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Tribological properties of graphene nanoplatelets or boron nitride nanoparticles as additives of a polyalphaolefin base oil

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ABSTRACT

In this work, antifriction and antiwear capabilities of hexagonal boron nitride nanoparticles (h-BN) or graphene nanoplatelets (GnP) as additives of a polyalphaolefin neat oil (PAO 40) were studied at pure sliding conditions. For this purpose, eight PAO 40 nanodispersions were prepared: four nanodispersions with h-BN and four others based on GnP. The mass concentrations of these dispersions are 0.25, 0.50, 0.75 and 1.00 wt% of h-BN and 0.05, 0.10. 0.25 and 0.50 wt% of GnP, having all of them a good stability against sedimentation (at least 96 h). Tribological assays were carried with prepared nanolubricants as well as with PAO 40 base oil at 20 N load. All nanolubricants based on h-BN or GnP showed lower friction coefficients in comparison to the non-additivated neat oil, with a maximum decrease in friction of 21% for the 0.50 wt% GnP nanodispersion. Regarding the produced wear, all disks lubricated with nanolubricants showed lower wear than those lubricated using PAO 40. The greatest wear reduction in wear track width (22%) was also achieved for the 0.50 wt% in GnP nanolubricant. Moreover, through the confocal Raman microscopy and roughness analyses of worn disks it can be concluded that the wear reductions are due to the surface repairing and tribofilm formation mechanisms.

1. Introduction

Nowadays, almost a quarter of the total energy consumed worldwide is due to friction and wear that occurs between tribological contacts [1]. Given this perspective, it is necessary to develop more efficient lubricants so as to minimize these energy losses. These reductions can also lead to longer machine lifetimes and a decrease in greenhouse gas emissions. A lubricant is a multicomponent mixture of different lubricant bases and additives in a ratio around 90% base oil and 10% additives. For this reason, many researchers have considered different technologies for discovering novel procedures to replace traditional environmental harmful additives that origin adverse emissions and include sulfur or phosphorous (for instance zinc dialkyldithiophosphate) without compromising on friendly environmental additives like nanoparticles or even ionic liquids [2–5]. Therefore, the utilization of nanoparticles as lubricant additives is a possible solution of these problems, owing to their outstanding chemical and physical characteristics [6]. Furthermore, suitable nanoparticles as additives are less chemically reactive than traditional additives because their films are produced mechanically, therefore they will react less with other additives and consequently be more durable [7]. In truth, low quantities of nanoparticles as lubricant additives can improve the tribological performance, since nanoparticles present better tribological properties that traditional solid lubricant additives, owing to they might insert in the tribological contact region and enhance the behavior of tribofilm [8,9]. This tribological improvement is due to the nanoadditives through different lubrication mechanisms that can be summarized in five types: tribofilm formation, transformation of microstructure, rolling bearing effect, synergistic effect as well as surface repairing effect. In the first mechanism, due to the big specific surface nanoparticles area, a protective film can be produced on the contact surfaces by chemical reaction or physical interactions, avoiding the direct contact metal-metal. As regards the transformation of structure mechanism, the initial microstructure of some nanoparticles is changed due to the high-level pressure and the heat generated in the friction procedure. These microstructure changes may result in the variation of tribological behavior. Rolling bearing effect appears when spherical-shaped nanoparticles roll between friction surfaces asperities and transforms sliding to rolling friction. Furthermore, the synergetic effect occurs when nanoparticles cooperate with other additives to achieve a better tribological performance. Regarding the surface repairing mechanism, due to their nature, nanoparticles can repair the contact surface imperfections reducing the surface roughness and enhancing the tribological performance [10].

Many studies confirmed that the use of nanoparticles in lubricants has important effects on the friction and wear enhancement [1,4,11–13]. Nowadays, there are several types of nanoparticles, which are mainly classified into several categories attending to their chemical structure [4]: carbon-based materials, metals, metal oxides, among others.

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Graphene nanoplatelets (GnP) are carbon-based nanomaterials, which have been analyzed as lubricant additives, observing that can improve both friction and wear behaviors in comparison to several oils without additives [14–19]. In fact, researchers have proven that little quantities of 0.02–0.5 wt% of GnP have enhanced both friction and wear lubricant properties [20,21]. For instance, Omrani et al. [14] obtained friction and wear reductions of 26% and 83%, respectively, using canola oil with an ideal mass percentage of 0.07 wt%, whereas Zhang et al. [15] observed for a synthetic oil additivated with GnP a maximum friction reduction of 17% for 0.02% GnP concentration, whereas the best GnP concentration for wear reduction (14%) is 0.06%., respectively.

