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EFFECT OF HEXAGONAL BORON NITRIDE WITH DIFFERENT PARTICLE GRANULATION ON THE LUBRICATION PROPERTIES OF MINERAL OIL WITH AN ADDITION OF SURFACTANTS

WPŁYW HEKSAGONALNEGO AZOTKU BORU O RÓŻNEJ GRANULACJI CZĄSTEK NA WŁAŚCIWOŚCI SMARNOŚCIOWE OLEJU MINERALNEGO Z DOMIESZKĄ SURFAKTANTÓW

Key words: hexagonal boron nitride, base oil, lubricity, sedimentation, succinic acid imide.

Abstract: This paper presents the results of tribological tests of lubricating compositions containing SN150 mineral oil as a base, an additive of solid lubricant in the form of hexagonal boron nitrides (h-BN) of various particle sizes (nano- and micro-) and surfactants: succinic acid imide and sodium di-2-ethylhexyl sulphosuccinate. Particular attention was paid to analysing the properties of hexagonal boron nitrides in the context of their effect on the tribological properties of the friction node under study. The effects of h-BN particle size, shape, specific surface area and porosity were considered. In addition, tests were carried out to check the surfactants' effect on the tested oil's hexagonal boron nitride sedimentation process. These tests confirmed the positive effect of succinic acid imide on maintaining a stable dispersion of h-BN particles in SN-150 mineral oil. Tribological tests were performed on a T-02 tribotester based on the PN-EN ISO 20623:2018-02 standard. The positive effect of hexagonal boron nitride on the lubricating properties of mixtures with SN150 mineral oil was established. In addition, hexagonal boron nitride, with a smaller particle size and a more developed porous structure, had a more favourable effect on improving the lubricity properties evaluated on the T-02 tribotester.

Słowa kluczowe: heksagonalny azotek boru, olej bazowy, smarność, sedymentacja, imid kwasu bursztynowego.

Streszczenie: W artykule przedstawiono wyniki badań tribologicznych kompozycji smarowych zawierających jako bazę olej mineralny SN150, dodatek smaru stałego w postaci heksagonalnych azotków boru (h-BN) o różnej granulacji cząstek (nano- i mikro-) oraz surfaktanty: imid kwasu bursztynowego oraz sól sodowa sulfobursztynianu di (2-etyloheksylu). Zwrócono szczególną uwagę na przeanalizowanie właściwości heksagonalnych azotków boru w kontekście oddziaływania na właściwości tribologiczne badanego węzła tarcia. Uwzględniono wpływ rozmiarów oraz kształtu cząstek h-BN, powierzchnię właściwą oraz porowatość. Dodatkowo wykonano testy sprawdzające wpływ surfaktantów na proces sedymentacji heksagonalnego azotku boru w badanym oleju. Testy te potwierdziły pozytywne działanie imidu kwasu bursztynowego na utrzymanie stabilnej dyspersji cząstek h-BN w oleju SN-150. Badania tribologiczne wykonano na tribotesterze T-02, opierając je na normie PN-EN ISO 20623:2018-02. Ustalono pozytywne działanie heksagonalnego azotku boru na właściwości smarnościowe mieszanin z olejem SN150. Ponadto heksagonalny azotek boru o mniejszej granulacji cząstek i bardziej rozbudowanej strukturze porowatej korzystniej wpływał na poprawę właściwości smarnościowych ocenianych na aparacie czterokulowym T-02.

INTRODUCTION

The use of oil or grease is a common procedure to minimise the effects of wear on friction junction components. Conventional lubricants are increasingly less able to fulfil this criterion, and limitations are particularly evident under extreme machine operating conditions. Furthermore, in addition to operational requirements, environmental requirements determine the use of agents that do

