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# HEXAGONAL BORON NITRIDE AS A SOLID LUBRICANT ADDITIVE (AN OVERVIEW)

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**Abstract**

An overview is given on utilizing of hexagonal boron nitride nanopowders as a solid additive to liquid lubricant materials.

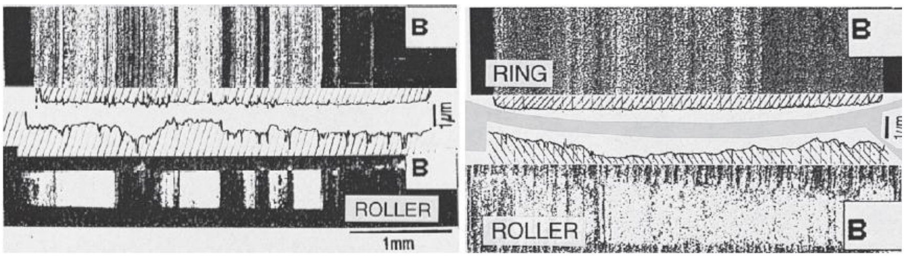
Lubricants are extensively used by industry to control friction and wear in a large variety of mechanical and tribological applications, because without lubricants most moving parts of machines and engines fail or wear out and become useless. Most lubricants come in a liquid- or grease-consistency. But, liquid lubricants alone cannot meet the increasingly more stringent application conditions under very high and low temperatures, ultra-high vacuum, radiation, extreme contact pressure, very low and high sliding speeds, etc. It is a reason, why the idea of combined uses of solid and liquid lubricants is becoming more popular.

Solid additives in current liquid lubricants containing heavy metals, carbon (graphite), sulfur and phosphorus bearing substances, etc. are environmentally harmful and cause pollutions. They can be displaced by light boron-based nano-scale powders in oils and greases providing much better tribological performance.

The most part of boron compounds are the superhard materials. But, some of them stand out as very effective solid lubricants. In particular, boron compounds with layered structures possess high potential to provide various surfaces with low friction and wear under both dry and lubricated sliding conditions [1]. Among them powdered hexagonal boron nitride, h-BN, has a capacity to significantly reduce friction and wear when mixed at low concentrations (as little as $\sim 1$ wt. %) with liquid lubricants – oils, greasers, and fuels. Previously tribological interest in h-BN was limited to lubrication for metalworking processes where lubrication at high temperatures and / or cleanliness of working environments is of critical concern. However, later it was reevaluated as a “clean” lubricant, which can be an alternative to above mentioned “dirty” ones (e.g., carbon C in graphite-structure or molybdenum disulfide $MoS_2$) in more general applications as a solid lubricant.

Hexagonal BN has a lamellar crystalline structure, in which the bonding between molecules within each layer is strong covalent, while the binding between layers is almost entirely by means of weak van der Waals forces. This structure is similar to that of graphite and $MoS_2$, which are highly successful solid lubricants too, and the mechanism behind their effective lubricating performance is understood to be owing to easy shearing along the basal plane of the hexagonal crystalline structures. BN captured attention as a potential solid lubricant for general use because of this similarity. However, a number of results showed that, as a solid lubricant, BN was inferior to graphite and $MoS_2$.

From the fundamental investigations of h-BN lubricant properties performed in [2], h-BN in general was found less effective than other solid lubricants except for high-temperature applications. However, a series of sliding experiments showed somewhat curious behavior of BN when added into lubricating oil, e.g., in the case of sliding of bearing steel versus itself. BN slightly increased the coefficient of friction, but drastically decreased wear. In sliding of steel bearing, boron is present in small cavities on wear scars (**Figure 1**). Not all boron is present there as h-BN, but in other forms too. Although boron nitride is known to have high thermal stability, the observation of the wear scars had shown coexistence of boron and oxygen at the same locations, evidence which suggests oxidation during sliding. BN introduced to the actually rubbing parts strongly adhered to steel surfaces, and works to decrease wear.