Nanolubricants that contain hexagonal boron nitride (h-BN) have also led to improved antifriction and anti-wear performances in comparison to those of several base oils [22–27]. This may be due to the quite soft van der Waals forces between loosely packed h-BN layers, resulting in exceptional lubrication properties [28]. Several studies confirm these good tribological properties, for instance Çelik et al. [29] observed that a SAE10W engine oil additivated with h-BN nanoparticles improve both the friction and the wear properties in comparison to neat oil, with reductions of 14% and 65%, respectively. Furthermore, Wan et al. [30] concluded that nanolubricants formed by a 15 W-40 oil with h-BN nanopowders considerably enhanced the antifriction performance of neat oil with friction reductions up to 77%. Besides, h-BN nanomaterials are very interesting because of they are considered an environmentally friendly material [24].

In brief, as can be concluded from the aforementioned studies, GnP and h-BN nanomaterials have potential tribological properties as oil base additives. Nonetheless, there is still not research comparing the addition of two different types of nanoparticles to polyalphaolefin (PAO) base oils in the literature. PAO oils are very important in lubrication, since they are the most widespread synthetic lubricants and they are generally used in automotive applications (crankcase, transmissions, gears) and other industrial applications (refrigeration compressors, turbines, gearboxes as well as hydraulic and metal working oils) [31]. Therefore, the goal of this research is analyzing the tribological behavior of two different nanomaterials: h-BN nanoparticles or graphene nanoplatelets as additives of PAO 40 base oil at pure sliding conditions, also observing the influence of concentration of both nanoparticles, obtaining the optimum concentration with the best tribological performance.

2. Material and methods

2.1. Base oil and nanoadditives

Polyalphaolefin PAO 40, supplied by REPSOL, is synthetized through 1-decene polymerization followed by a hydrogenation. PAO 40 sample has a density and dynamic viscosity at 313.15 K of 0.8346 $g \cdot cm^{-3}$ and 335.57 mPa s, respectively, as well as a viscosity index of 149.3. An aliquot of this PAO 40 was previously characterized by infrared spectroscopy (FTIR) with no evidence of the presence of carbon-carbon double bonds [32].

Graphene nanoplatelets (GnP, CAS number 1034343–98-0) with a 99.5% purity, an average particle size of 15 μm and 11–15 nm thickness were supplied by Iolitec. A sample of this nanomaterial was previously characterized using scanning electron microscopy (SEM), Transmission Electron Microscopy (TEM), FTIR, Raman as well as X-ray spectroscopy (EDX) [20]. The characterization showed that GnPs have a bended and wrinkled appearance with a multilayer arrangement [20]. Furthermore, hexagonal boron nitride nanoparticles (h-BN with CAS Number: 10043–11-5) with a 99.5% purity and an average size around 70 nm (Iolitec, GmbH, Germany, lot MNC018001), were previously characterized by FTIR, SEM and TEM [33]. These analyses proved that the nanoparticles have a disk-like shaped morphology and the typical h-BN vibration mode around 1367 cm^{-1} [33].

2.2. Nanolubricants preparation

Nanolubricants were formulated with different mass percentages of GnP (0.05, 0.10, 0.25 and 0.50 wt%) and h-BN (0.25, 0.50, 0.75 and 1.00 wt%) in PAO 40. Both concentration ranges were selected based on previous studies [20,33], in which the optimal concentrations of both nanomaterials were determined. In this work, a two-step method was employed to prepare the aforementioned nanodispersions. For this aim, a Sartorius MC 210P microbalance ($\pm$0.00001 g) was utilized to achieve the mass concentration of nanoparticles in the oil. As regards nanodispersions homogenization, an ultrasonic bath (FB11203 Fisherbrand), with a continuous sonication time of 4 h and a power of 180 W at a 37 kHz frequency was utilized. During the sonication process the temperature is controlled to avoid overheating of samples.

Stability of the nanodispersions was analyzed by both sediment photograph capturing of samples and measurement of the evolution of the refractive index over time through a Refractometer Mettler Toledo. More details about this last technique were described in a previous article [34].

2.3. Tribological assays

Friction tests with rotational configuration were carried out using a CSM Standard tribological device for the designed PAO 40 nanolubricants as well as for the base oil, operating in ball-on-disk at pure sliding conditions. The following settings were utilized: room temperature (~23 °C), load of 20 N (2.0 GPa of maximum contact pressure), 3 mm radius, 340 m sliding distance and 0.10 $m\ s^{-1}$ speed. The tribological specimens were stainless-steel disks (AISI 52100/535A99, 5 mm radius, surface finish: Ra $<$ 0.02 μm and hardness: Hv30 190–210) and chrome steel balls (AISI 52100/535A99, 3 mm radius, hardness: 58–66 Rockwell Scale and roughness $<$0.05 μm). Both steel balls and disks were washed previously tribological tests through an ultrasonic bath of acetone so as to remove any element that could disturb our experiments and afterwards dried with hot air. After that, the disks were lubricated with each prepared lubricant (around 0.2 mL). To ensure a good repeatability not less than three replicates were carried out.