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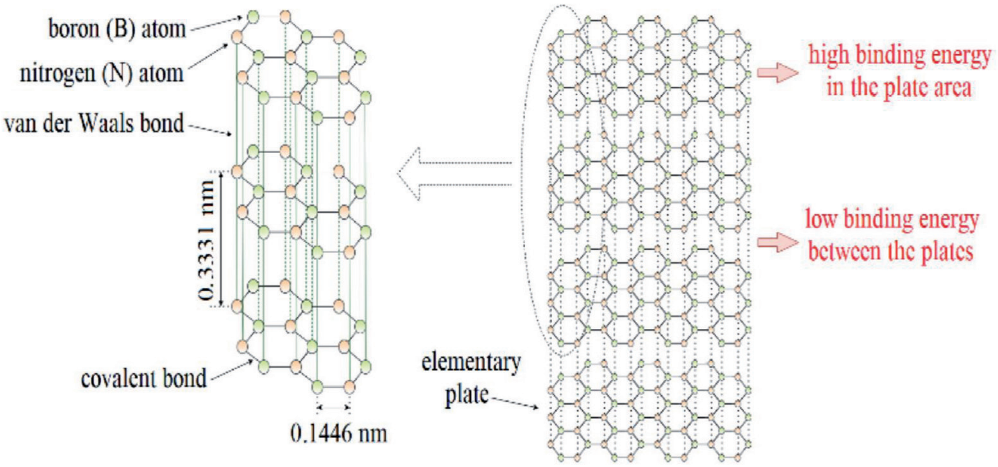

Fig. 1. Lamellar structure of hexagonal boron nitride [L. 3]
Rys. 1. Lamelarna struktura heksagonalnego azotku boru **[L. 3]**

not have a negative impact on the environment (so-called eco-friendly) **[L. 1]**. A diverse range of additives for lubricants and base oils are therefore used. One of these is solid lubricants with a lamellar structure, such as molybdenum disulphide (MoS2), graphite, graphene, tungsten disulphide (WS2) or hexagonal boron nitride (h-BN) **[L. 2]**.

The selected research material was hexagonal boron nitride (h-BN or α-BN). Compared to other solid lubricants, it is distinguished by the fact that it does not react with other substances, is safe for the environment, resistant to oxidation, chemically inert and particularly resistant to high temperatures **[L. 2, 4–6]**. Hexagonal boron nitride is one of the polymorphic varieties of boron nitride. It is characterised by a lamellar structure (**Fig. 1**) (similar to graphite). Chemical bonds between atoms are very strong, while interlayer bonds are very weak van der Waals bonds. This structure allows the h-BN layers to move between each other. Thus, friction between friction pairs is replaced by friction between individual nitride layer s **[L. 2, 7, 8]**. This mechanism is considered to favour better lubricity (reduction in wear and friction coefficient) **[L. 5, 9]**, favouring this solid lubricant's use in lubrication technology. However, not only the particle structure determines the efficiency of lubrication with hexagonal boron nitride. Researchers **[L. 10–12]** also pay attention to the added compound's appropriately selected concentration and granulation. In addition, a closer examination of the physicochemical properties, e.g. the granulometric distribution of the particles, identification of the particle shape and size, or the specific surface area of the material, is recommended. Such analysis enables a better understanding of the additive's effect in different tribological systems **[L. 3]**.

In recent years, nanoparticles have been an increasingly analysed area. Their sufficiently small size may result in better penetration of the friction node **[L. 13]**. By entering the gaps between friction parts, nanoparticles support the formation of additional layers with effective tribological properties. Also, some studies indicate a repairing effect of nanoparticles (filling microcracks and pores) **[L. 14–16]**. While nanoadditives have many advantages, they also have some limitations. The problem is the dispersibility and agglomeration of particles in the solution. One of the methods of obtaining a stable suspension of nanoparticles in lubricating oil is adding a dispersant **[L. 17–19]**.

The article aims to determine the effect of two types of hexagonal boron nitride on the lubricating properties of the SN150 mineral base oil. Surfactants were added to the mixtures. Before starting tribological tests, the properties of the hexagonal nitrides were identified, and the stability of the prepared mixtures was visually checked.

HEXAGONAL BORON NITRIDE – IDENTIFICATION OF THE PROPERTIES

The selection of the two analysed hexagonal boron nitrides was conditioned by their different properties. The objects were from two different manufacturers. The nitrides were characterised, among other things, by the fragmentation of the particles. The following code designations were adopted:

- A – hexagonal boron nitride with a fragmentation of less than 100 nm,
- B – hexagonal boron nitride with a fragmentation of less than 25 µm.

Tests were carried out to identify the physicochemical properties of both compounds. In addition to confirming the manufacturer's claimed results, a more complete characterisation may enable a better understanding of the effects of hexagonal boron nitride in a friction node. Detailed test methodologies are presented in a previous article **[L. 3]**.

Morphology and Particle Size Distribution

Scanning electron microscopy was used to identify the structure of hexagonal boron nitrides. Samples for the study were prepared by carefully spreading a small amount of powder onto carbon glue tape. In order to obtain better quality microscopic images, the samples were coated with a layer of gold. **Figure 2** shows the SEM images. Both samples contained lamellar-shaped particles, and agglomeration of the particles can be seen.