**(a)** **(b)**

**Figure 1.** Distribution of B on wear scars (a) steel ring / steel roller and (b) steel ring / cast iron roller [2].

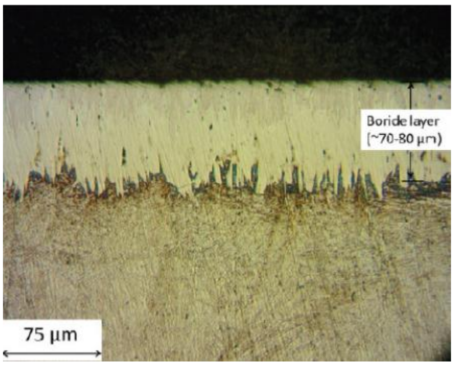



**Figure 2.** Cross section micrograph of surface-treated (electrochemically borided) steel sample [3].

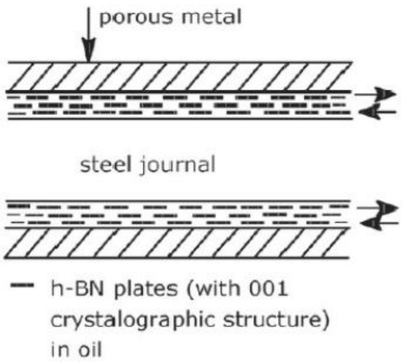



**Figure 3.** Schematic representation of the lamellar lubrication in journal bearing [5].

Different approach to modification of sliding surface by h-BN lubricant has been suggested for gears. Gears and other mechanical assemblies are some of the key components for conversion of wind energy to electrical energy in wind turbines, but their durability and efficiency are severely impaired by some tribological issues like micropitting, wear, scuffing, and spalling. To address these issues, in [3] a combinational approach was proposed to incorporate surface treatment in coordination with the use of nanocolloidal lubricant additives. In particular, boron nitride based solid lubricants were manufactured and flat gear steel samples were borided using an electrochemical boriding process. The borided surfaces (**Figure 2**) enhanced the mechanical properties of the surface layer, leading to improved wear resistance.

As for the boron nitride itself, it was observed to be stayed well dispersed within the oil and formed a stable tribofilm, which was important to achieve improved tribological performance.

It looks that h-BN particles interact with sliding surfaces and form low-friction boundary films protecting these surfaces against wear and providing low friction due to their low shear. Correspondingly, there was found a preferential orientation of h-BN nanocrystalline sheets of hexagonal boron nitride (formed from the tribochemical reaction between a borated additive and a nitrogenous compound using mineral oil containing a little amount of synthetic base as the base-oil) parallel to the sliding direction [4]. It was evidenced the formation of a lamellar solid from the tribochemical reaction of a borated additive and a succinimide additive. The result is the formation of h-BN in the tribofilm. The tribofilm was mainly composed of an amorphous borate matrix containing highly-dispersed h-BN nanoparticles in the form of sheets 10 nm wide and 5 nm thick. Thus, when present at sliding interface the atomic layers of hexagonal BN, the self-lubricated solid, align themselves parallel to the direction of sliding motion and then shear with relative ease to provide the levels of low friction.

Such orientation of nanosheets (**Figure 3**) has a favorable effect on friction – this statement was proved by the combination phospholipid molecules with h-BN. Phospholipid in vivo and hexagonal boron nitride in vitro are good examples of frictionless lubricants. Phospholipid molecules and BN have the ability to form multibilayer or layered structures similar to lamellate solid. It has been confirmed experimentally that phospholipid molecules as lamellar lubricants protect the surface of joints against wear while acting as frictionless lubricant. The experiment strongly suggested [5] that h-BN has the ability to lubricate under load with very low friction coefficient comparable to phospholipids. Relatively low surface energy and low adhesion between the crystallites are giving the additives low friction coefficient. The results of the experimental studies showed that h-BN as an additive in vaseline possesses friction reducing properties, and excellent antiwear properties.

