capacity of 3.5 L using a mixer with a rated power of 300 W. The process lasted 60 min and was conducted at the lowest speed level of the device. After mechanical blending, the samples were additionally treated in an ultrasonic bath for 30 min to improve the dispersion of solid particles within the grease matrix. During sonication the samples were placed in sealed metal containers. All preparation steps were performed at room temperature.

TABLE 1. Selected parameters defining the physicochemical properties of h-BN [19]

Parameter	Unit	Value
Apparent Diameter of Particles	nm	130
Part of Nanoparticles	%	33.4
Specific Surface Area	$m^2 \cdot g^{-1}$	30
Total Pore Volume	$cm^3 \cdot g^{-1}$	0.15

Compositions obtained by introducing hexagonal boron nitride into the Li grease were designated Li-3BN and Li-5BN. Analogous samples were prepared from the Li-Add formulation and labelled Li-Add-3BN and Li-Add-5BN. The penetration values and dropping points previously determined for Li, Li-3BN and Li-5BN were reported in [20]. In the present study, these data served as reference values for evaluating the influence of h-BN in greases additionally containing ZDDP and SIB.

The dropping point of the greases was determined using a high temperature dropping point apparatus (Model K19410, Tusnovics Instruments, Kraków, Poland) in accordance with the procedure specified in standard [24]. Three measurements were carried out for each sample and the mean value of the dropping point, denoted as DP, was calculated.

Penetration measurements were performed using a Stanhope-Seta 1700 penetrometer (Stanhope Seta, Chertsey, Surrey, United Kingdom) together with a mechanical grease worker Stanhope-Seta 1780. A standard cone with a base diameter of 76.2 mm and a mass of 150 g was used. The measurements followed the procedure described in standards [25, 26]. First, penetration after 60 working strokes was measured to determine the NLGI consistency grade of the greases. Subsequently, penetration after prolonged working consisting of 10000 strokes was determined. Mechanical stability of the greases (ΔP) was calculated as the difference between the penetration measured after 10000 strokes and the value obtained after 60 strokes. Each measurement was performed three times and average penetration values were used to determine mechanical stability.

Tribological properties of the greases were investigated using a T-02 four-ball apparatus (Łukasiewicz Research Network – Institute for Sustainable Technologies, Radom, Poland). The tribological system and the configuration of the friction pair are schematically illustrated in Fig. 1. The experiments were conducted at room temperature under a constant load of 300 N applied to the friction node, corresponding to an initial Hertzian contact pressure of approximately 3150 MPa. The load level was selected according to the relevant standard [27]. Test durations were 60, 300, 900 and 1800 s, while the rotational speed of the upper ball was maintained at 1450 rpm. The friction pair consisted of standard balls used in four-ball testing with a diameter of 12.7 mm and a hardness of 63.4 HRC. The balls were manufactured from 100Cr6 bearing steel [28], corresponding to AISI 52100 and DIN 1.3505. After each test the wear of the steel balls was evaluated by

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determining the wear scar diameter. Measurements were performed on each ball in directions parallel and perpendicular to the wear traces with an accuracy of 0.001 mm using an MP-1 microscope. Each grease was tested three times and the mean wear scar diameter (MWSD) was calculated.
Following the tribological tests, selected sample surfaces were analysed using scanning electron microscopy (SEM) combined with energy dispersive spectroscopy (EDS). Observations were carried out with a Zeiss Ultra Plus microscope (Zeiss, Oberkochen, Germany) equipped with a Bruker Quantax 400 EDS system (Bruker, Billerica, MA, USA). SEM imaging was performed using an accelerating voltage of 3 kV, whereas chemical composition analysis was carried out at 15 kV. Elemental composition was determined in selected microareas located within the wear scars. For comparison purposes, an unworn steel ball was also analysed.