Once the friction tests have been carried out, before analyzing the produced wear, worn disks were cleansed in the acetone ultrasonic bath. With the aim of quantifying the wear produced on the disks an Optical 3D Profiler Sensofar S Neox (confocal mode, 10×) was utilized. Thus, wear was analyzed through the following parameters: wear track depth (WTD), wear track width (WTW) as well as cross-sectional area. For this purpose, these wear parameters were measured at three different zones of the worn surfaces in order to achieve representative values.

Worn surfaces roughness (Ra) of disks lubricated using the prepared nanolubricants and PAO 40 base oil were also evaluated with the 3D Optical Profiler to describe the anti-wear capability of the nanofluids. For this aim, ISO4287 standard (International Organization for Standardization, Vernier, Switzerland) was employed using a Gaussian filter (0.08 mm wavelength cut-off). Firstly, an area inside the scanned worn scar is extracted and afterwards in this selected area it is taken a perpendicular profile to the sliding direction of tribological tests. The software permits to determine the Ra value of this profile. Moreover, to achieve information of the tribofilm composition on the worn track, a WITec alpha300R+ confocal Raman microscope was utilized.

3. Results and discussion

3.1. Nanodispersions stability

Nanolubricants stability was evaluated through sediment photograph capturing. This technique consists of examining the sedimentation of nanoparticles over time. Fig. 1 shows there is no sedimentation for the more and less concentrated h-BN and GnP nanolubricants before first 96 h after formulation of nanolubricant. This stability time is bigger than that required to carry out the friction tests (around 4 h per lubricant).

Additionally, to evaluate the nanoparticles stability in PAO 40 oil, refractometry method was also employed. Thus, nanolubricants refractive index was evaluated every hour until 50 h, studying its progress (Fig. 2) for both 0.05 wt% h-BN and GnP nanolubricants. Guimarey et al. [35] have also analyzed the nanolubricants stability with refractometry, in this case ZrO_2 nanoadditives in various base oils. In the aforementioned work, the refractive index in two base oils showed a 0.4% increase after 100 h of analysis. Previously [32], we have evaluated the graphene oxide (GO) and reduced graphene oxide (rGO) refractive index of PAO 40 and ester based nanolubricants. This research evidenced that after 50 h for both base oils the refractive index increased around 0.1% and 0.4% for