It should be noted that hexagonal BN is a versatile ceramic material with unique properties and a wide application area in industry. Because of complex of important properties – resistance to oxidation up to high temperatures, lubricity, high corrosion resistance, high thermal conductivity and high electrical resistivity – it is used mostly as a high temperature lubricant material. However, powdered h-BN when added into plastics not only reduces friction coefficient, but also increases their thermal conductivity, decreases thermal expansion and increases use temperatures. There are number of suggestions on practical utilizing BN-based lubricants useful for metallic, ceramic, polymer, etc. interfaces.

Although, aluminum is one of the most prominent metals in the fabrication of metal matrix composites, frequently the oxidation of aluminum prevents the precisely measuring the wetting of ceramics. In [6], an improved sessile drop method was devised to prevent the oxidation of the aluminum. Using this method, the contact angle between h-BN and molten Al was measured in a purified He + $H_2$ atmosphere and in a very high vacuum at high-temperature. It was confirmed that AlN was produced at the solid / liquid interface and caused the contact angle to decrease to 0 °. AlN had good structural properties, whereas h-BN did not. Accordingly, it was suggested that h-BN particles, which have good wetting, be inserted into the Al-melt. This causes the surface of the h-BN to be converted into AlN which has good structural properties. Using this process, a metal matrix composite, which has good structural properties, should be produced. In particular, since h-BN is lubricous, a material should be produced which has high wear resistance.

Results of friction force in small-size bearings lubricated with the amount of oil contained boron nitride additives were presented in [7]. The investigated bearings were porous journal bearing and thrust bearing of the type sphere-on-plane operating with low velocity under spinning friction regime. The comparison of the results obtained for oil with boron nitride additives with one without such additives, magnetically active lubricant and oil with other than BN additives showed a significant reduction of the friction coefficient and contact temperature.

The antiwear capacity of a borate containing nitrogen was evaluated in [8]. Rubbing surface analyses revealed that there were borate and boron nitride under high load in comparison to only borate found under low load. Therefore, it can be considered that, under mild conditions, the borate ester is absorbed physically and chemically on the rubbing surface providing antiwear function at low load, and under severe conditions some of absorbed borate films degrade and form boron nitride tribochemically developing antiwear performance at high load.

In [9], the modified borate powder also was synthesized as an oil additive. The results of studying its tribological properties showed that it had good extreme pressure, antiwear and friction reduction properties. The viscosity of base-oil and the water content in the modified borate had great effect on the wear resistance with extreme pressure. It can form a tribological mixed reaction-film on friction surface. Boron mainly existed in the form of BN on friction surface, while nitrogen content was higher than sodium content.

Thus, borate esters possess friction-reducing, antiwear, and antioxidant characteristics when blended in lubricating oils. However, they are susceptible to hydrolysis. The formation of a stable five-member ring structure in the ester molecules, involving coordination of nitrogen with boron, contributes substantially to the resistance to hydrolysis of borate esters. The susceptibility of borates to hydrolysis can be reduced by introducing N,N-dialkylaminoethyl groups with alkyl radicals containing more than three carbon atoms [10]. It was revealed that the borate ester can be adsorbed on the rubbing surface, and some of the adsorbed borate film degrades and forms boron nitride. Wear tests indicated that the combination of oil-soluble metals (copper, tin, and cadmium) compounds with organoborates gives better antiwear properties than the components separately. An antiwear synergistic mechanism can be postulated, in which borates with electron-deficient boron p-orbitals catalyze the triboreduction of the metal compounds on the rubbing surfaces, producing elemental metals.