The particle size distribution histograms for the samples are shown in **Figure 3**. The area of each grain visible in the SEM images was determined and then converted to the same area of a circle of apparent diameter. The histograms show the arithmetic mean of the set of such apparent diameters. By analysing the photos under a microscope and assuming that the apparent diameter of the circle is the diameter of the sphere, the volume of each sphere was calculated and divided into size classes. After summing up the volumes of individual classes, the diameter D50

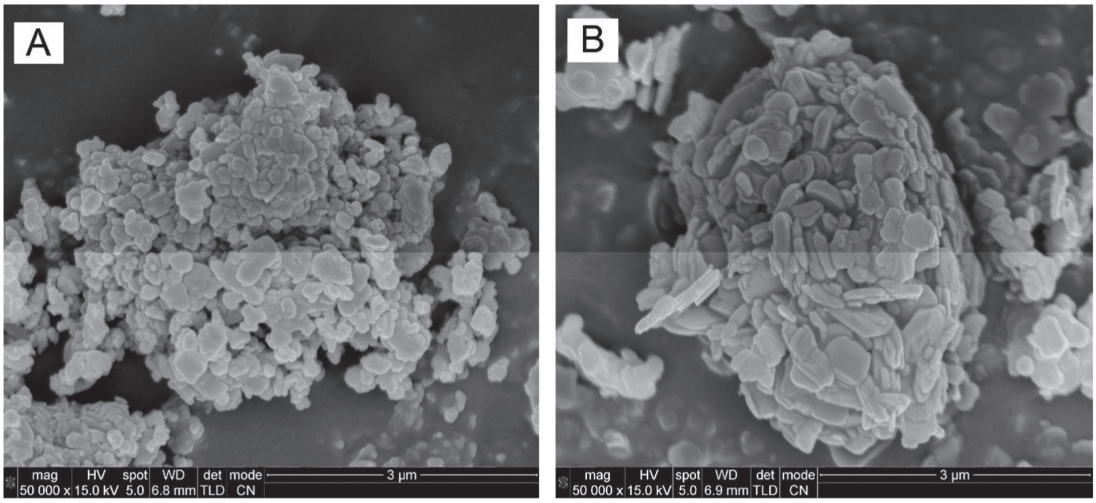

Fig. 2. SEM pictures of hexagonal boron nitride "A" and "B"
Rys. 2. Zdjęcia SEM heksagonalnego azotku boru „A" i „B"

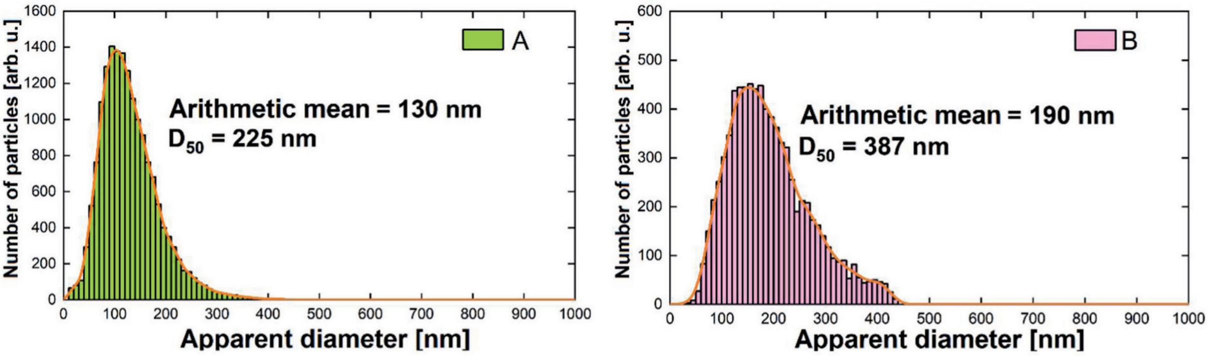

Fig. 3. Particle size distribution for h-BN "A" and "B"
Rys. 3. Rozkład granulometryczny cząstek h-BN „A" i „B"

was calculated, for which the smaller and larger grains represent half of the total volume. Sample A contained particles with the smallest diameters, a significant proportion of which can be classified as nanoparticles on this basis. Sample B contained larger particles.

