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3 authors:

Osman Nuri Celik
Eskisehir Osmangazi University

Nuran ay
Anadolu University

yapıncak Göncü
Anadolu University

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O. N. Çelik [a] , N. Ay [b] & Y. Göncü [c]
[a] Department of Mechanical Engineering , Eskisehir Osmangazi University , Eskisehir , Turkey
[b] Department of Material Science and Engineering , Anadolu University , Eskisehir , Turkey
[c] BORTEK, Boron Technologies and Mechatronics Inc. , Eskisehir , Turkey
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O. N. ÇELIK,[1] N. AY,[2] and Y. GÖNCÜ[3]

[1]*Department of Mechanical Engineering, Eskisehir Osmangazi University, Eskisehir, Turkey*
[2]*Department of Material Science and Engineering, Anadolu University, Eskisehir, Turkey*
[3]*BORTEK, Boron Technologies and Mechatronics Inc., Eskisehir, Turkey*

The aim of this study was to investigate the effects of nano hexagonal boron nitride(hBN) particles on the friction and wear properties of AISI 4140 steel material when the hBN particles are used as an oil additive. Nano hexagonal boron nitride powders, which were produced using a special process, were dispersed in engine oil (SAE10 W) to enhance lubrication. The amount of nano hexagonal boron nitride in the engine oil was varied from 0 to 10% by volume, and four different lubricant samples were prepared. Wear tests were conducted using ball-on-disc geometry. The worn surfaces of substrates were analyzed using scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS). The experiments showed that the nano hexagonal boron nitride particles that were used as an oil additive affected the friction and wear behavior. A 14.4% improvement in the friction coefficient and a 65% decrease in the wear rate were achieved through the use of the nano hBN as an oil additive.

Keywords: Friction, Lubricant, Nano hexagonal boron nitride, Nanoparticles, Wear

Introduction

Wear is one of the major causes of metal loss in industrial processes. Enhancement of lubricants has been shown to reduce the friction and wear of mechanical parts, which could save billions of dollars (Tung and McMillan 2004; de Barros' Bouchet, Martin et al. 2005).

The addition of solid lubricants into lubricating oil significantly reduces the friction coefficient and wear problems in mechanical systems (Donnet and Erdemir 2004, Lee et al. 2009). The major solid lubricants include MoS_2, graphite, boric acid, boron nitride, PTFE, diamond, soft metals, and lubricious oxides (Ladavière et al. 2003, Donnet and Erdemir 2004, Erdemir 2009, Hu et al. 2009). The tribological properties of oil additives, such as MoS_2 (Gansheimer and Holinski 1973; Ilie and Tita 2006; Prasad et al. 2011), graphite (Huang et al. 2006; Martorana et al. 2010; Prasad et al. 2011), graphene (Eswaraiah et al. 2011; Wei et al. 2011), boric acid (Erdemir 2009; Lovell et al. 2010; Vadiraj et al. 2012), nano-diamond (Wu et al. 2007; Chou and Lee 2008, 2010; Chu et al. 2010), PTFE (Rico et al. 2007), and others (Xue et al. 1997; Chen et al. 1998; Sunqing et al. 1999; Liu and Chen 2000; Rapoport et al. 2003; Hernandez Battez et al. 2006; Wu et al. 2007; Gu et al. 2008; Hernández Battez et al. 2008; Yang et al. 2009; Hernández Battez et al. 2010; Peng et al. 2010), have been reported in the literature.

Address correspondence to: O. N. Çelik, Department of Mechanical Engineering, Eskisehir Osmangazi University, 26480, Eskisehir, Turkey. E-mail: celikon@gmail.com