AISI–1045 steel was duplex surface modified by deposition of a Si–B–N composite film and a $MoS_2$-based film [11]. It was found that composite film was composed of h-BN and c-BN, which had much larger hardness than the steel substrate and were able to form interfacial transition layer with the steel substrate and, hence, the tribological behavior of the steel was greatly improved. Moreover, the friction and wear behavior of the Si–B–N film was further significantly improved by the introduction of the Mo.

The tribological performance of grease lubricant plus h-BN micro-particles additive was studied on steel / steel tribopair under vibrating fretting and sliding fretting conditions [12]. The grease compounded with boron nitride was found to be the best for industrial applications.

In [13], it was performed a comparative study on the tribological behavior of hexagonal boron nitride as lubricating microparticles – an additive in porous sliding bearings for a car clutch. The main effect of h-BN microparticles lamellar lubricant plus oil in comparison to a standard Mobil-lubricant appears to be that the impregnation of h-BN microparticles in Cu – Fe porous bearings persists for a long period of time and the microparticles are gradually released, being supplied to the contact surfaces. In the bench test, porous bearings have demonstrated that such impregnation will satisfy up to ~ $10^5$ h of clutch operation in a real engine.

Ni-based alloy / nano-hexagonal boron nitride (nano-h-BN) self-lubricating composite coatings were successfully produced on medium carbon steel substrate by means of Nd:YAG laser cladding [14]. A novel high energy ball milling method was adopted to clad nano-Ni onto nano-h-BN with an aim to enhance the compatibility between the h-BN and the metal matrix during laser cladding process. It was found that nano-h-BN was hardly to be composed into Ni 45 matrix coating even if increasing the weight percentage of h-BN from 5 to 10 wt. % with a conventional mechanical mixing. The high energy ball milling of nano-Ni onto nano-h-BN significantly improved the interfacial compatibility between h-BN and Ni 60 matrix. The friction coefficient of the laser clad Ni 60 / nano-Ni-clad h-BN coating was reduced obviously.

The results of the recent experimental studies have demonstrated [15] the high potential of h-BN as an additive for preventing fretting sliding, and can be very useful for application in grease-based compound lubrication of a steel surface in industrial equipment.

Cubic, amorphous and hexagonal boron nitride films were deposited onto a silicon substrate and a reciprocating tribometer was used to examine friction and wear properties for these three BN films [16]. The c-BN film showed the highest wear and peeling resistance of the tested films. The lubricating performance of the c-BN film proved significant following a long lubricating life and low friction. In contrast, the a-BN and h-BN films showed short lubricating endurance lives and large friction changes in spite of the fact that they are good, in general, as solid lubricants. These unexpected results were speculated to reflect the premature debonding of the h-BN and a-BN films during sliding and the subsequent discharge of their flakes out of the nip between the substrate and the ball indenter, owing to their lower adhesion to the substrate.

The high-temperature friction and wear characteristics of ceramic matrix composites incorporated with various solid lubricants including hexagonal BN have been investigated from room temperature up to ~ 1000 °C [17]. On the basis of general design considerations relevant to solid lubrication proposed for friction and wear data of self-lubricating composites, such optimized composites appear to be promising candidates for long-duration, extreme environment applications with low friction and small wear rate.

The results of investigation of the tribological behavior of $Si_3N_4$–h-BN composites with different hexagonal boron nitride volume fractions under distilled water lubrication showed [18] that the addition of h-BN into $Si_3N_4$ matrix significantly decreased the friction coefficient for $Si_3N_4$ against $Si_3N_4$ pair under titrimetric water lubrication. The morphological analysis and chemical characterization of the worn surfaces via scanning reveal that under titrimetric water lubrication, the h-BN in $Si_3N_4$–h-BN spalled off during the wearing tests and the spalled pits formed on the wearing surface of $Si_3N_4$–h-BN, and then the debris dropped into the pits to react with water, thus a tribochemical film formed on the wearing surface. It facilitated smoothing the surfaces of $Si_3N_4$–h-BN and $Si_3N_4$ to create a fine lubrication environment.