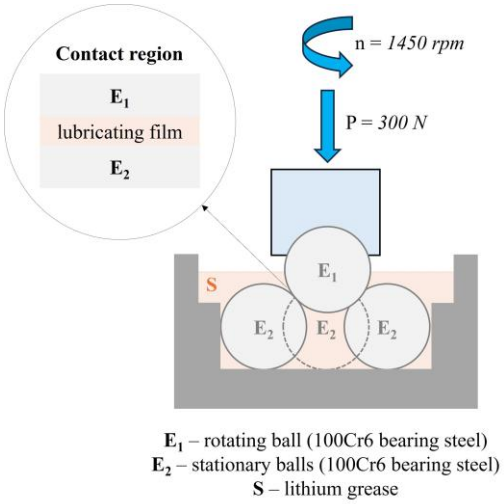


Fig.1. Schematic representation of the tribological system in a four-ball apparatus

3. RESULTS AND DISCUSSION

The introduction of additives into the lithium grease noticeably influenced its thermal stability (Fig. 2). The base grease (Li) exhibited a dropping point of 204.2°C, which was the lowest value among the analysed compositions. The addition of zinc dialkyldithiophosphate and sulfurized isobutylene increased this parameter to 213.0°C, corresponding to an improvement of approximately 4.3% relative to the base grease. A comparable effect was observed for the Li-3BN composition, where the dropping point reached 211.6°C, representing an increase of 3.6% compared with the reference grease. A more pronounced improvement in thermal stability was obtained after the addition of 5 wt.% hexagonal boron nitride, resulting in a DP value of 216.1°C. The highest dropping point values were recorded for greases containing the combined system of h-BN, ZDDP and SIB. In the case of Li-Add-3BN and Li-Add-5BN, the dropping point increased by 7.0% and 9.6%, respectively, relative to the base grease, reaching 218.4°C and 223.9°C.
The consistency and mechanical stability of the investigated greases were also affected by their composition (Fig. 3). The worked penetration of the base grease (Li) was 287, corresponding to NLGI consistency grade 2. A slightly higher value of 289 was recorded for the Li-Add grease; however, this difference did not result in a change in the consistency class relative to the base sample. A similar situation was observed for the Li-3BN composition, for which the worked penetration reached 294. The remaining greases exhibited penetration values between 296 and 305, corresponding to NLGI grade 1.5. More distinct differences became evident when mechanical stability was analysed. The introduction of ZDDP and SIB into the base grease led to a considerable increase in penetration change during prolonged working. The Li-Add composition showed the largest difference between penetration measured after 60 and 10000 strokes, reaching ΔP = 40. In comparison, the base grease Li exhibited a smaller change in this parameter (ΔP = 29). The addition of hexagonal boron nitride significantly reduced penetration changes during prolonged working. For greases containing h-BN, the ΔP values ranged from 11 to 15, indicating a substantial improvement in mechanical stability relative to both the base grease and the Li-Add sample.

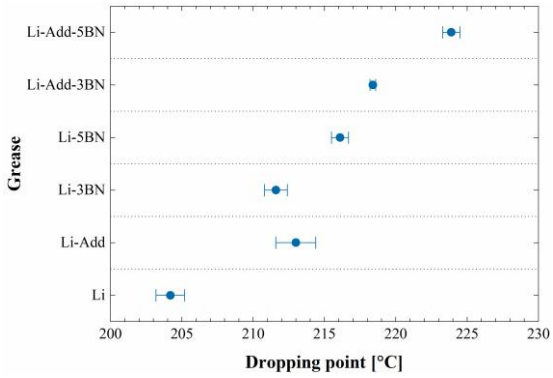


Fig.2. Dropping point of greases

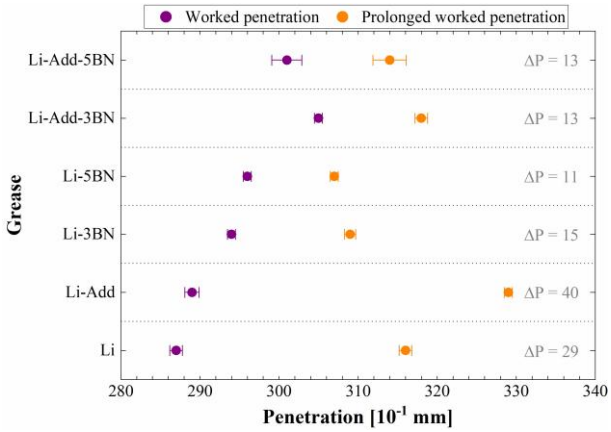


Fig.3. Penetration values and mechanical stability of greases

The values of the mean wear scar diameter (Fig. 4a) clearly demonstrated that the composition of the grease strongly influenced the wear process under the test conditions applied in the four-ball apparatus. The highest wear of the steel balls was observed when the base grease (Li) was used as the lubricant. In this case, the mean wear scar diameters were 0.397, 0.600, 0.652 and 0.738 mm, respectively. The use of the Li-Add sample led to a systematic reduction in wear throughout the analysed loading time range of the four-ball test. Compared with the base grease Li, the reduction in mean

