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RESEARCH ARTICLE

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Tribological properties of hexagonal boron nitride nanoparticles or graphene nanoplatelets blended with an ionic liquid as additives of an ester base oil

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Abstract

Antifriction synergies between an ionic liquid (IL) and nanopowders as additives of an ester base oil were analysed at rolling conditions. For this aim, two nanodispersions based on hybrid combinations of hexagonal boron nitride nanoparticles (h-BN) or graphene nanoplatelets (GnP) with tri(butyl) ethylphosphonium diethylphosphate ionic liquid as additives in triisotridecyltrimellitate (TTM) base oil and the mixture of TTM and IL were analysed using two tribometer techniques. Film thickness characterisation and tribological tests were performed at rolling conditions [5% slide-roll-ratio (SRR)], under 50N and at operating temperatures of 30, 50 and 80°C. Stribeck curves showed that friction coefficients for the prepared lubricants are lower than those found for the TTM, being the best friction behaviour for the hybrid GnP nanolubricant. Furthermore, friction torque loss tests were performed in rolling ball bearings, observing that for the two hybrid nanolubricants lower friction torque values were obtained.

KEYWORDS

ester, friction torque, ionic liquid, nanoadditives, Stribeck curve

1 | INTRODUCTION

Nowadays, friction consumes one-fifth of all energy used worldwide and around one-third of the energy used in transports goes towards overcoming friction.^{1,2}

There are several studies which show evidence on how tribology plays a fundamental role in reducing global energy consumption through controlling friction and wear by using appropriate lubricants.^{1,3} These energy reductions help to achieve longer machine lifetimes as

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well as important greenhouse gas emissions reductions. However, new tribological methods are needed to keep up with the continuous developments in technology. The mechanical devices are increasingly smaller, but the operating conditions are also increasingly severe. Hence, new lubrication strategies that can improve the tribological performances of traditional lubricants are needed.⁴ The inclusion of nanoparticles and ionic liquids (IL) as lubricant additives might be a viable solution.^{5,6}

Several studies have shown that the use of nanoparticles as lubricants additives can efficiently reduce friction and wear in different application fields such as automotive, industrial applications or mining.⁷⁻¹¹ Carbon-based nanoparticles, such as Graphene nanoplatelets (GnP), have been analysed as antifriction and anti-wear additives of oils, showing better tribological properties in comparison to some base fluids.¹²⁻¹⁷ Likewise, hexagonal boron nitride (h-BN) based nanolubricants have also shown improved antifriction and anti-wear performances in comparison to those of base oils.¹⁸⁻²³ These good properties can be explained due to the relatively weak van der Waals interactions in the h-BN sheets.²⁴ Furthermore, h-BN is an environmentally-friendly material.²⁵

On the other hand, several ionic liquids (ILs) have also been studied as oil additives showing good tribological behaviour.²⁶⁻²⁹ Zhou et al³⁰ reviewed the miscibility of many ILs in different base oils and lubricants, showing that ILs based on phosphonium or ammonium cations have better miscibility than other ILs. The use of hybrid additives composed by nanomaterials and ionic liquids can lead to positive synergies improving the nanolubricants stability as well as the tribological efficiency.³¹⁻³³ Even though there is still very little literature about these lubricant formulations, a few studies have already shown positive synergies and consequently good tribological performances. For instance, Sanes et al³⁴ have studied the tribological behaviour of 1-octyl-3-methylimidazolium tetrafluoroborate IL combined with graphene as additives of two different oils, an isoparaffinic base oil and a fully formulated oil (SAE 10W30). Good anti-wear results were observed for the first one, although for the fully formulated nanolubricants no positive synergies were found. Hence, to better understand this type of lubricants, more studies based on IL and nanoadditives synergies are needed.