Crystal Structure

The X-ray diffraction pattern for hexagonal boron nitrides is shown in **Figure 4**. The hexagonal boron nitride was identified by matching a suitable XRD reference standard. Sample A contained two types of hexagonal boron nitride with different structures. Space group No. 194 (P63/mmc) represents a material with a typical hexagonal crystal structure, and space group No. 160 (R3m) is a trigonal crystal system **[L. 20]**. This trigonal system is considered to be the rhombohedral version of the hexagonal structure **[L. 21]**.

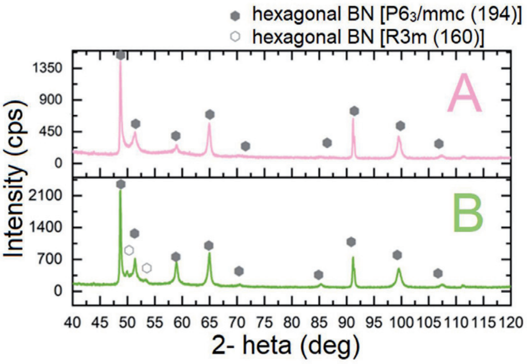

Fig. 4. X-ray diffraction patterns of h-BN samples "A" and "B"

Rys. 4. Dyfraktogramy rentgenowskie próbek h-BN „A" i „B"

Porosity

In order to determine the specific surface area and porosity characteristics, the pore size distribution (PSD) functions were determined (**Fig. 5**). The graphs also show the results of the SBET specific surface area and the total pore volume Vt. Similar values for these parameters were recorded in both samples. The dominant pore sizes are marked with arrows in the graphs. The peaks at 23 nm and around 4 nm characterise the mesopores, and sample A has smaller pores. A sharp peak at 0.5 nm, indicating the presence of micropores in the 'A' sample, is also characteristic.

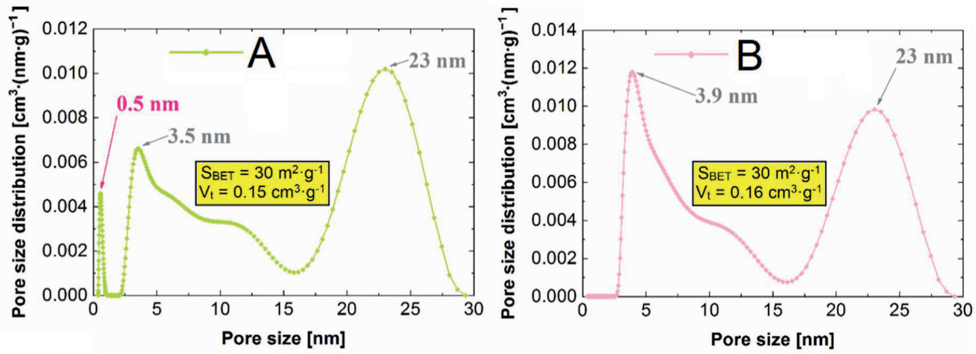

Fig. 5. Pore size distribution (PSD) functions for h-BN samples "A" and "B"

Rys. 5. Funkcje rozkładu wielkości porów (PSD) dla próbek h-BN „A" i „B"

MATERIALS AND METHODS

The composition of the mixtures

The hexagonal boron nitrides (A, B) research objects were applied to the oil base, and the mixture was supported with surfactants. Characteristics of the hexagonal boron nitrides were presented in the previous section.

Base oil

SN150 base oil (**SN150**) is used for the composition (together with an additive package) of lubricants. It is obtained from the vacuum distillation of crude oil. The basic properties of SN150 are shown in **Table 1**.

Table 1. Characteristics of SN150 base oil

Tabela 1. Charakterystyka oleju bazowego SN150

Parameter	Value
Density at 15°C	0.873 g/cm³
Kinematic viscosity at 100°C 40°C	 5.23 mm²/s 31.4 mm²/s
Viscosity Index	95

Surfactants

Succinic acid imide (**IKB**) (Merck, Darmstadt, Germany) is an ashless dispersant that enables solids to be retained in an oil suspension. The basic properties of succinic acid imide are shown in **Table 2**.

Sodium di-2-ethylhexyl sulfosuccinate **(S)** (PCC Exol SA, Brzeg Dolny, Poland) is a biodegradable surfactant with good dispersing, emulsifying and wetting properties. The typical properties of sodium di-2-ethylhexyl are shown in **Table 3**.