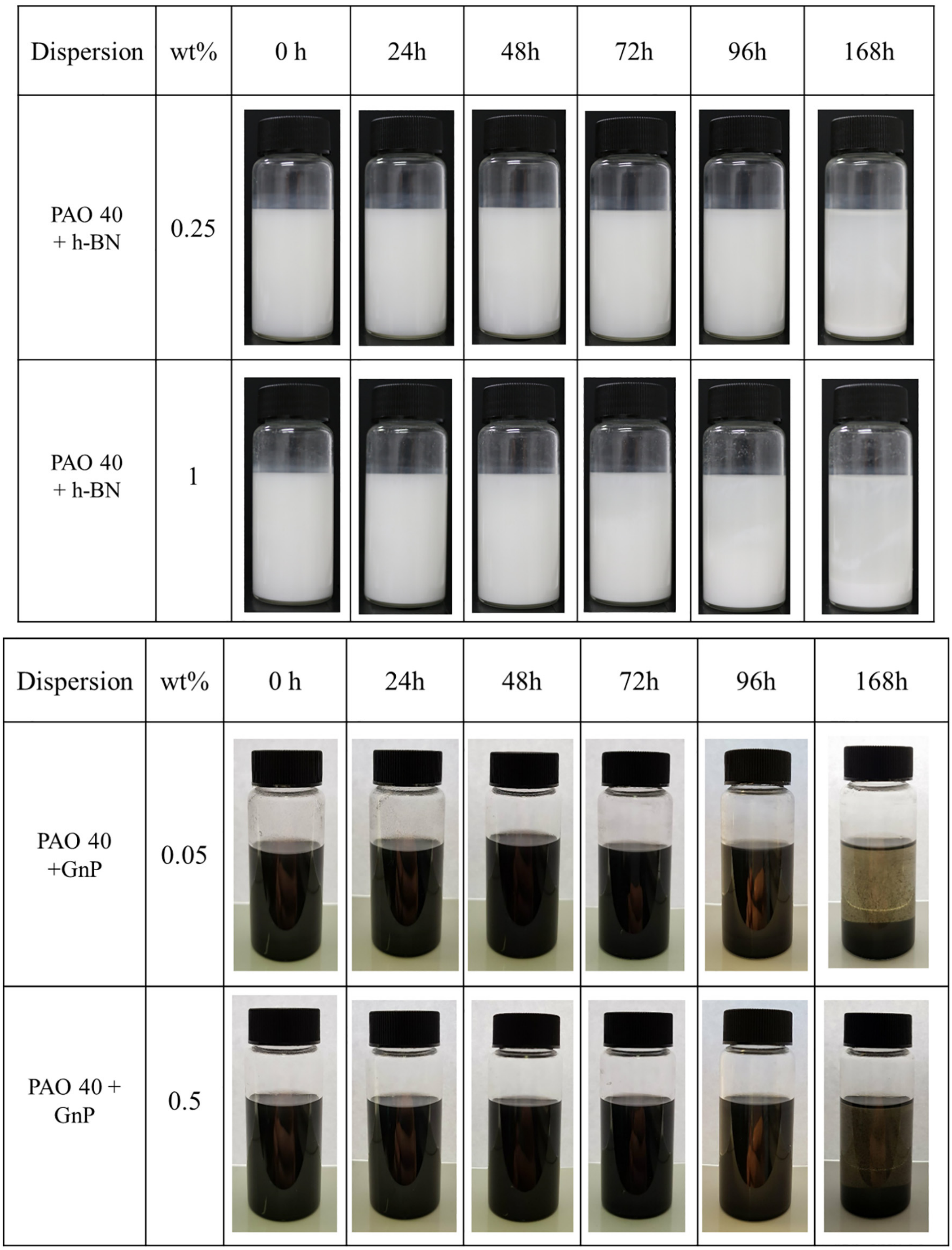

Fig. 1. Stability visual observation of PAO 40 nanolubricants based on h-BN and GnP.

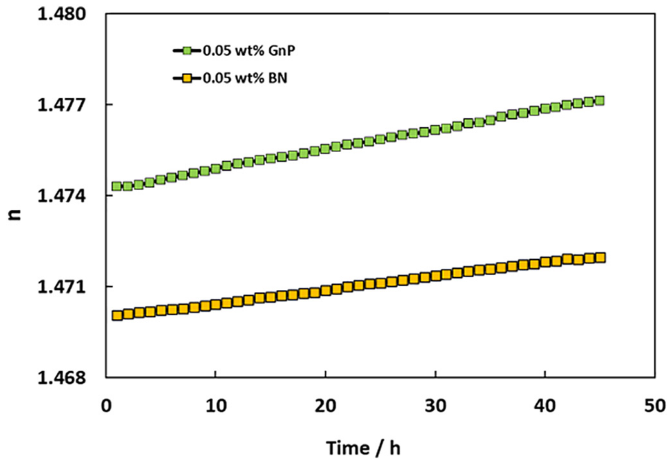

Fig. 2. Evolution of PAO 40 nanolubricants refractive index, n, composed by 0.05 wt% in h-BN or GnP.

rGO and GO nanolubricants, respectively. In our study, the refractive index evolution over 50 h indicates growths about 0.1 and 0.2% for both 0.05 wt% h-BN and GnP nanolubricants, respectively. These results demonstrate a quite good stability for the new formulated nanolubricants.

3.2. Tribological results

Fig. 3 and Table 1 show the friction coefficients (μ) mean values of the designed PAO 40 lubricants, observing that the coefficients of friction found for all the nanolubricants are smaller than that achieved for the non-additivated PAO 40. The best friction performance was achieved for the 0.50 wt% GnP nanolubricant. This GnP nanolubricant showed a friction coefficient of 0.064 against the 0.081 achieved for PAO 40 (Table 1), which leads to a 21% maximum friction reduction. Moreover, for h-BN nanolubricants the best antifriction behavior was reached for the nanolubricant of 0.75 wt% (20% reduction).

Regarding the produced wear, 3D and cross-sectional profiles of wear scars created in disks after the friction tests are shown in Figs. 4. The values obtained for WTW, WTD and the cross-sectional area of the wear scar on the disks are shown in Table 1. It can be observed that with all the nanolubricants (based on GnP and h-BN) the wear is smaller than for the PAO 40 in WTW and cross-sectional area. For such parameters, the maximum reductions were found with the 0.50 wt% GnP nanolubricant, being 22% in WTW and 19% in cross-sectional area. As regards h-BN nanolubricants the best anti-wear performance was obtained with the 0.75 wt% h-BN nanolubricant with reductions for these last parameters of 20 and 13%, respectively. These findings prove a suitable correlation among friction and wear performances.

