Influence of Lubricants Degradation Level over Tribological Properties

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Abstract: *The paper presents the tribological aspects of the solid-fluid interaction, in order to develop an easy to use fast diagnoses method for the liquid lubricants, with minimal investments and a high precision level. The test program, using a TIMKEN equipment, analyzed the influence of the lubricant state of degradation, considering the tribological behavior of the friction couple during the working time. Two different lubricants (15W-40 and H46-EPS/1) were tested, in both fresh and used state. Tests were performed using three values of the normal load and two sliding velocities. During the tests, the wear trace (width and depth) was measured. Finally, pictures of the damage area were obtained with NEOPHOT 21 metallographic microscope. Spectrometric analysis was performed to identify the geometry, shape and concentration of the wear particles for fresh and used lubricants. The main result of this research is an altenative method of evaluating and quantifying the wear degree and lubricants durability.*

Key Words: *Degradation, Lubricant, Wear, Microscopy*

1. INTRODUCTION

Identifying ways to damage engineering surfaces and lubricants used requires the selections of appropriate experimental techniques to characterize morphology, tribology, rheology as well as microstructural properties. The phase of expertise-diagnosis consists in determining the causes and the degradation processes of a mechanism or part of it, based on the tribological analysis of the examined device and thanks to specific techniques selected for the expertise [1, 2].

In terms of component degradation, in addition to fatigue and corrosion, wear is one of the three most common problems encountered in the industry. In the technical literature, among the many parameters that are reported to control the wear mechanism, it is clear that most of them are extrinsic to the materials [3, 4]. Very few relations are related to the intrinsic properties of the contact materials, such as tensile strength, Young's modulus, hardening coefficient, etc. Nonconforming friction couples are particular to the hertzian contact pressure, as well as to some reduced contact zones, deformable under a certain stress, thus, a thin lubricant film is provided [5, 6].

It is demonstrated that, in certain conditions of roughness, material, stress, lubricant, relative velocity and temperature, for these nonconforming friction couples, after running-in period, it can still be provided an optimal lubrication with a thin and continuous lubricant film as EHD system requires [7, 8]. The limit and mixed systems were known long before the elastohydrodynamic system of lubrication. Initially, this system was only theoretical interpreted for point contacts (sphere/sphere or sphere/plane surface). Later on, the researches were extended to the linear contact. In both cases, the hertzian conditions (plain surfaces with elastic deformation under stress and without friction) were considered [9, 10].

Regarding the lubricant properties, among all experimental methods for assessing the degradation degree, we will focus on two methods: rheological properties measurement [11, 12] and electron microscopy investigation of the wear particles identified in used lubricants [13, 14]. Concerning the measurement of the rheological properties, generally it can be noticed that the viscosity of the degradated oil decrease compared to the fresh oil due to the change in physico-chemical properties of oil [15, 16].

Electron microscopy is important by completing the structural analyzes performed by optical microscopy and X-ray diffraction. Electron microscopy applied to oils is rarely used due to problems that occur in the case of: sample preparation (the method is difficult and requires repetitions for safety of information preservation) and shortcomings that can be brought to keep the vacuum high in the device column (if traces remain fat in the sample) [17, 18].

This paper presents the tribological aspects of the solid-fluid interaction, in order to obtain an alternative evaluation method for the lubricant durability. Its purpose is the development of an easy to use fast diagnoses method for the liquid lubricants, with minimal investments and a high precision level. The test program, using a TIMKEN equipment, analyzes the influence of the lubricant state of degradation, considering the tribological behavior of the friction couple during the working time. Two different lubricants (15W-40 and H46-EPS/1) were tested, both in fresh and used state.

Tests were conducted using three values of the normal load and two sliding velocities. During the tests, the wear trace (width and depth) was measured. Finally, pictures of the damage area were obtained with NEOPHOT 21 metallographic microscope. Spectrometric analysis was performed, in order to identify the geometry, form and concentration of the wear particles for fresh and used lubricants. The principal result of this research is an alternative method of evaluating and quantifying the wear degree and lubricants durability.

2. EXPERIMENTAL PROCEDURE

Tribological tests were performed using a TIMKEN tribometer, in order to permit the measurement of the wear for the friction couple. The geometry of the friction couple is presented in Figure 1, and a general view of the stand is shown in Figure 2.