In a previous work,³¹ the synergetic effects and stability of hybrid nanoadditives containing tri(butyl)ethylphosphonium diethylphosphate ($[P_{4,4,4,2}][C_2C_2PO_4]$) IL and hexagonal boron nitride (h-BN) nanoparticles or graphene nanoplatelets (GnP) mixed with triisotridecyltrimellitate (TTM) base oil were analysed. Good stability results were achieved, finding that 3 weeks

after their preparation, none of the dispersions showed signs of instability. Regarding the tribological behaviour, positive antifriction synergies were found for all prepared dispersions, showing friction reductions of 33% for TTM/IL/GnP nanolubricant, 19% for TTM/IL/GnP nanolubricant and 14% for the TTM/IL mixture in comparison to TTM without additives for pure sliding conditions.³¹ Due to the different surface damage mechanisms, different types of surface protective additives often are used in lubricants for sliding or rolling contacts.³⁵ Therefore, the lubricating improvements provided by the hybrid IL-nanoadditives found in sliding contacts may not be translatable to rolling contacts. Besides, most of mechanical components such as gears and roller bearings experience the combination of sliding and rolling motions,³⁶ while there are very few studies that compare the tribological behaviour of nanolubricants and their role in friction over different lubrication regimes.³⁷⁻⁴¹ For instance, Hernández Battez et al.³⁷ studied the film thickness and friction properties of a mineral base oil and its mixtures with two phosphonium cation-based ionic liquids under rolling conditions, concluding that the mixtures with ILs slightly outperformed the base oil at slide-roll-ratio (SRR) of 50% and with rough disks. Kogovšek and Kalin³⁸ analysed the friction behaviour of a PAO base oil additivated with graphene nanoplatelets by performing Stribeck curves. These authors have shown that these additives could decrease the friction in the steel/steel contacts up to 44% (especially in the boundary-lubrication regime). Zin et al⁴² have studied, the tribological performance of nanolubricants composed by an engine oil and carbon nano-horns as additives, observing that lubricants which contain nano-horns exhibited the best anti-friction behaviour in all lubrication regimes. However, up to our knowledge there is only a previous research that analyzes the tribological behaviour of hybrid nanolubricants composed by nanoparticles and ionic liquids as additives of base oils.⁴³ Senatore et al⁴³ have analysed dispersions of a polyalkylene glycol base oil with 1-ethyl-3-methylimidazolium acetate and graphene oxide (GO) nanopowders as additives at two operating temperatures finding reductions in the coefficient of friction up to 17% at 298.15 K.

To have a better understanding of the behaviour of these hybrid nanolubricants, a ball on disk tribometer was used in this work to determine the film thickness of the aforementioned hybrid lubricants. Their friction behaviour was evaluated through Stribeck curves performed at a 5% SRR, under a load of 50N and at the operating temperatures of 30, 50 and 80°C. Additionally, a dedicated rolling bearing test rig was used to measure the friction torque of thrust ball bearings lubricated with each of the studied lubricants at 70°C and under an axial load of 7000N.

2 | EXPERIMENTAL SECTION

2.1 | Materials

The triisotridecyltrimellitate base oil (TTM, CAS Number: 72361-35-4) was supplied by Verkol. This oil was characterised by infrared spectroscopy (FTIR) and high performance liquid chromatography (HPLC) coupled with mass spectrometry in a previous work.³¹ The mass spectrum of this oil confirmed that the TTM base oil has a molecular weight of $757.63 \text{ g}\cdot\text{mol}^{-1}$ and a molecular formula: $\text{C}_{48}\text{H}_{84}\text{O}_6$.³¹

Hexagonal boron nitride nanoparticles (h-BN, CAS number: 10043-11-5) with an average particle size of 70 nm, a purity of 99.5% and a specific average area of $19.4 \text{ m}^2/\text{g}$ as indicated by the producer (Iolitec, GmbH, Germany, lot MNC018001), were previously characterised through FTIR, scanning electron microscope (SEM) and transmission electron microscopy (TEM).²³ These analyses showed that these nanoparticles have the typical vibration mode of h-BN at 1367 cm^{-1} and present a disk-like shaped morphology.²³ Graphene nanoplatelets (GnP, CAS number 1034343-98-0) with an average particle diameter of $15 \mu\text{m}$, a 11–15 nm thickness as well as a 99.5% purity were also provided by Iolitec. A GnP sample was previously characterised by SEM, TEM, FTIR, Raman and Energy-dispersive X-ray spectroscopy (EDX).¹⁴ These analyses indicated that: (a) the nanoplatelets are bended and wrinkled; (b) their atomic composition is about 95% carbon and 5% oxygen as well as (c) a multilayer GnP structure revealed through a high intensity Raman G-band.¹⁴