Hexagonal boron nitride (hBN) has unique characteristics that make it an attractive performance-enhancing alternative to inorganic solid lubricants, such as graphite and molybdenum disulfide. Hexagonal boron nitride has a lamellar structure in which van der Waals forces exist between sheets of covalently bonded boron and nitrogen atoms. The lubricating performance of hexagonal boron nitride arises from the easy shearing along the basal plane of its crystalline structure. Recently, several studies on the lubricating properties of hexagonal boron nitride have been reported. Based on these studies, hBN can be inferred to be an effective solid lubricant. However, few studies on the addition of hBN to oil have been reported. Kimura et al. (Kimura et al. 1999) noted that the addition of hexagonal boron nitride to paraffinic mineral oil reduced wear significantly. The hexagonal boron nitride particles used in their study were shaped like flat plates; they exhibited an average diameter of 2.85 μm, and the ratio between the thickness and the average diameter was approximately 0.07. Watari et al. (1999) "studied the effects of the addition to oil of turbostratic boron nitride (tBN) and/or hexagonal boron nitride with several surfactants to obtain a high-performance cutting or grinding oil. Tequi et al. (2004) suggested that MoS_2 be added to transmission oil that contained hydrated alkali metal borate and hexagonal boron nitride. Hutchinson and Reid (2007) improved a lubricant composition composed of boron nitride particles dispersed in propylene glycol or a water-soluble polyglycol base for baking ovens. Rahim and Walker (2007) invented a food-grade boron nitride-based lubricant additive. The additive is composed of boron nitride in combination with a dispersant and an oil carrier. Mosleh et al. (2009) modified the composition of fluids for sheet-metal forming fluids and added solid lubricant nanoparticles such as MoS_2, WS_2, and hBN to the fluids. Little research on the tribological properties of AISI 4140 steel with an engine oil additive that includes nano hexagonal boron nitride has been reported in the literature.

In this study, nano hexagonal boron nitride particles were produced using a special process that involves the reaction of boron oxide with ammonia and grinding operations. Different amounts of nano hBN particles were added to engine oil. The tribological properties of AISI 4140 steel with the nano hexagonal boron nitride used as an additive in engine oil were investigated using a ball-on-disc tribometer. Both the coefficient of friction and the specific wear rate were found to be significantly improved.

Experimental Procedure

Preparation of Nano Hexagonal Boron Nitride

For the synthesis of boron nitride powder, a mixture of boron oxide and different types of filling materials were reacted with high-purity ammonia as the reacting gas. The obtained raw boron nitride was in the form of agglomerates, with particle sizes that ranged from 50 to 1000 micrometers. Dry milling was performed in a ball mill to reduce the particle size of the raw boron nitride. The proper milling conditions for obtaining a minimum particle size and a maximum specific surface area were chosen. Ground raw boron nitride powder was purified with HCl solution, and turbostratic boron nitride (tBN) powder was prepared. The tBN powders were transformed to nano hBN under an argon atmosphere.

Particle size analyses of powders were conducted with a Malvern Master Sizer Hydro G2000. X-ray powder diffraction patterns were obtained on a Rigaku Rint 2000 x-ray diffractometer equipped with a CuKα radiation source ($\lambda = 1.5418$ Å); the 2θ range was 10–60°, and the scan speed was approximately 2°/min. The densities of the nano hexagonal boron nitride samples were measured using a helium pycnometer (Quantachrome Multipycnometer, Quantachrome Instruments, USA). The specific surface area (m^2/g) ($SSA = 6/\rho$ d(3.2)) was derived from the results of Lecoq et al.(1999). In this equation, ρ is the density of nano hexagonal boron nitride (g/cm^3) and d(3.2) is the surface-area weighted mean diameter. These measurements can be performed automatically within the laser diffraction system software, which provides a means of rapidly estimating the particle surface area. SEM images were recorded on a Zeiss Supra 50VP (Carl Zeiss AG, Germany).

Preparation of Engine Oil with the Nano hBN Additive

The prepared nano hexagonal boron nitride powders were dispersed in engine oil (SAE10 W). Lubricant samples were prepared using mechanical stirring and an ultrasonic dispersing probe without the use of surfactants, dispersants or chemical materials. The amount of nano hexagonal boron nitride in the engine oil was varied from 0 to 10% by volume, and four different lubricant samples were prepared. The codes of the lubricant samples were B0 for engine oil without nano hexagonal boron nitride, and B1, B2, and B3 for engine oil with increasing amounts of nano hexagonal boron nitride. The viscosity of the base oil and lubricant samples were measured with a Bohlin Gemini rheometer with a concentric cylinder geometry.