Table 2. Characteristics of succinic acid imide [L. 4]
Tabela 2. Charakterystyka imidu kwasu bursztynowego **[L. 4]**

Parameter	Value
Density at 15°C	0.927 g/cm^3
Kinematic viscosity at 100°C	440 mm^2/s
Flashpoint (PMC)	190°C
Total Base Number	42 mg KOH/g

Table 3. Characteristics of sodium di-2-ethylhexyl
Tabela 3. Charakterystyka soli sodowej sulfobursztynianu di(2-etylkoheksylu)

Parameter	Value
Density at 20°C	1.1–1.2 g/cm^3
Viscosity at 20°C	<20 cP
Cloud point	<20°C

Lubricant compositions

Test samples were made by measuring the appropriate amount (% m/m) of base oil, hexagonal boron nitride and surfactants and mixing them. The concentration of the hexagonal boron nitride was 2%, while the surfactants were 0.5%. The substances were then mixed with a mechanical stirrer (Steinberg Systems SBS-ER-3000, Poland) for 15 minutes and with an ultrasonic stirrer (Bandelin, Sonoplus HD 2200, Berlin, Germany) for 20 minutes at room temperature. Mixing was carried out immediately before the start of observations and before tribological tests to neutralise agglomerate formation. Six test samples were prepared in this way. Additionally, pure oil base, oil base with surfactants and oil base with hexagonal boron nitrides alone were prepared and tested for comparative purposes. Samples for sedimentation evaluation and tribological tests were prepared in the same way. The test sample designations adopted are presented in **Table 4**.

Table 4. Marking of prepared lubricating compositions
Tabela 4. Oznaczenia przygotowanych kompozycji smarowych

Sample	Lubricant compositions
P1	SN150
P2	SN150+0.5%IKB
P3	SN150+0.5%S
P4	SN150+0.5%IKB+0.5%S
P5	SN150+2%A
P6	SN150+2%A+0.5%IKB
P7	SN150+2%A+0.5%S
P8	SN150+2%A+0.5%IKB+0.5%S
P9	SN150+2%B
P10	SN150+2%B+0.5%IKB
P11	SN150+2%B+0.5%S
P12	SN150+2%B+0.5%IKB+0.5%S

APPARATUS AND RESEARCH METHODS

Stability of the compositions

The prepared test mixtures (P5-P12) were placed in litre cylinders with a scale of 100 ml. The measurement was based on visual observation of the scale and noting changes, and observations were made every 24 hours for 15 days. The test for samples P8 and P12 was terminated early due to no observed changes.

Tribological tests

Tribological tests were carried out using a T-02 four-ball apparatus designed and manufactured at the Institute for Sustainable Technologies in Radom, Poland **[L. 22]**. The standard PN-EN ISO 20623: 2018-02 **[L. 23]** was used to develop the research methodology. Tests lasting 10 seconds were carried out. The tests were carried out under a constant load at a spindle speed of 1450 rpm for 10 sec. The load was from the normative range (58.84; 68.64; 78.45; 88.25; 98.06; 107.87; 127.48; 137.28; 156.90; 176.51; 196.12; 215.73; 235.35; 274.56; 313.80; 353.02; 392.42; 441.28; 490.31; 549.12; 617.76; 686.40; 784.84; 882.56; 980.60; 1098.24; 1235.52; 1372.80; 1569.68; 1765.12; 1961.20; 2206.40; 2451.51; 2746.04; 3088.56; 3481.38; 3922.40; 4412.71; 4903.00; 5492.08; 6079.72; 6864.20; 7844.80 N). The test result was the mean wear scar diameter (MWSD), which is the mean of six wear scar diameter measurements, two from each of the stationary balls, taken in the direction of rubbing the balls and at right angles to this. Wear scars were measured with an optical microscope (Nikon ECLIPSE LV, Tokyo, Japan). The following parameters were determined:

- wear-load curve – a logarithmic plot of the load against the mean wear scar diameter;
- weld load **(WL)** – a fusion of metal between the rubbing surfaces sufficient for metal to merge and the balls to weld together in the form of a tetrahedron;
- Load-Wear Index **(LWI)** – index of the ability of a lubricant to minimise wear at applied load (load-carrying property of a lubricant) – determined based on 20 test runs before the weld load is reached;
- initial seizure load **(ISL)** – the lowest load at which seizure occurs – the value read from the weld-load curve.

RESEARCH RESULTS AND DISCUSSION

Stability

The stability of mixtures of hexagonal boron nitrides dispersions in the base oil was assessed by observational tests. This aimed to investigate the period during which hexagonal boron nitride can remain dispersed in the oil after the blending process. **Figure 6** shows the mixtures after 24 hours of standing. No changes were observed for the tests with hexagonal boron nitride with smaller particle size (A) supported by succinic acid imide. In the remaining samples, instability of the mixtures was observed. The worst effects were observed for sulfosuccinate-assisted samples (P8, P12). After 15 days of observation (**Figure 7**), samples P6 and P10 remain stable. The test for samples P8 and P12 was decided to end after nine days of observation due to no change.