Tri(butyl)ethylphosphonium diethylphosphate ($[\text{P}_{4,4,4,2}]$ [$\text{C}_2\text{C}_2\text{PO}_4$], Cyphos 169, CAS Number: 20445-94-7) was supplied by Cytec Industries, Inc (US) with a purity of 96.3%. This ionic liquid (IL) has a kinematic viscosity of 225 cSt at 40°C and a viscosity index of 82.⁴⁴ FTIR and Raman spectra of this IL were previously reported in.³¹

2.2 | Nanolubricants preparation

A mixture and two nanodispersions were tested in this work: TTM + 2 wt% IL, and TTM + 2 wt% IL + 0.1 wt% h-BN and TTM + 2 wt% IL + 0.1 wt% GnP (from now on referred as TTM/IL, TTM/IL/h-BN and TTM/IL/GnP). The dispersions were prepared using a modified two-step method.³¹ Stability of both nanodispersions was previously analysed by photography and through refractometry techniques.³¹

2.3 | Film thickness tests

An EHD2 ball-on-disk tribometer (PCS Instruments – London, United Kingdom), equipped with optical interferometry, was used to determine the film thickness of each lubricant: the base oil, the TTM/IL mixture and the TTM/IL/h-BN and TTM/IL/GnP nanodispersions. The measurements were carried out in the contact formed between the upper specimen, a rotating glass disk (coated with a 20 nm chromium and 500 nm silica layer) and the lower specimen, a rotating steel ball of 19.05 mm of diameter (Figure 1). The optical interferometer of the tribometer provides the central film thickness through the returned wavelength of the light from the central plateau of the lubricant contact.⁴⁵ The ball specimen has a high-grade surface finish ($R_a = 20 \text{ nm}$) whereas the glass disk specimen ($R_a = 5 \text{ nm}$) can be tested up to about 0.7 GPa of maximum Hertz pressure.

The speeds of both specimens are controlled independently by two electric motors while the load-applying system is based on pressing the ball against the disk. The film thickness was measured with the ball partially immersed in the lubricant to be studied (a total volume of approximately 130 ml is used in each test), as shown in Figure 1. The tests were carried out at

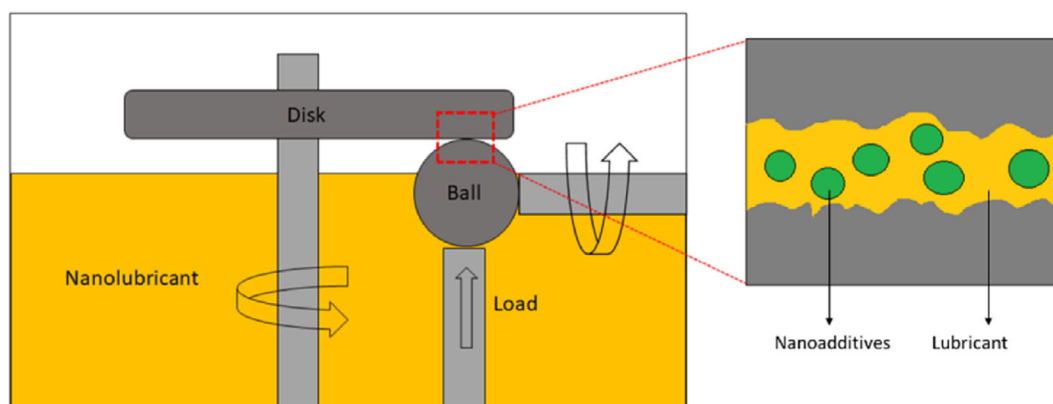


FIGURE 1 Scheme of the contact configuration [Color figure can be viewed at wileyonlinelibrary.com]

operating temperatures of 30, 50 and 80°C, a load of 50N, which corresponds to a maximum Hertz pressure of 0.66 GPa, and with 5% SRR, defined by the following expression:

$$\text{SRR}(\%) = 2 \times \frac{|U_{\text{disk}} - U_{\text{ball}}|}{(U_{\text{disk}} + U_{\text{ball}})} \times 100 \quad (1)$$

where U_{disk} and U_{ball} are the disk and ball speeds on the contacting surfaces, respectively.