Wear Tests

Friction and wear tests were performed using a CSM ball-on-disc tribometer (Figure 1). WC 6% Co balls with a diameter of 3 mm were used as a counterpart. Discs made of AISI 4140 steel were austenitized at 860°C for 1 h, oil quenched, and subsequently, tempered at 300°C for 20 min. The mechanical properties and surface characteristics of the balls and discs are given in Table 1. Surface profiles of discs were measured using a Mitutoyo SJ-400 profilometer (Mitutoyo Corp., Japan) before and after the wear tests. Wear-test settings were established in accordance with the DIN 50324 standard. Tests were conducted at 20°C and at a relative humidity of 32%. Wear tests were performed in a 50 mL oil tank, and the substrate surface was flooded with lubricant at least 5 mm above the sliding surface. The ball-on-disc sliding speed was 2.5 cm/s, and the diameter of the wear track was 6 mm. The chosen normal load in all of the wear tests was 10 N. The calculated Hertzian contact stress was 2.93 GPa. The chosen experimental parameters simulated the extreme conditions in machine systems that create the boundary lubrication conditions. The friction coefficient and the tangential forces were continuously recorded for a sliding distance of 40 m. All wear tests were repeated 4 times, and the results were averaged. After the wear tests, the steel discs were cleaned ultrasonically in acetone and allowed to dry at ambient room temperature. The worn surfaces of the discs were examined with a scanning electron microscope (SEM, Zeiss Supra 50VP). Elemental analysis was performed using an electron microscope equipped with an energy-dispersive x-ray probe (EDS, Oxford Instruments, UK) to determine the boron and nitrogen contents of the wear debris.

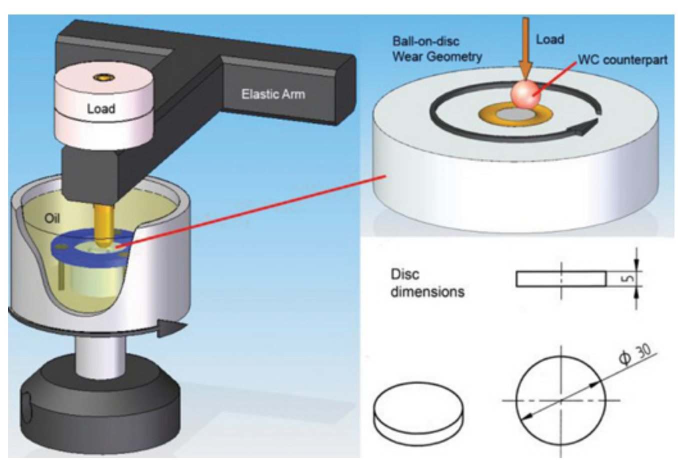

Fig. 1. A schematic wear test device, the wear geometry and the disc dimensions. (Figure available in color online.)

Table 1. Mechanical properties and surface roughness of the balls and discs

	Hardness	Elastic modulus (GPa)	Poisson ratio	Surface roughness Ra (µm)
Balls (WC 6%Co)	91.5 HRA	690	0.24	0.01
Discs (AISI 4140)	52 HRC	205	0.29	0.02

Results and Discussion

Characterization of Nano Hexagonal Boron Nitride

Nano hexagonal boron nitride powders were produced via special synthesis from raw boron nitride. The nano hexagonal boron nitride powder had a high density of 2.27 g/cm^3, a specific surface area of 14.4 m^2/g, and mean particle size of 114 nm. Figure 2 shows the particle size distribution of nano hBN used as a lubricating additive in SAE10 W oil. The x-ray difractograms of the nano hBN powder are presented in Figure 3. The sharp and well-defined peaks observed in the powder x-ray diffraction pattern correspond to hexagonal boron nitride (JCPDS file 034-0421). Then nano hBN particles exhibited thin plate-like morphologies with rounded surfaces. The diameters and thicknesses of the nano hBN particles were approximately 50–190 nm and 30–70 nm, respectively (Figure 4).

Friction and Wear Behaviors

The viscosity of lubricants affects the tribological behavior of materials. No major difference was observed between the viscosities of the lubricant samples (Table 2).Figure 5 shows the friction coefficients of the lubricants that contained different amounts of nano hBN. The steady-state values of the coefficient of friction (COF) for samples B0, B1, B2, and B3 were 0.111, 0.122, 0.095, and 0.106, respectively. When compared to the coefficient of friction value of B0, that of B1 was 10% greater, and that of B2 was 14.4% smaller. The addition of nano hBN to the engine oil resulted in differences between the samples' coefficients of friction. Several mechanisms (e.g., the ball bearing effect, a protective film, the mending effect and the polishing effect) have been invoked to explain the difference between the COF values of lubricants that include nanoparticles (Lee et al. 2009). The mending-effect mechanism is valid for engine oil that contains nano hBN particles because of the layered structure and softness of the nano hBN. The amounts of nano hBN particles in samples B2 and B3 were sufficient for the nano particles to completely cover asperities, as shown in Figure 6. The particles in the wear debris were analyzed by EDS, and the results showed evidence of the hBN plates. Therefore, the mending effect occurred, and the COF values of the B2 and B3 samples decreased. Sample B1 did not contain sufficient nano hBN particles for the same mechanism to