Roughness (*Ra*) of worn disks surfaces were also studied to analyze the anti-wear nanolubricants capacity. Table 2 indicates that worn scars lubricated with h-BN or GnP nanolubricants have lower roughness than that with PAO 40. Specifically, a Ra of 181 nm was found for the lubricated PAO 40 worn surface while for the scar tested with the 0.25 wt% GnP nanolubricant the smallest Ra value was achieved (111 nm) i.e. a 39% roughness reduction. Concerning h-BN nanolubricants, the lowest Ra (131 nm) was obtained with 0.75 wt% h-BN.

Raman spectra and elemental mapping of the worn tracks lubricated with nanolubricants based on PAO 40 and h-BN or GnP with the best tribological behavior for each nanoadditive were recorded with a Raman confocal microscope (532 nm wavelength) in order to identify the role that nanoadditives has in wear parameters decrease. PAO 40 Raman spectrum is displayed in Fig. S1 observing that characteristic peaks of the oil coincide, as expected, with several of those found in the worn tracks lubricated with nanolubricants (Fig. 5). Furthermore,

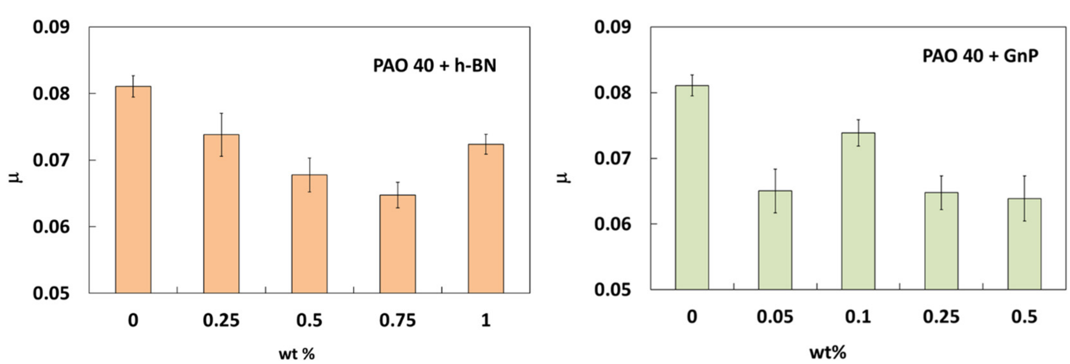

Fig. 3. Mean friction coefficients, μ, obtained for all the designed lubricants and for the PAO 40 base oil.

Table 1
Mean friction coefficients, μ, and mean worn track parameters: depth, WTD, width, WTW as well as cross-sectional area and their standard deviations for all studied lubricants.

Lubricant	μ	σ	WTW/μm	σ/μm	WTD/μm	σ/μm	Area/ 10^3μm^2	σ/ 10^3μm^2
PAO40	0.081	0.002	442	16	6.04	0.33	2.14	0.12
+ 0.05 wt% GnP	0.065	0.002	383	14	5.98	0.27	1.95	0.16
+ 0.10 wt% GnP	0.074	0.003	402	12	6.14	0.24	2.12	0.10
+ 0.25 wt% GnP	0.065	0.002	369	11	5.88	0.26	1.88	0.14
+ 0.50 wt% GnP	0.064	0.003	345	10	5.95	0.37	1.74	0.11
+ 0.25 wt% h-BN	0.074	0.004	401	17	6.14	0.32	2.11	0.17
+ 0.50 wt% h-BN	0.068	0.003	381	13	6.02	0.43	1.98	0.13
+ 0.75 wt% h-BN	0.065	0.002	353	12	5.78	0.23	1.87	0.10
+ 1.00 wt% h-BN	0.072	0.002	416	13	5.75	0.26	2.03	0.22