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wear scar diameter ranged from 10.6% to 23.7%. A comparable improvement was obtained when hexagonal boron nitride was introduced into the base grease. For the Li-3BN and Li-5BN compositions, the decrease in mean wear scar diameter relative to Li varied between 13.2% and 24.2%. In most measurement points, slightly lower wear values were observed for the Li-5BN sample. Even greater reductions in wear were recorded for greases containing both conventional additives and hexagonal boron nitride. For the Li-Add-3BN and Li-Add-5BN compositions, the mean wear scar diameter was smaller by 27.2–39.8% compared with the base grease Li. Among all analysed variants, the lowest wear was obtained for the Li-Add-5BN sample, for which the MWSD values were 0.285, 0.361, 0.434 and 0.490 mm, respectively.

The evolution of the mean wear scar diameter over time (Fig. 4b) revealed different wear dynamics for the analysed compositions. For the base grease (Li), the increase in MWSD between 60 s and 1800 s reached 0.341 mm. The addition of hexagonal boron nitride into the reference sample reduced the rate of MWSD increase, lowering the increase in MWSD to the range of 0.302–0.309 mm. A more pronounced reduction in the increase in MWSD was observed for the Li-Add composition, for which the MWSD increased by 0.257 mm. A further decrease in the increase in MWSD over time was recorded for the Li-Add-3BN grease, where the increase in MWSD amounted to 0.213 mm. The smallest change in wear between the initial and final test points was obtained for the Li-Add-5BN composition, reaching only 0.205 mm. Such a clear reduction in the rate of MWSD increase suggests improved durability and stability of the protective layer formed in the presence of all three additives during prolonged loading of the friction node in the four-ball test.

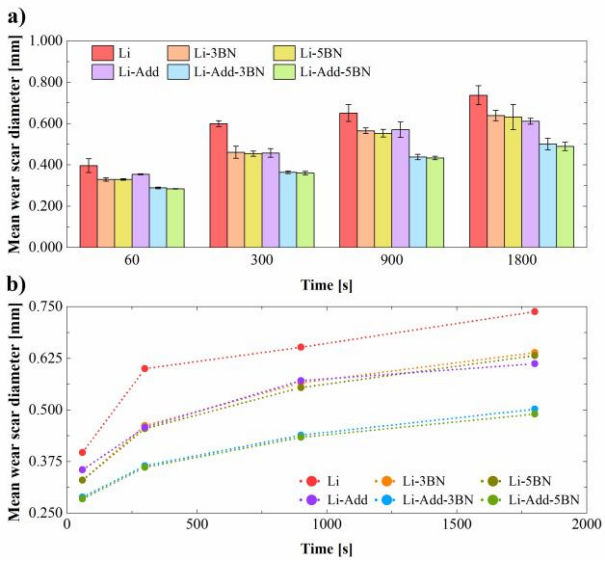


Fig.4. Tribological tests results: a) mean wear scar diameter after different test duration, b) changes in wear scar diameter as a function of test duration

The morphology of the wear scars was examined for selected grease compositions after the longest tribological test lasting 1800 s. The surface lubricated with the base grease Li (Fig. 5a) showed pronounced damage and a distinctly heterogeneous structure. A more uniform wear scar was observed for the Li-Add sample (Fig. 5b), although local imperfections were still present in the analysed area. The application of the Li-5BN composition (Fig. 5c) also resulted in an improvement in the condition of the friction surface. This was manifested by a smaller number of pronounced grooves and a reduction in localized damage compared with the surface lubricated with the base grease. The most homogeneous surface morphology was recorded for the Li-Add-5BN grease (Fig. 5d), which was consistent with the lowest MWSD values obtained in the tribological tests.