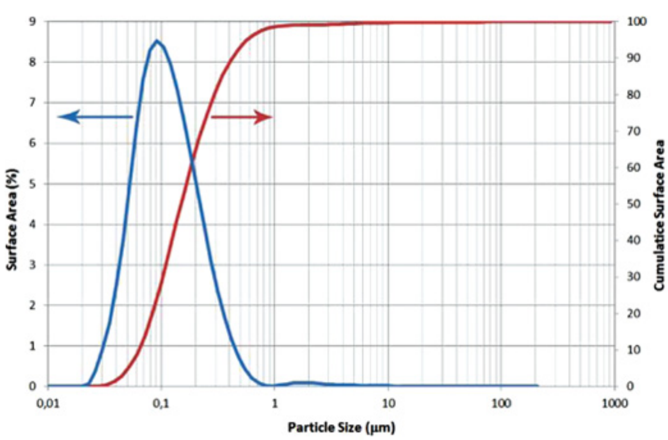

Fig. 2. Particles size distribution of the nano hBN powder. (Figure available in color online.)

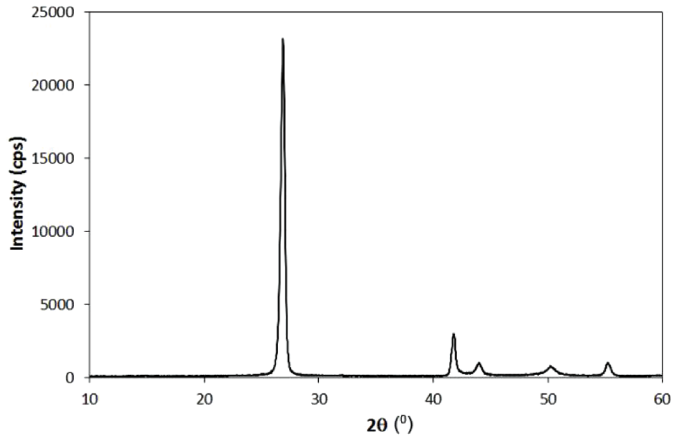

Fig. 3. XRD pattern of the nano hBN powder.

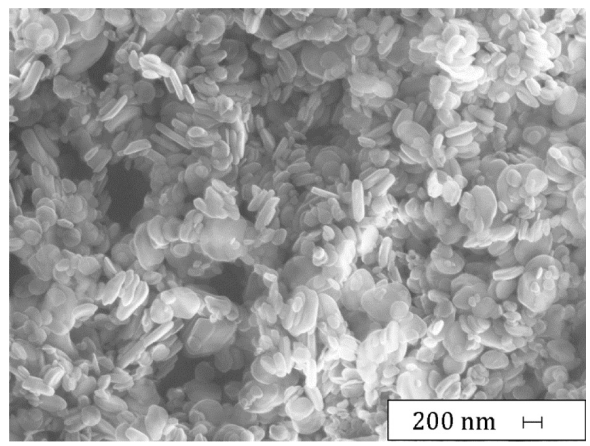

Fig. 4. SEM image of the nano hBN particles.

Table 2. Dynamic viscosity values of the lubricant samples

	Lubricant sample codes			
	B0	B1	B2	B3
Viscosity (Pa.s)	0.0900	0.0926	0.0921	0.0943

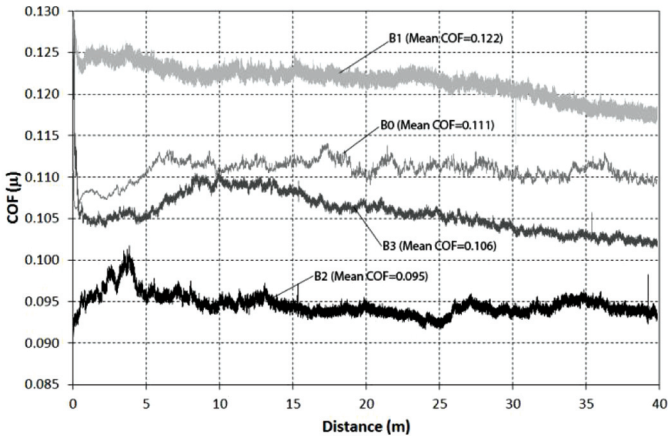

Fig. 5. Coefficients of friction as functions of the sliding distance (2.5 cm/s sliding speed).

be involved that was involved in samples B2 and B3. The lack of hBN particles in the B1 sample may lead to a decrease in the real contact surface area between asperities and, consequently, lead to the distribution of the load over a small contact area. No evidence of a linear relationship between the concentration of an additive and the coefficient of friction has been reported in the literature (Yi et al. 2008; Xu et al. 2009; Song et al. 2012).