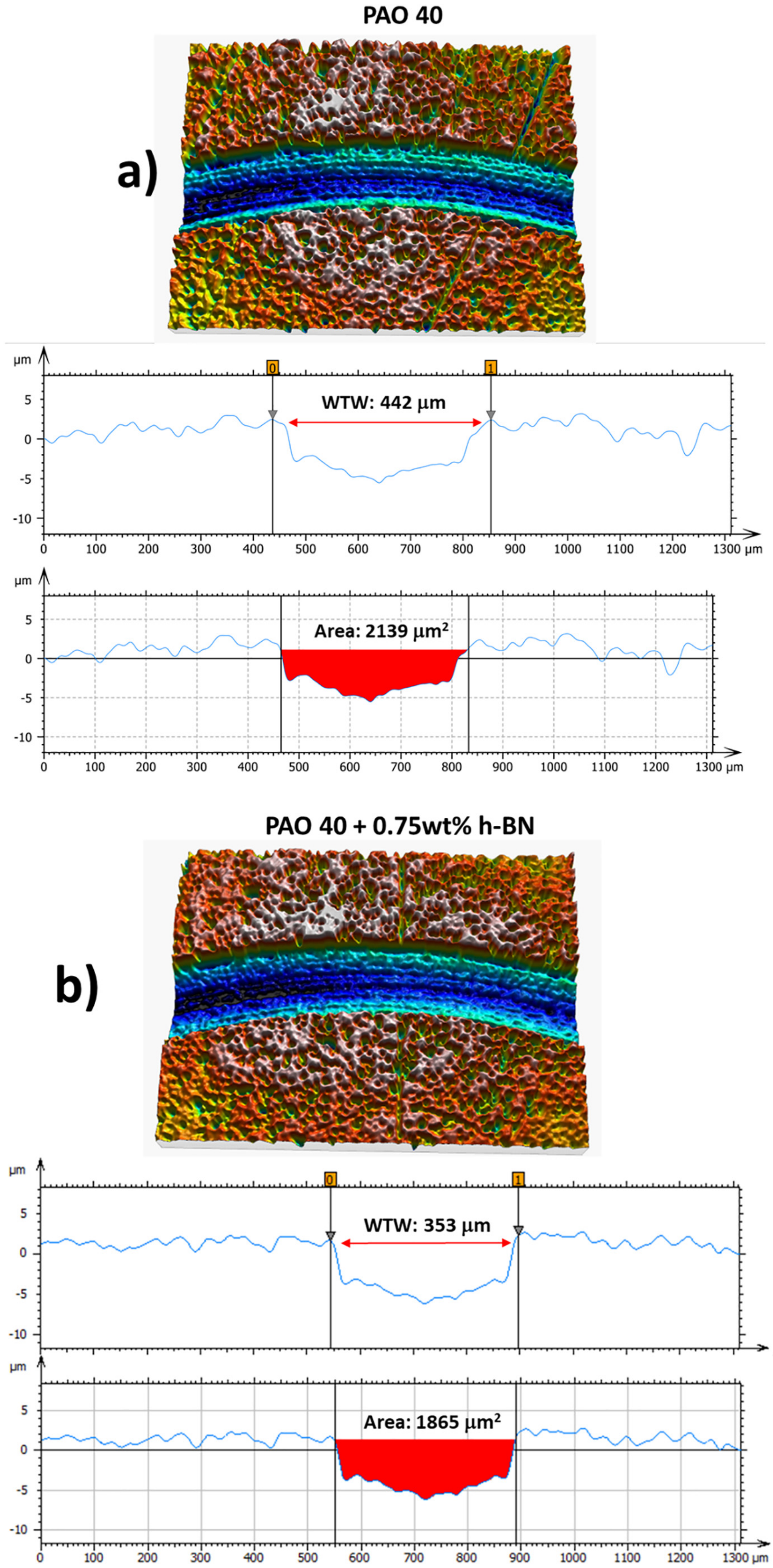

Fig. 4. 3D profile (10×) and areas of the wear tracks on the disks lubricated with a) PAO 40, b) PAO 40 + 0.75 wt% h-BN and c) PAO 40 + 0.50 wt% GnP.