Regarding the entrainment speed (U_s), it is defined by the equation:

$$U_s = \frac{(U_{\text{disk}} + U_{\text{ball}})}{2} \quad (2)$$

This entrainment speed was ramped from 0.01 to 2 m/s. The EHD2 device automatically controls disk and ball speeds. Accordingly, the lowest disk speed is 0.102 m/s and the highest disk speed is 2.049 m/s, while the lowest ball speed is 0.097 m/s and the highest ball speed is 1.950 m/s, ensuring the 5% SRR at all times. The average film thickness is obtained from three repetitions, two ramps with increasing speed and one decreasing.

2.4 | Friction tests

The previously described ball-on-disk apparatus (Figure 1) was also used to evaluate friction. In this case, the ball specimen runs against a steel disk (polished or rough) instead of the aforementioned glass disk. The steel disks and balls are made of AISI 52100 carbon steel,

62 Rockwell hardness C scale (HRC), with 100 and 19.05 mm diameters, correspondingly. The steel balls are the same as for film thickness measurements. In the steel-steel contact, the load of 50N leads to a contact pressure of 1.11 GPa. The surface roughness of the steel disks was measured with a Hommelwerke Profiler, obtaining average roughness (Ra) of approximately 50 and 500 nm for the polished and rough disks, respectively.

The friction was evaluated by performing Stribeck curves in both rough and polished disks under the same conditions as in the previously described film thickness tests: lubricant temperature of 30, 50 and 80°C, 5% SRR and an entrainment speed ramp from 0.01 to 2 m/s. The average friction coefficient values are obtained from the three measurements with 5% SRR, as in the previous case for film thickness tests.

2.5 | Rolling bearings friction torque tests

The rolling bearing tests were carried out with a four-ball machine modified by Marques et al.,⁴⁶ who replaced the original four ball configuration by a rolling bearing assembly. This new machine allows precise and accurate measurement of rolling bearings friction torque operating at constant conditions. As Figure 2 shows, this apparatus is based mainly on two different sections: the upper part (a) which is directly connected to the machine shaft and the lower one (b) where the assembly bearing system is fitted. The tests were performed on thrust ball bearings (SKF 51107), using approximately 50 ml of lubricant in each test. The temperature of the assembly is measured

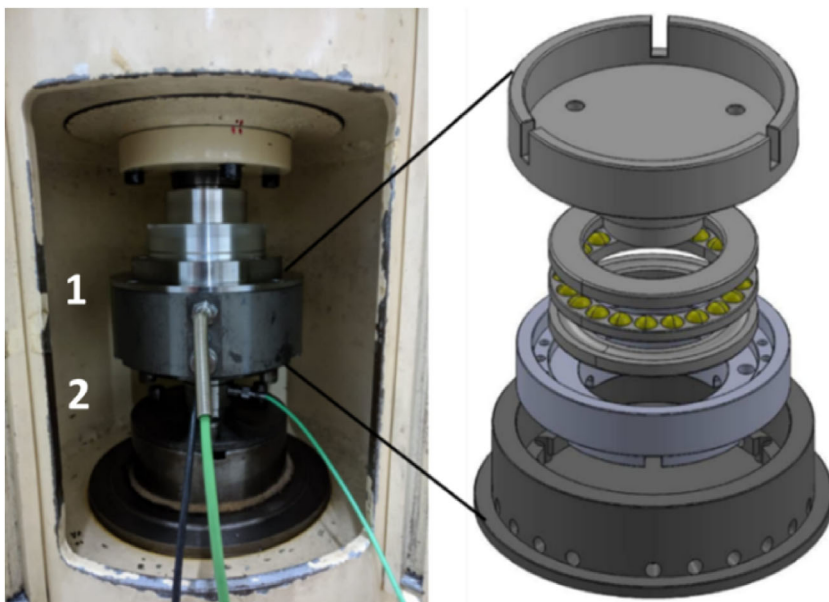


FIGURE 2 Rolling bearing assembly [Color figure can be viewed at wileyonlinelibrary.com]

with three thermocouples located at different positions to evaluate: the room temperature, the oil bath temperature and the rolling bearing raceway temperature. The oil bath temperature is controlled by a liquid cooling–heating system where a thermal fluid is carried through the centre of the driving shaft. More details about the temperature control were previously reported by Marques et al.⁴⁶ Regarding friction torque measurements, a piezoelectric torque cell Kistler 9339 was used, which ensures high-accuracy measurements.