The wear rates of B2 and B3 decreased 65% and 25%, respectively, compared to that of B0 (Figure 7), and the wear rate of B1 increased by approximately 49.1%. The major reason for the increased wear rate in B1 is probably the lack of nano hBN particles and discontinuity in the oil film (Vadiraj et al. 2012). Therefore, the real contact area between asperities becomes smaller and micro deformations arise (Cora and Koç 2009). Sample B3 contained the largest amount of nano hBN particles. Although these compact, solid, lubricant-rich films are helpful in improving wear resistance, the wear rate of sample B3 was higher than that of B2. A possible reason for this result is that nano hBN particles accumulate around

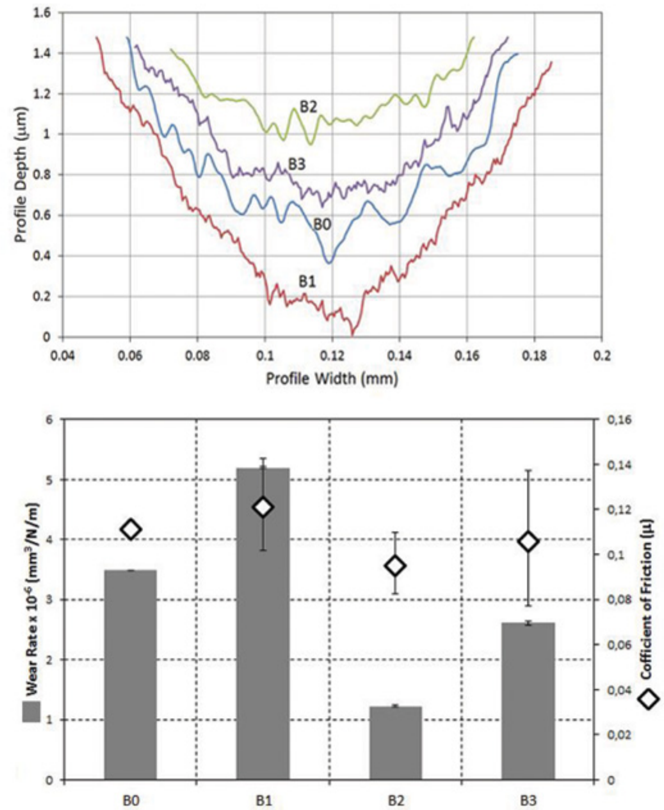

Fig. 7. Wear profiles, the specific wear rates and the coefficient of friction of samples. (Figure available in color online.)

asperities and create a barrier for the lubricant. This situation causes the formation of a partially discontinued lubrication film; hence, the wear rate increases (Burris and Sawyer 2005; Lee et al. 2009; Mosleh et al. 2009). Similar results have been previously obtained with oleic acid-modified graphene (Wei et al. 2011). In that study, although the graphene behaved as a lubricant up to a critical value, it blocked the oil film and degraded the anti-wear properties when that value was exceeded.