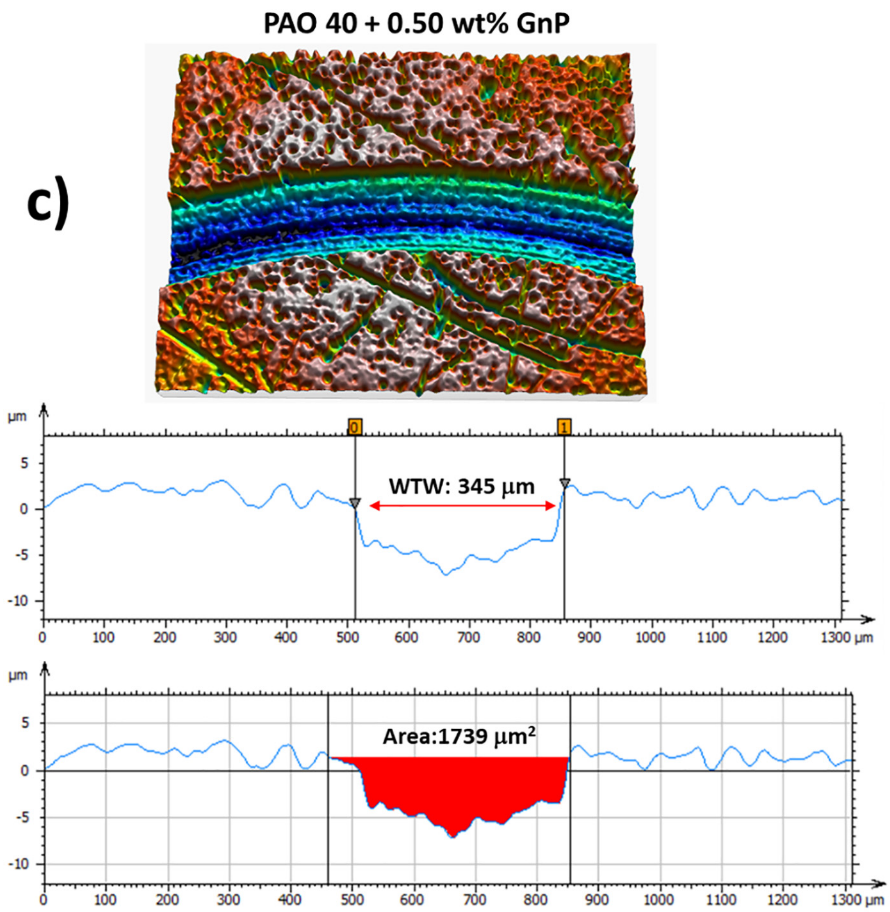

Fig. 4 (*continued*).

GnP nanoplatelets Raman spectrum (Fig. S2) displays three distinctive bands: D-band over 1350 cm^{-1}, G-band about 1580 cm^{-1} and 2D band around 2690 cm^{-1} [20]. h-BN Raman spectrum (Fig. S3) shows the usual band around 1370 cm^{-1}, similar to graphene G band [20]. Fig. 5 shows a significant presence of PAO 40 (green color) in the worn scar mapping lubricated with GnP or h-BN nanolubricants. Fig. 5a evidences the presence of spots due to graphene nanoplatelets (red color) in the worn track lubricated with PAO 40 + GnP nanodispersions, whereas Fig. 5b, corresponding to the plate lubricated with the nanolubricant PAO 40 + h-BN illustrates the boron nitride position (red color) in the worn track. Both Raman spectra (h-BN and GnP) obtained in the mapping agree with GnP nanoplatelets Raman spectrum (Fig. S2) and with that of the h-BN nanopowders (Fig. S3), respectively.

Table 2
Roughness parameter values, *Ra*, and their uncertainties σ of worn surfaces lubricated with the all the studied nanolubricants and PAO 40 using a Gaussian filter with a long wavelength cut-off of 0.08 mm.

Lubricant	Ra/nm	σ
PAO 40	181	18
0.05 wt% GnP	127	10
0.10 wt% GnP	121	13
0.25 wt% GnP	111	10
0.50 wt% GnP	115	12
0.25 wt% h-BN	142	15
0.50 wt% h-BN	135	13
0.75 wt% h-BN	131	14
1.00 wt% h-BN	137	10

The h-BN and GnP distribution in the worn track is parallel to friction lines, which shows that nanomaterials can enhance the lubrication capability of the base oil forming tribofilms at the surface contact. Considering that the worn surface after tests with nanolubricants of h-BN or GnP are smoother than that with the PAO 40 (Table 2), and the corresponding Raman analyses, it can be assumed that for h-BN and GnP nanodispersions the key tribological effects are the surface repairing and the formation of tribofilms due to nanoparticles. Çelik et al. [29] and Choudhary et al. [36] also determined that the surface repairing effect explains the antifriction and antiwear properties of some lubricating oils with h-BN and graphene family additives, respectively. Furthermore, in the case of GnP, microstructure changes can also occur. Zhao et al. [37] studied the lubrication behavior of different graphene nanoadditives with several exfoliation degrees, observing that the few-layer graphene with bigger interlayer spacing can enhance the lubrication properties of oil. These authors concluded that tribological improvement is due to the formation of ordered graphene tribofilms at the friction interface and owing to the graphene microstructure changes during tribological tests. There again, rolling bearing and synergistic effects are discarded due to the fact that h-BN and GnP nanoadditives present non-spherical shape and there is not cooperation with other additives.

Fig. 3 also shows that for the friction coefficient of nanolubricant with highest h-BN concentration (1.00 wt% in h-BN) is quite higher compared to lower concentrations (0.50 wt% and 0.75 wt%). This fact may be owing to the nanoparticles agglomeration at high concentration (1.00 wt%), h-BN nanoparticles are likely not just to fulfill worn surface valleys but also generate new asperities. These produced asperities increase the roughness of worn surface (Table 2) and might produce