Before each rolling bearing test, a warm up step was carried out. For this purpose, the device initially works at 500 rpm for 10 min at very low axial load (approximately 1000N) using a dead weight lever system. After that, an axial load of 7 kN is applied, producing a maximum contact pressure of around 2.3 GPa in each ball–raceway contact. The cooling–heating system was then switched on in order for the oil bath to reach the required temperature (70°C). Once the temperature was steady, four friction torque measurements were performed for each of the five speed steps (100, 200, 500, 1000 and 1500 rpm), to get better repeatability.

3 | RESULTS AND DISCUSSION

3.1 | Film thickness results

The film thickness curves are shown in Figure 3 as function of the entrainment speed, in logarithmic scale. As usual, when the entrainment speed increases, the film becomes thicker, whereas when temperature rises, viscosity

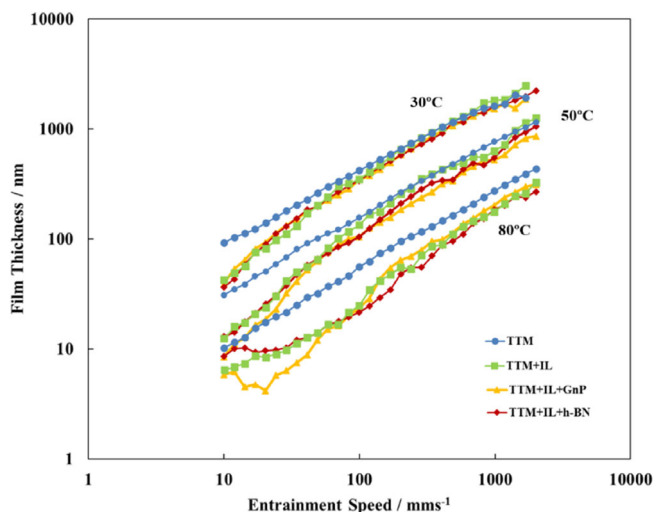


FIGURE 3 Film thickness, h_0 of the triisotridecyltrimellitate base oil and each dispersion, plotted versus the entrainment speed U_s in logarithmic scale (see online version for colours) [Color figure can be viewed at wileyonlinelibrary.com]

diminishes and consequently the film thickness values decrease.^{31,47} All the tested lubricants show similar film thickness values for all operating temperatures (30, 50 and 80°C) following their also similar viscosities, which were determined in a previous work.³¹ Nevertheless, for all the operating temperatures it is observed that TTM base oil presents slightly higher film thickness at low entrainment speeds when compared to TTM/IL mixture and both TTM hybrid nanodispersions. It is interesting to notice that the four lubricants present comparable film thickness values and should therefore, operate under the same lubrication regime.

3.2 | Friction results

The Stribeck curves obtained at 30, 50 and 80°C for all the tested lubricants are shown in Figure 4. The Stribeck curves describe the lubrication regimes according to the sliding speed and the applied load. In this work, the coefficient of friction, μ , is plotted against the specific film thickness, Λ , calculated using the following expression:

$$\Lambda = \frac{h_0}{\sigma}, \quad (3)$$

being h_0 the central film thickness described in the previous section and σ the average roughness of both specimens given by: $\sigma = \sqrt{(\sigma_{\text{disk}})^2 + (\sigma_{\text{ball}})^2}$.

To better understand the friction results it is necessary to identify the lubrication regime during the tests. The friction tests were measured with the same operating conditions (load, temperature and contact geometry) as the film thickness tests, but steel disks were used instead of a glass disk. Therefore, to plot the friction results as function of the Λ ratio, it would be necessary to know the film thickness in a steel/steel contact. However, in this work, the film thickness values measured for the glass/steel contact were used directly. The reason behind it is that, besides the surface material, all other operating conditions are the same and calculating the ratio between the Young's moduli of both materials and the weight it has on the film thickness (according to the Hamrock and Dowson's Equation), a value of around 0.94 is obtained.^{48,49} Hence, the film thickness in a steel–steel contact would be less than 6% smaller than the film thickness measured experimentally, which should not affect the analysis reported in this manuscript.