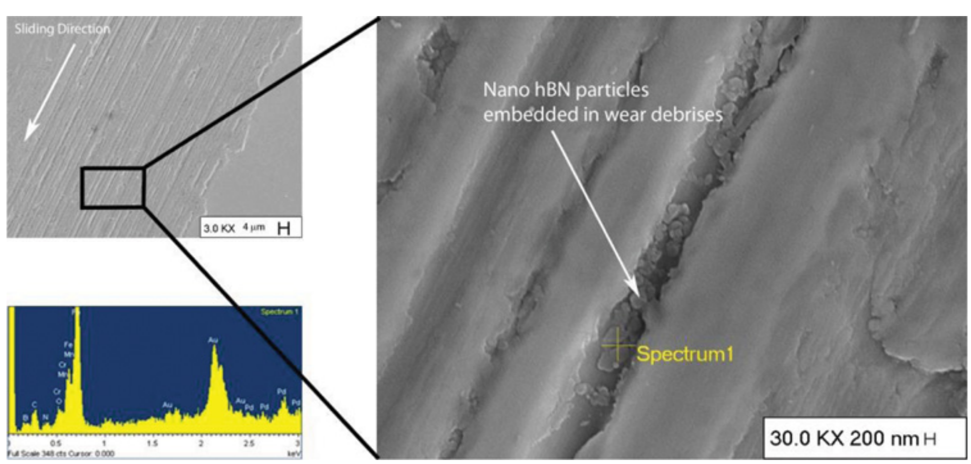

Fig. 6. The wear track, the wear debris and the chemical analysis of the wear debris for sample B2. (Figure available in color online.)

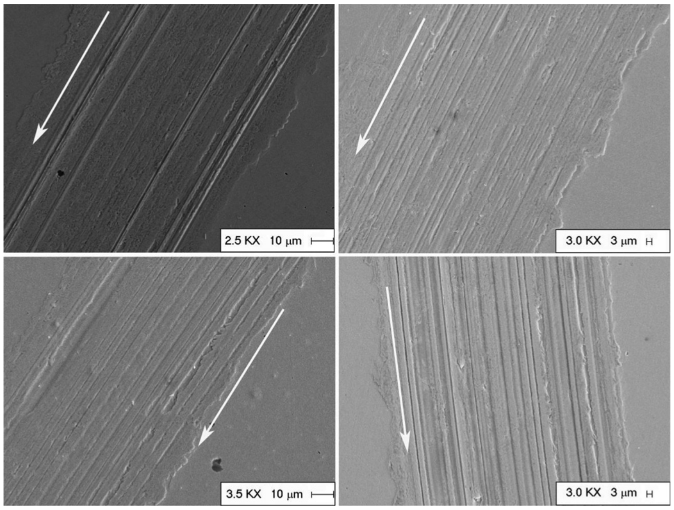

Fig. 8. SEM images of worn surfaces of discs using different lubricants: (a) B0, (b) B1, (c) B2 and (d) B3.

Table 3. Surface roughness values of discs after wear tests using different lubricants

	Ra (μm)	Rq (μm)
B0	0.049	0.069
B1	0.058	0.077
B2	**0.023**	**0.033**
B3	0.035	0.053

The effects of the lubricants (B0, B1, B2, and B3) on the discs resulted in different wear tracks (Figure 8).

The wear-track widths of the samples showed trends similar to the corresponding results for the values of the wear rates. The measured wear-track widths were 0.114, 0.136, 0.093, and 0.110 mm. The minimum wear-track width was obtained with sample B2. The same load was applied during all of the tests, and equal stress levels were assumed to have occurred in sample B0. Because the amount of solid lubricant (nano hBN) was varied in the samples, the stress levels and wear-track widths also varied.

The average surface roughness (Ra) values of the discs were measured and were found to be 0.02 μm before the tests. After the tests, both the Ra and the Rq (root mean squared roughness) values were measured. These values are given in Table 3. The lowest Rq value among the samples was 0.033 for the B2 sample. These results are compatible with the results of the wear-rate tests of sample B2.

Conclusion

In this study, the effects on friction and wear behavior of AISI 4140 steel resulting from the addition of nano hexagonal boron nitride particles to engine oil were determined. We found that the amount of nano particles in the oil affects the friction and wear properties. The addition of the nano hexagonal boron nitride particles did not change the viscosity of the lubricants. The addition of nano hBN to the engine oil created a difference in the coefficients of friction. The highest COF value was obtained with sample B1 because of its inadequate amount of nano hBN particles. The friction coefficients of samples B2 and B3 were less than those of samples B0 and B1. Compared to the friction coefficient and wear rate of the sample that contained no additive (B0), those of sample B2 decreased 14.4% and 65%, respectively. The lowest wear rate was obtained with sample B2. The specific wear rates of samples B2 and B3 were 65% and 25% lower than that of sample B0, respectively. Nanoparticles completely covered the asperities in samples B2 and B3; therefore, the mending effect occurred in these samples, and they exhibited the lowest wear rate and the wear-track widths found among the samples. The surface roughness values of sample B2 supported these results. The presence of sufficient nano hBN additives in oil prevents direct contact and results in a decrease in friction and wear. This study represents a first step toward a fundamental understanding of the friction and wear properties of nano hexagonal boron nitride.

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