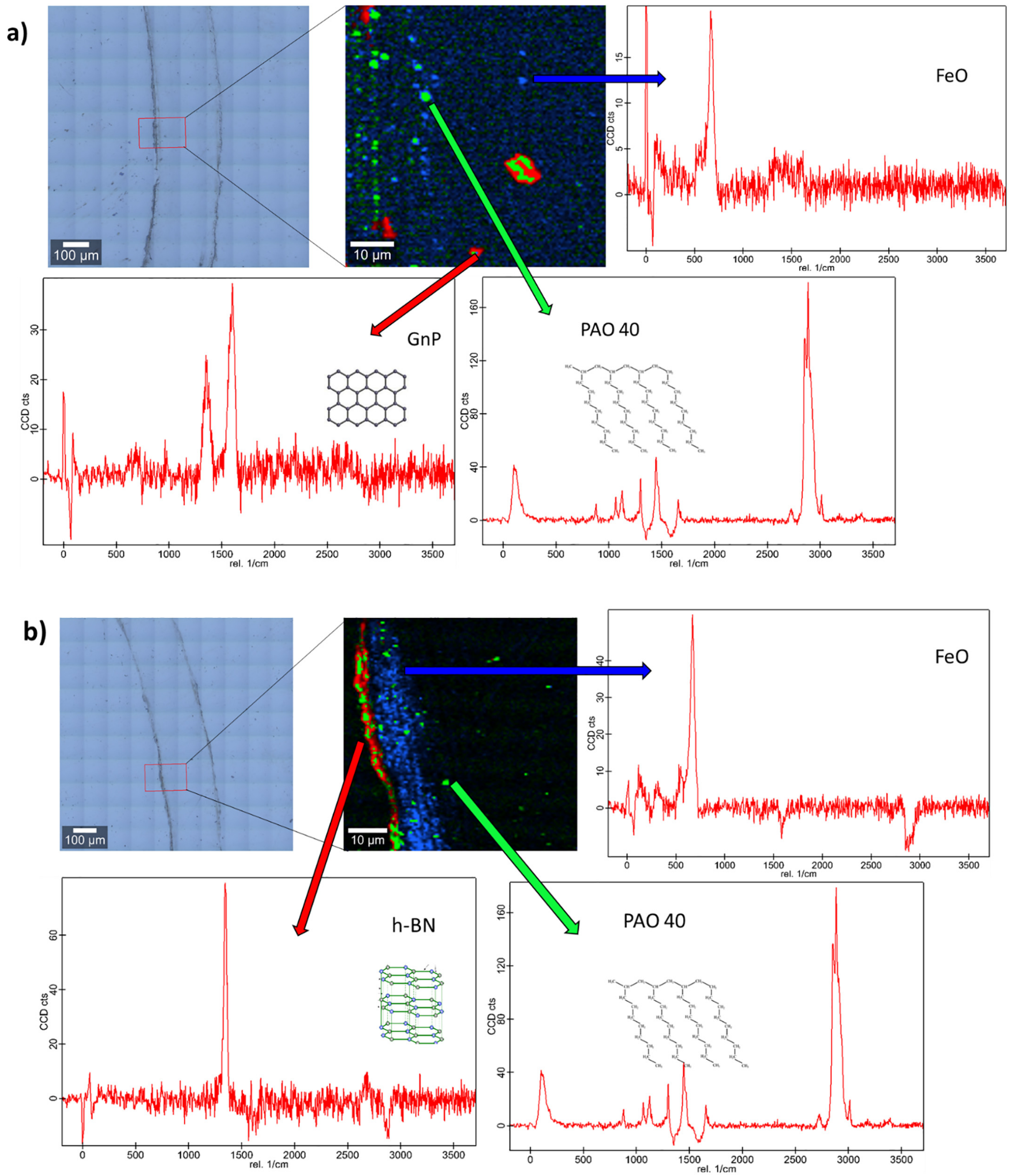

Fig. 5. Elemental mapping and Raman spectra of the worn tracks tested with the nanolubricants: a) PAO 40 + 0.50 wt% GnP and b) PAO 40 + 0.75 wt% h-BN.

higher friction when they are compared to nanoparticles with slight agglomeration [29].

4. Conclusions

The subsequent conclusions were reached in this work:

- Eight PAO 40 nanodispersions were prepared, four nanodispersions in hexagonal boron nitride nanoparticles (h-BN) and four others based on graphene nanoplatelets (GnP), showing stability times of at least 96 h.
- Coefficients of friction achieved for all the nanolubricants are smaller than that obtained for PAO 40 without additives.
- The best friction reduction was reached for the 0.50 wt% GnP nanolubricant, with a 21% maximum friction reduction with respect to base oil.
- For h-BN nanolubricants the best antifriction behavior was achieved for the 0.75 wt% nanolubricant, with a friction reduction of 20%.
- For all the nanolubricants (based on GnP and h-BN) the produced wear in disks is smaller than for the PAO 40. Specifically, the maximum reductions in the WTW and cross-sectional area were found with the 0.50 wt% GnP nanolubricant, being 22% and 19% respectively.
- For h-BN nanolubricants the best anti-wear performance was obtained for the 0.75 wt% h-BN with reductions of 20% and 13% in WTW and cross-sectional area, respectively.
- Through the Raman and roughness analyses, the surface repairing and tribofilm formation mechanisms due to the nanoparticles were confirmed.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.molliq.2021.115911.

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