In general, the Stribeck curve can be divided in four different lubrication regimes: boundary, mixed, elastohydrodynamic and hydrodynamic or full film lubrication. Most authors consider that boundary lubrication occurs when $\Lambda < 1$, mixed lubrication when $1 < \Lambda < 3$,

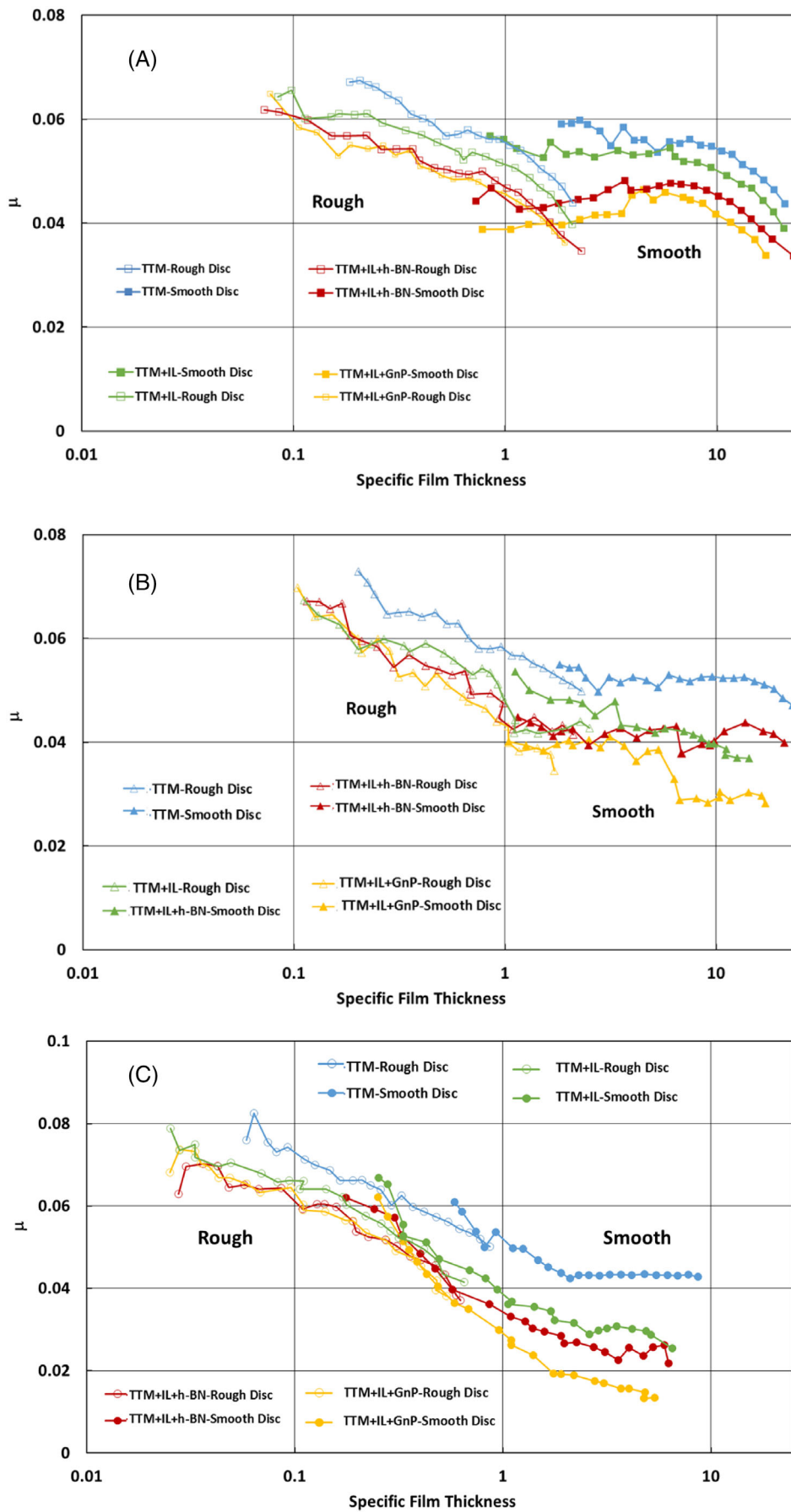


FIGURE 4 Full Stribeck curves (friction coefficient, μ , of the base oil and studied dispersions tested at 5% slide-roll-ratio for rough and polished disks at (a) 30°C, (b) 50°C and (c) 80°C (see online version for colours) [Color figure can be viewed at wileyonlinelibrary.com]