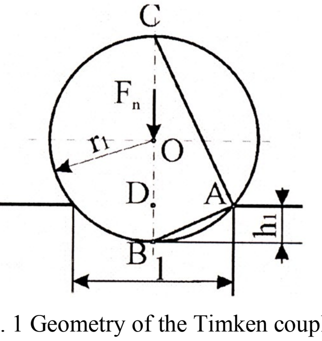


Fig. 1 Geometry of the Timken couple

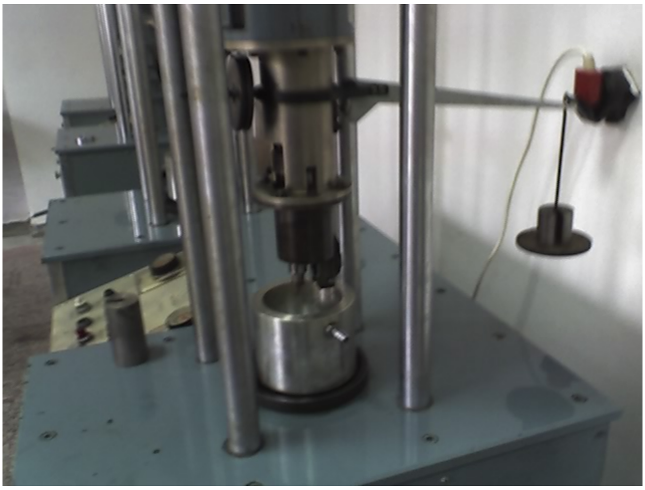


Fig. 2 View of the Timken tribometer

The testing program analyzes the influence of the lubricant state of degradation, considering the tribological behavior of the friction couple, during the working time. Each of the parallelepiped samples has two active plane surfaces, measuring 17 x 15 x 10 mm, made of C45 steel, and the final process of mechanical processing of the active surfaces was rough grinding.

The cylindrical samples, with the diameter 25 mm, were made also of C45 steel and the final process of mechanical processing was finish grinding. C45 grade steel is a medium carbon steel offering moderate tensile strengths. The material is capable of hardening by quenching and tempering on limited sections, but can also be flame or induction hardened to HRC 55.

The cylinder-plane friction couple was immersed in a tank filled with this lubricant. During all tests, the oil temperature was kept at a constant value ($T = 40^0$ C). Tests were performed using three values of the normal load F_n (30 N, 40 N and 50 N) and two sliding velocities (1.31 m/s and 1.83 m/s), during 1 minute. For each friction couple, the wear trace (width and depth) was measured. The linear wear was estimated using the impression method.

All the tests were conducted in the presence of two lubricants, 15W-40 and H46-EPS/1, both in fresh and used state. 15W-40 oil was used in an essence motor vehicle, with 80000 km way, while H46-EPS/1 oil was used by operation for 6500 hours in a hydraulic system (high pressure cylinders). The physical and chemical properties of the fresh tested lubricants are presented in Table 1.

Table 1: Physical and chemical properties of the fresh lubricants

Characteristic parameter	15W-40	H46-EPS/1
Density at 15°C	868 kg/m^3	900 kg/m^3
Viscosity at 40°C	90.48 cSt	46.13 cSt
Viscosity at 100°C	13.59 cSt	6.71 cSt
Viscosity Index	152	98
Pour point	-33° C	-
Flash point COC	238° C	206° C

In order to quantify the degradation degree of oils, rheological tests were performed on a Brookfield CAP2000+ viscometer (Figure 3). Pictures of the damage area were obtained with NEOPHOT 21 metallographic microscope. Non-crystalline and crystalline particles from fresh and used lubricants were analised by TEM and SAED determinations and they have been correlated with statistical interpretations (statistically indicators, dimensional distributions, average values, etc).

Fig. 3 Brookfield CAP2000+ viscometer B

The equipment used for these determinations was a JEOL Transmission Electronic Microscope - JEM – 200 CX type (Figure 4), having the following characteristics: resolution of grating plans: 1.4 Å, point-by-point resolution: 2.6 Å, acceleration voltage: 80, 100, 120, 160, 200 kV, magnification:1000 x – 330.000 x.