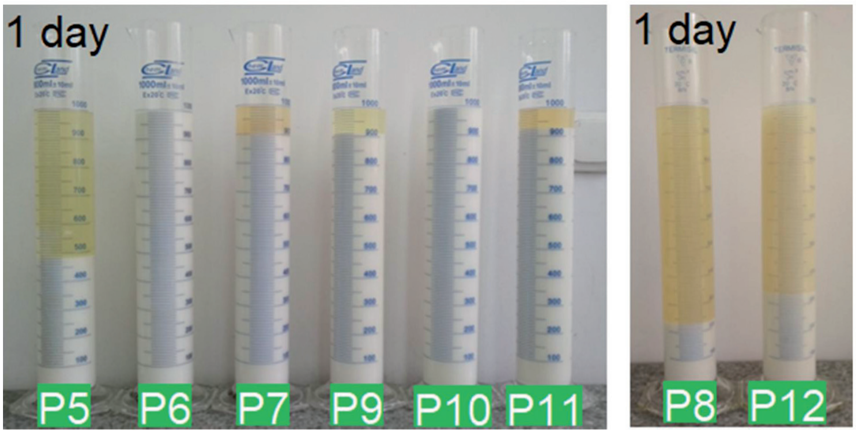

Fig. 6. Stability results of the mixtures checked after 24 hours
Rys. 6. Wyniki stabilności mieszanin sprawdzone po 24 godzinach

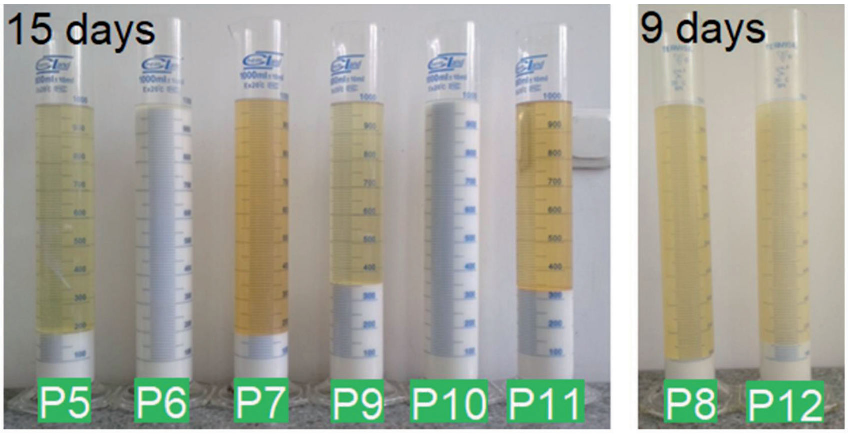

Fig. 7. Stability results of mixtures checked after 15 days for samples P5-P7, P9-P11 and nine days for samples P8 and P12
Rys. 7. Wyniki stabilności mieszanin sprawdzone po 15 dniach dla prób P5-P7, P9-P11 oraz 9 dniach dla prób P8 i P12

Tribological tests

Despite the lack of stability in all tests as a function of time, it was decided to carry out comprehensive tribological tests on a T-02 tribotester. The mixtures were tested immediately after preparation, and particle agglomeration was thus avoided. The P1-P12 mixtures were investigated.

The wear-load curve is shown in **Figure 8**. From the curve, the lowest load at which seizure occurred for the individual tests was read. When analysing the results of the initial seizure load results, it was noted that the oil base test achieved the worst value (P1). This was improved by the use of hexagonal boron nitride, which prevented galling. Putting together the samples that achieved the best results in the stability test, it can be seen that improvements were also achieved in the tribological tests. Better values were achieved by sample P6 with a nitride with a higher nanoparticle content and a more developed porous structure (P5-P8). This is likely to be due to the phenomenon of better penetration of smaller particles into the friction node areas and better bonding of the nitride particles to the oil. The combination of surfactant-free mixtures also supports the above hypothesis. The effect of succinic acid imide itself on the improvement of the ISL parameter was ruled out by combining pure base oil with a mixture of base oil and this surfactant. Both tests achieved the same value. The effect of the second surfactant – sulfosuccinate (S) – is surprising. All trials containing it achieved the highest values. This indicates a good anti-wear effect of this substance.