EHL when $\Lambda > 3$ and for $\Lambda > 5$, full film lubrication.^{34,50-54} However, there is controversy on the Λ range corresponding to the mixed regime. Depending on the authors considered,^{34,51-54} the mixed lubrication could span from 0.05 to 3.³⁴ In this work, the Λ ratios varied between a minimum around 0.04 (rough disk at 80 °C) to more than 20 (smooth disk at 30 °C), finding mixed conditions at $\Lambda < 1$ for all lubricants at the entire temperature range. As usual, higher friction coefficients were obtained when the rough disk is used in comparison to the tests performed with the smooth disk.

According to Figure 4, the presence of nanoparticles and/or IL as additives in TTM, enhances the anti-friction performance of the neat oil in all the studied lubrication regimes, especially in the case of hybrid additives and, of these, those containing GnP. This fact could be due to the extremely thin laminated structure of graphene nanoplatelets, which offers lower shear stress and prevent interaction at the rubbing interface.^{14,55} When graphene derivative additives enter into the frictional contact zone, they are subjected to normal load forces and the relative motion of the two contact surfaces produces shear forces.⁵⁶ The layered graphene is sheared and can be effectively involved in lubrication.^{56,57} In addition, the planar shape of GnPs confers them a lower possibility of indenting and deforming the asperities of shearing surfaces, under the same conditions, than other NPs.⁵⁸ Therefore, taking into account these considerations and the previous Raman and roughness analysis at sliding conditions,³¹ it can be assumed that the major tribological mechanisms are the formation of protective tribofilms due to both IL and nanoparticles, and the mending/patching effect. Kogovsek and Kalin³⁸ found, using SEM and optical micrographs for nanodispersions of PAO10 with GnP (concentrations from 0.5 to 5%) at similar tribological conditions, great friction reductions due to the physical effects of the patchy tribofilm formed at the surfaces.

The tribological behaviour obtained by these hybrid additives is very good, since generally at similar viscosity values of base oil and nanolubricants, the friction behaviour is quite similar or at most slightly improved,^{37,41} nonetheless an important friction reduction was achieved in this work for all designed lubricants. In addition, no measurable wear was observed at rolling-sliding conditions. The anti-friction enhancement shown by the hybrid lubricants is even more accentuated as the temperature increases and the lubrication regime shifts towards boundary lubrication. In the EHL regime the surface roughness is lower than the generated hydrodynamic film and hence, the probability of metal-metal contact is very low. Since all lubricants show similar film thickness values (Figure 3), a TTM based lubricant could be enhanced by the studied nanodispersions leading to

an improved tribological performance. These results are in great agreement with the tribological tests previously carried out for these same lubricants at sliding conditions.³¹ The highest friction reduction was also found for the nanolubricant TTM + 2 wt% IL + 0.1 wt% GnP with a 33% friction reduction, following by the TTM + 2 wt% IL + 0.1 wt% h-BN nanolubricant with a 19%. Higher friction reduction in boundary regime (up to 44% with 5% of GnP) than in EHL regime (no friction reduction) was also found by Kogovsek and Kalin³⁸ for PAO10/GnP nanodispersions. These researchers concluded that when the contact velocity decreases a larger quantity of the tribofilm is present in the contact, indicating that more GnP is trapped in the decreasing space between the surfaces. In conclusion, in this work good tribological results were obtained mixing an ionic liquid and GnP nanoparticles as oil additives, managing to expand the previous studies that existed before.