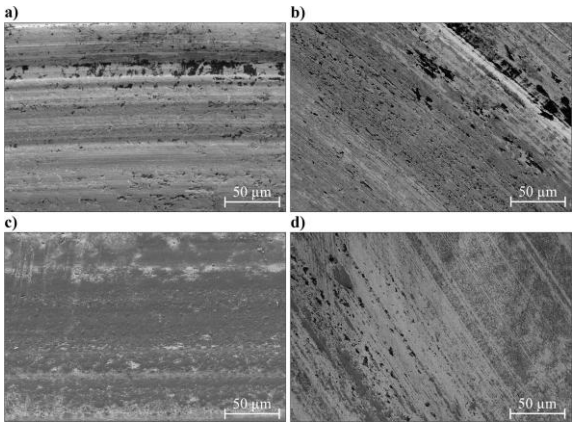


Fig.5. Wear scars surfaces: a) Li, b) Li-Add, c) Li-5BN, d) Li-Add-5BN

The microanalysis of the chemical composition within the wear scars provided additional information supporting the tribological results (Table 2). On the surface of the reference ball, elements typical of bearing steel were identified, including iron, chromium, manganese and silicon. In the wear scar produced under lubrication with the Li grease, the presence of oxygen was additionally detected. For the Li-Add grease, apart from the elements characteristic of the steel, zinc, phosphorus, sulfur and oxygen were also identified. In contrast, the wear scar formed during lubrication with the Li-5BN grease additionally contained boron. The best anti-wear performance was observed for the Li-Add-5BN sample, which combined hexagonal boron nitride with zinc dialkyldithiophosphate and sulfurized isobutylene. The chemical microanalysis of the corresponding wear scar confirmed the simultaneous presence of boron, sulfur, phosphorus, zinc and oxygen.

TABLE 2. Mass participation of elements detected on the friction surface

Element	Unworn ball	Li	Li-Add	Li-5BN	Li-Add-5BN
Fe	97.95	92.45	71.71	85.55	85.37
Cr	1.57	1.30	1.17	1.30	1.27
Mn	0.19	0.14	0.26	0.31	0.19
Si	0.29	0.21	0.17	0.22	0.28
O	-	5.90	11.28	4.13	3.92
P	-	-	4.44	-	0.51
S	-	-	4.79	-	1.48
Zn	-	-	6.18	-	0.36
B	-	-	-	8.49	6.62

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The combined analysis of tribological results, surface morphology observations and chemical microanalysis indicated that the composition of the grease significantly influenced the wear process under the applied friction conditions. The highest wear observed for the base grease Li can be attributed to the absence of additives with anti-wear functionality, which limited the formation of a sufficiently durable protective layer within the friction zone.

For the Li-Add composition, the reduction in wear and the improvement in surface condition compared with the base grease were associated with the presence of zinc, phosphorus and sulfur detected in the surface layer. The action of ZDDP is related to tribochemically initiated reactions that lead to the formation of protective films composed mainly of metal sulfides as well as zinc phosphates and polyphosphates [22, 23]. Sulfurized isobutylene, in turn, participates in reactions resulting in the formation of sulfur-containing surface layers under friction conditions [22, 23]. The detection of zinc, phosphorus and sulfur within the wear scar therefore suggests the formation of an anti-wear tribochemical layer characteristic of the operation of these additives.

The addition of hexagonal boron nitride into the lithium grease also resulted in a reduction of wear compared with the reference composition. The presence of boron detected within the wear scars confirmed that h-BN particles were able to reach the friction zone and participate in the formation of the lubricating film. Their action within the contact region may involve mechanisms characteristic of layered materials, including interlayer shear processes associated with the lamellar structure of hexagonal boron nitride.

The lubricating effectiveness of h-BN in the contact zone may also be related to particle size and the significant contribution of the nanoparticle fraction (Table 1). As reported previously for similar greases [21], h-BN nanoparticles can deposit in areas of local material loss on the rubbing surfaces. The accumulation of such particles in microdefects reduces the real contact between asperities and consequently lowers local contact stresses. Compared with larger particles, nanoparticles exhibit a greater ability to penetrate micro-contact regions. In addition, their developed porous structure and high surface energy promote their retention within the friction zone and facilitate the formation of a stable protective layer. As a result, the presence of a nanometric fraction in the investigated h-BN may have played a significant role in the observed reduction of wear.