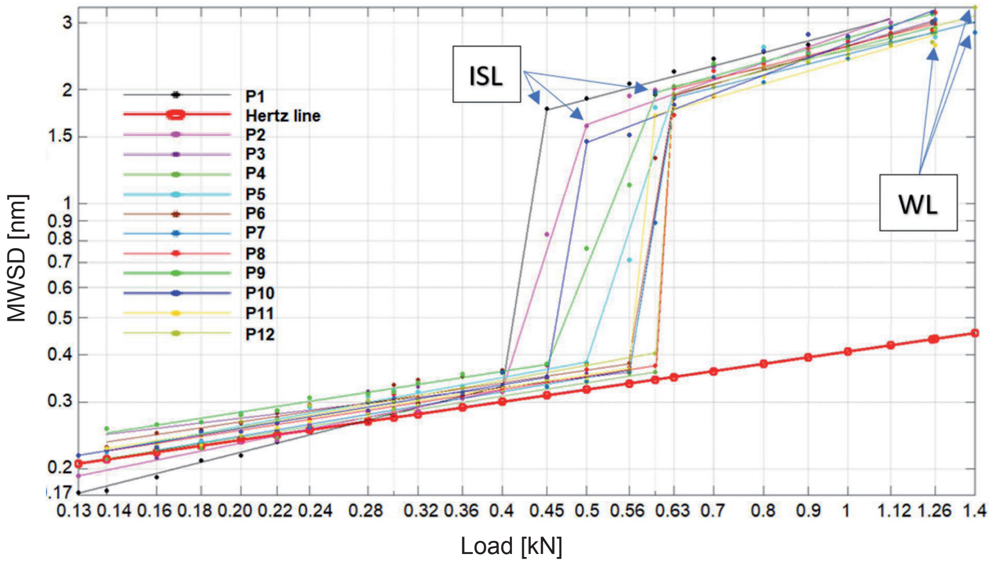

Fig. 8. Wear-load curve
Rys. 8. Krzywa zużycie–obciążenie

Comparing the results of the weld load (WL) parameter, it can be concluded that the hexagonal boron nitride has a greater effect on preventing the welding of the balls of the node of the four-ball apparatus in relation to the pure oil base (**Fig. 10**). A better value was achieved by h-BN with a smaller particle size. The parameter's value was the same with nitride alone and with the addition of succinic acid imide. In the case of using IKB alone, the result was the same as for the pure base oil. The samples with the addition of sulfosuccinate achieved good values. The exception, however, is sample P8.

The LWI index, calculated based on 20 test runs, made it possible to compare the mixtures in terms of load carry loads. This parameter characterised the entire scope of the test (**Fig. 8**). The results are presented in **Table 5**. The lowest values were obtained for pure base oil (P1) tests and the mixture of base oil and succinic acid imide. The samples reached the highest values with sulfosuccinate (P3, P4, P7, P8, P11, P12). The sulfosuccinate may form an additional lubricating film on the surfaces, increasing the lubricity. Values higher than pure oil base indicated mixtures with hexagonal boron nitrides. Again, test results with hexagonal boron nitride A are better than those for hexagonal boron nitride B.