3.3 | Friction torque results

Machinery power losses are meaningfully affected by the friction torque produced inside rolling bearings. The friction torque results of rolling ball bearings lubricated with TTM base oil, TTM/IL mixture, as well as TTM/IL/h-BN and TTM/IL/GnP nanolubricants under an axial load of 7000N and an operating temperature of 70°C are shown in Figure 5.

To determine the lubrication regime of these tests, the central film thickness (h_0) at the operating temperature (70°C) was predicted with Hamrock and Dowson's Equation.⁴⁸ Given that the average roughness of the

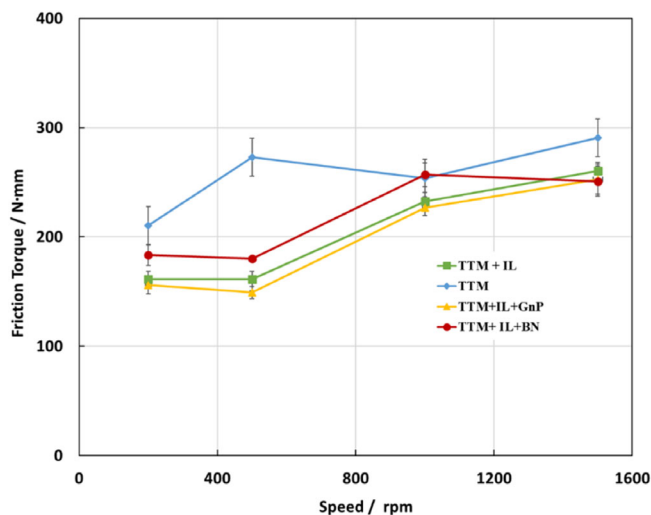


FIGURE 5 Friction torque loss results at 70°C for the rolling bearing lubricated with all designed lubricants (see online version for colours) [Color figure can be viewed at wileyonlinelibrary.com]

thrust ball bearings raceways is approximately 0.14 μm , the lambda ratio of these tests is very low ($\Lambda \approx 0.06$) and therefore, they should be operating in boundary film lubrication.

The friction torque values are lower for all the designed lubricants than those obtained with TTM without additives, being the best friction torque behaviour the obtained for the TTM + 2 wt% IL + 0.1 wt% GnP nanolubricant. This improvement in friction torque is very pronounced, especially at low speeds (200 and 500 rpm). The reason behind this behaviour should be related to the fact that the nanoparticles are expected to exert their role mainly at boundary conditions when the generated film is smaller than the composite surface roughness. As the speed increases (1000 and 1500 rpm), the friction performance of designed lubricants continues to be better than that obtained with the TTM without additives, although to a lesser extent than at low speeds. Therefore, the maximum friction torque reductions in comparison with TTM base oil were found for the TTM + 2 wt% IL + 0.1 wt% GnP nanolubricant, corresponding to a reduction of 26% at 200 rpm, 45% at 500 rpm, 11% at 1000 rpm and 14% at 1500 rpm. The fact that the mixture TTM/IL showed similar reduction to TTM + 2 wt% IL + 0.1 wt% GnP nanolubricant might indicate that the addition of IL by itself might provide the most important enhancement although the exact mechanism behind this behaviour is unknown.

4 | CONCLUSIONS

In this work, the following features were achieved:

- The experimental film thickness values of all tested TTM-based lubricants at full film lubrication are similar and in agreement with their also similar viscosities. Although, at low entrainment speeds, TTM film thickness values are slightly thicker than those obtained for the designed lubricants.
- For all studied lubricants, full Stribeck curves were obtained. Friction coefficients obtained for all designed lubricants are quite lower than those found for the TTM without additives at all operating temperatures (30, 50 and 80°C) and 5% SRR. The best friction behaviour in the studied temperature range was obtained with the TTM + 2 wt% IL + 0.1 wt% GnP nanolubricant.
- All the hybrid lubricants lead to a lower rolling bearing friction torque in comparison with the TTM base oil. This improvement in friction torque is very pronounced, especially at low speeds (200 and 500 rpm). The best friction torque reductions in comparison with TTM base oil were found for the TTM + 2 wt% IL

+ 0.1 wt% GnP nanolubricant with a maximum reduction of 45% at 500 rpm.

- Positive antifriction synergies were found between the ionic liquid and h-BN and GnP nanopowders.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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