The ring compression tests of the interfacial friction and flow behavior of an $Al_2O_3$-based ceramic composite have indicated [19] that boron nitride lubricant can be used effectively at elevated temperature range of 1400 – 1600 °C.

It is known that the processability of polymers by extrusion is related to the interface between the polymer melt and the die wall, because the wall surface energy affects the flow of polymer melts in extrusion die. On the other hand, BN powders act as a solid lubricant lower the surface energy. It is the reason why the effects of BN powders on the rheological properties and the extrusion processability of metallocene-catalyzed low-density polyethylene were investigated [20]. Small crystal size and uniform size distribution were found to be more effective. Also, the influence of a hot-pressed BN die on the instability of capillary flow was found to be quite effective in delaying surface and sharkskin defects and postponing gross melt fracture. A synergistic effect of processability improvement could be obtained when both BN powder and die are used together.

During the die flow of metallocene polyethylenes, flow instabilities may occur. Namely, wall slip, "sharkskin", and stick-slip (pressure oscillations) and gross fracture may be obtained depending on the volume flow rate and die geometry. Fluoroelastomers and boron nitride powders with hexagonal crystal structure can be used as suitable processing aids in melt extrusion processes. Fluoroelastomers at low concentrations act as die lubricants and may eliminate flow instabilities such as surface and stick-slip melt fracture. On the other hand, specific BN powders may not only eliminate surface and stick-slip melt fracture, but also postpone gross melt fracture to higher volume flow rates. In [21], it was shown a way for quantitative differentiation of the influence of polymer processing additives on rheological behavior.

Boron nitride as a solid lubricant was tested [22] as an additive to the lubricants used in the polytetrafluoroethylene paste extrusion in order to identify enhanced processing aids. It was found that the addition of boron nitride not only increases the extrusion pressure but at the same time improves the final mechanical properties of the final extrudates. This offers possibilities for controlling these properties by controlling the degree of fibrillation, i.e., by adding a small amount of solid BN lubricant to adjust pressure, fibrillation, and thus the final mechanical properties.

Calcined petroleum coke and hexagonal boron nitride were used as the friction modifiers to improve the friction and wear properties of phenolic resin-based friction composites too [23]. It was found that the hybrid of the two friction modifiers was effective to significantly decrease the wear rate and stabilize the friction coefficient of the friction composites at various temperatures by forming a uniform lubricating and / or transferred film on the rubbing surfaces. The uniform and durable transfer films were also able to effectively diminish the direct contact between the friction composite and the cast iron counterpart and hence prevent severe wear of the latter as well. The effectiveness of the hybrid of calcined petroleum coke and h-BN in improving the friction and wear behavior of the phenolic resin-based friction modifiers could be attributed to the complementary action of the "low temperature" lubricity of calcined petroleum coke and the "high temperature" lubricity of h-BN. The optimum ratio of these two friction modifiers in the friction composites was suggested to be 1:1, and the corresponding friction composite showed the best friction-reducing and antiwear abilities.

Some of our recent works [24 – 29] are devoted to obtaining by a chemical method of nanopowdered h-BN (with disc-shaped particles; mean diameter and mean thickness, respectively, 240 and 25 nm) and utilizing.

Summarizing above brief overview on powdered h-BN lubricity, one should emphasize that in general the larger grain size and higher crystallinity lead to better lubricating properties and high temperature-stability of h-BN. However, there is a problem of sedimentation: micro-scale powders added into lubricating oil and other liquid materials may settle out unless very effective surfactants or dispersing agents are added too. In case of nano-scale particles, mainly because of their very large surface-to-volume ratio, as well as light weight (due to BN low specific gravity), these powders may stay in dispersion for a long time without the use of additional surfactants. Fortunately, boron nitride can be produced in nano-powders and, therefore, is easy to incorporate within liquid lubricants.

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