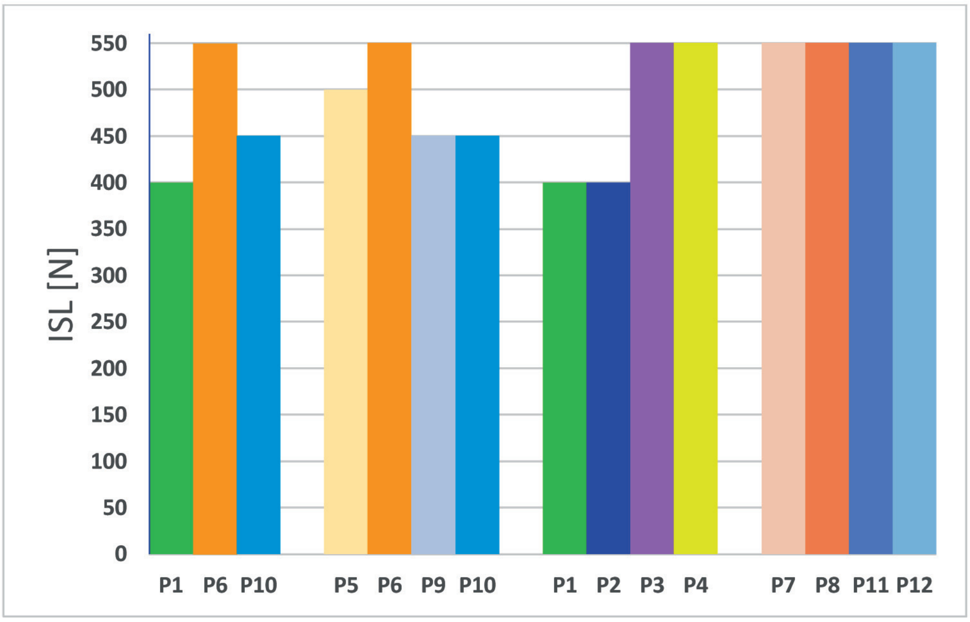

Fig. 9. The results of the initial seizure load (ISL) tests
Rys. 9. Wyniki badań najniższego obciążenia zacierającego (ISL)

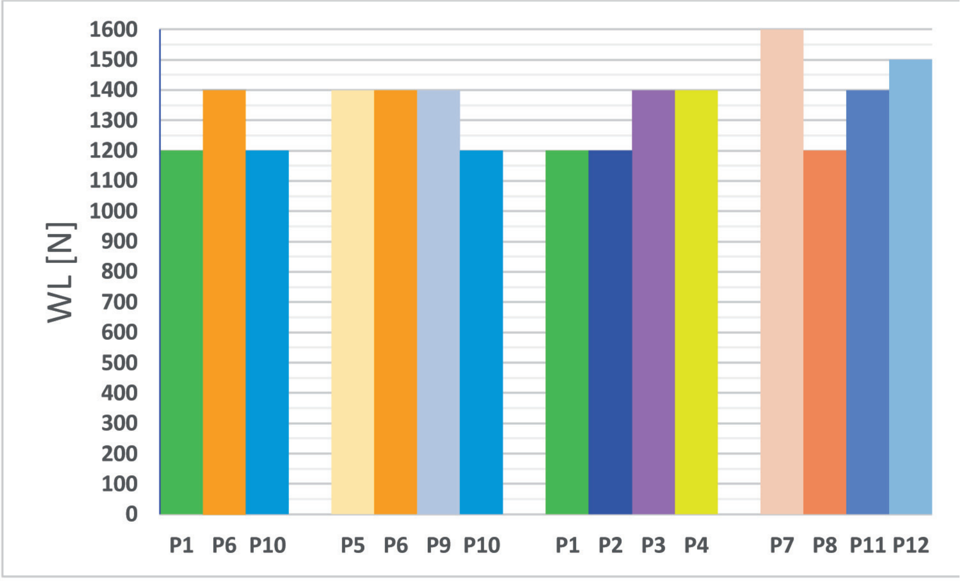

Fig. 10. The results of the weld load (WL) tests
Rys. 10. Wyniki badań obciążenia zespawania (WL)

Table 5. The results of the Load-Wear Index (LWI) parameter
Tabela 5. Wyniki parametru Load-Wear Index (LWI)

Sample:	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10	P11	P12
LWI(10s) (N)	180	180	230	240	210	220	240	230	190	190	240	230

CONCLUSIONS

The article's main aim was to evaluate the effect of hexagonal boron nitride with different particle sizes on the tribological properties of mineral oil with surfactant admixture.

Based on the research, the following conclusions were made:

1. Identification of selected hexagonal properties of boron nitrides (morphology, shape, size, porosity) allows for a more detailed explanation of the differences in the results of tribological tests.
2. Succinic acid imide assisted in maintaining a stable oil dispersion, allowing h-BN particles to work effectively in the friction area. Samples containing this dispersant performed well in anti-seizure tests. On the other hand, the sulfosuccinate showed excellent lubricity properties, but it accelerated the sedimentation process of h-BN particles in the oil.
3. Friction tests on a T-02 tribotester confirmed the positive effect of hexagonal boron nitride on the lubricating properties of mixtures with SN150 mineral oil.
4. Hexagonal boron nitride (A) with a smaller particle size affected the tested friction node more effectively, and it provided better lubrication and better anti-seize protection compared to the second-tested nitride (B). Likely, the better-developed porous structure of the particles of this compound was also important.
5. The prospect of further analyses is to conduct studies on mixtures of hexagonal boron nitride with other base oils, at other additive concentrations and in other friction nodes. It is also important to consider testing at high-temperature conditions. Such studies are currently being continued at the Military University of Technology in Warsaw.

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