The most favorable tribological performance observed for greases containing ZDDP, SIB and h-BN concurrently may indicate the combined action of tribochemical processes initiated by ZDDP and SIB together with mechanisms associated with h-BN nanoparticles and microparticles. The presence of boron, zinc, phosphorus and sulfur within the wear scar suggests that all these elements participated in the formation of the protective surface layer. Tribochemical reactions related to the activity of ZDDP and SIB likely led to the formation of a film acting as the primary anti-wear barrier. At the same time, h-BN particles could interact within the contact zone by filling local discontinuities and stabilizing the developing layer in regions where it was weakened. The simultaneous operation of these mechanisms may therefore result in the formation of a lubricating film with enhanced resistance to wear.

The obtained results suggest that the observed improvement in tribological performance is associated with complex interactions occurring within the tribological system. These interactions may involve not only the tribochemical activity of ZDDP and SIB and the participation of h-BN particles in the contact zone, but also additional phenomena related to the interaction between the greases and metallic surfaces, including their possible catalytic influence. The combined effect of these processes may contribute to the formation and modification of the protective layer responsible for reducing wear.

An important factor influencing the tribological performance of the analysed compositions was the change in the thermal and mechanical stability of the grease resulting from the applied additives. The base sample Li exhibited the lowest dropping point among the investigated formulations and showed moderate mechanical stability. The introduction of ZDDP and SIB into the lithium grease led to an increase in the dropping point, which may be associated with the high thermal stability of these compounds. At the same time, the Li-Add composition exhibited the lowest mechanical stability. The presence of ZDDP and SIB, as components dissolved in the oil phase, likely affected the relationship between the liquid phase and the thickener network, modifying the structural balance of the grease and reducing its resistance to mechanical stress.

In contrast, the addition of hexagonal boron nitride had a beneficial effect on both the thermal and mechanical stability of the compositions. For greases containing only h-BN, an increase in dropping point and a noticeable reduction in penetration changes were observed. A similar tendency was recorded for compositions containing ZDDP, SIB and h-BN simultaneously. In this case, the solid particles appeared to compensate for the destabilizing influence of additives dissolved in the oil phase on the mechanical stability of the grease. As demonstrated previously [20], properly selected nanoparticles and microparticles of h-BN can integrate with the thickener network, complementing its structure and improving the resistance of the grease to mechanical degradation. The strengthening of the spatial structure of the grease may also support the continuity of the protective layer and limit its local damage during prolonged tribological tests. Consequently, a lower wear development over time was observed for compositions containing all three additives.

Ultimately, the improvement in anti-wear performance resulted not only from protective processes initiated by ZDDP and SIB and from mechanisms associated with h-BN nanoparticles and microparticles, but also from favorable modifications of the structural properties of the grease, particularly those related to the presence of the solid lubricant. The combined action of these effects contributed to the formation of a lubricating film with enhanced durability and resulted in a grease composition characterized by stable and well-balanced tribological, thermal and mechanical properties.

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4. CONCLUSIONS

Based on the experimental studies conducted, the results obtained and their analysis, the following conclusions can be formulated:

- The effectiveness of the analysed greases in reducing wear was determined by the simultaneous presence of zinc dialkyldithiophosphate, sulfurized isobutylene and hexagonal boron nitride. Their mutual interactions, as well as their influence on the thermal and mechanical stability of the compositions, played a key role.
- The best performance was obtained for the Li-Add-5BN composition. Compared with the base grease, the mean wear scar diameter was reduced by 27.2–39.8%, the dropping point increased by 9.6%, and the change in penetration reflecting mechanical stability decreased by 55.2%.
- The reduction in wear was associated with tribochemical processes initiated by ZDDP and SIB, leading to the formation of a protective layer whose durability was supported by the presence of h-BN nanoparticles and microparticles.
- The physicochemical properties of h-BN, particularly particle size, the content of nanoparticles and the developed porous structure, were important for stabilizing the grease structure and enhancing the durability of the protective layer. This resulted in reduced wear and a lower rate of wear progression over time.

5. ACKNOWLEDGEMENTS

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