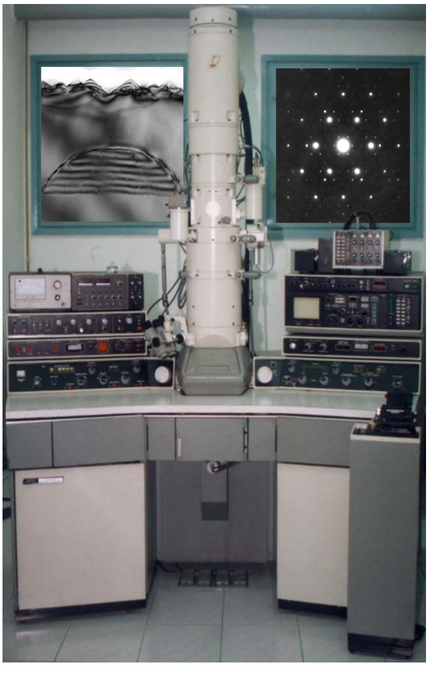


Fig. 4 JEOL Transmission Electronic Microscope - JEM – 200 CX

3. RESULTS

The experimental results concerning the rheological properties (variation of the shear stress versus shear rate) are presented in Figures 5 and 6.

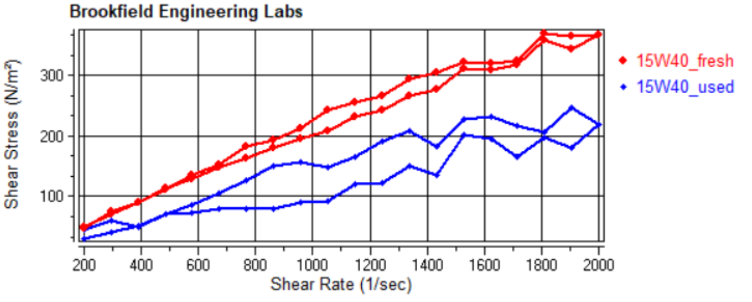


Fig. 5 Rheogram for 15W-40 oil, in fresh and used state

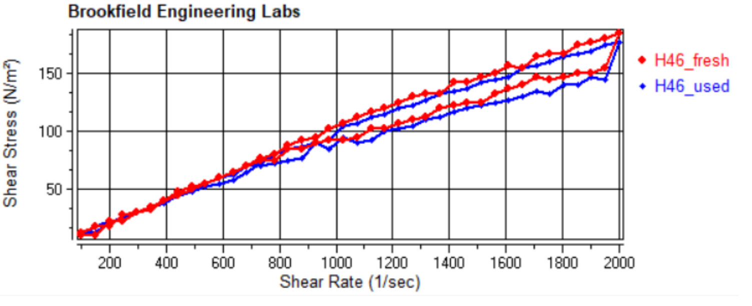


Fig. 6 Rheogram for H46-EPS/1oil, in fresh and used state

By processing the rheological experimental results by the regression analysis method, the viscosity values for both oils can be obtained. (Table 2).

Table 2: Viscosity for 15W-40 and H46-EPS/1 oils, in fresh and used state

Lubricant	Lubricant state	Viscosity, Pa·s	Correlation coefficient, %
15W-40	fresh	0.189	99.5
	used	0.122	96.8
H46-EPS/1	fresh	0.098	99.8
	used	0.092	99.7

The main tribological parameters (width and depth of the wear trace), function on the normal load and sliding velocity, are presented in Tables 3 and 4.

Table 3: Tribological results for sliding velocity 1.31 m/s

Load [N]	Lubricant	Lubricant state	Wear trace width [mm]	Wear trace depth [mm]
30	15W-40	fresh	0.832	0.008
		used	1.014	0.012
	H46-EPS/1	fresh	0.895	0.007
		used	0.998	0.014
40	15W-40	fresh	1.045	0.012
		used	1.132	0.018
	H46-EPS/1	fresh	1.008	0.011
		used	1.383	0.018
50	15W-40	fresh	1.230	0.015
		used	1.512	0.023
	H46-EPS/1	fresh	1.230	0.015
		used	1.688	0.025

Table 4: Tribological results for sliding velocity 1.83 m/s

Load [N]	Lubricant	Lubricant state	Wear trace width [mm]	Wear trace depth [mm]
30	15W-40	fresh	0.924	0.009
		used	1.245	0.014
	H46-EPS/1	fresh	0.998	0.008
		used	1.145	0.016
40	15W-40	fresh	1.158	0.015
		used	1.256	0.020
	H46-EPS/1	fresh	1.245	0.013
		used	1.542	0.021
50	15W-40	fresh	1.463	0.019
		used	1.791	0.025
	H46-EPS/1	fresh	1.542	0.017
		used	1.846	0.028

In order to have a better comparison concerning the behavior of the fresh and used lubricant, Figures 7, 8, 9 and 10 show the variation of the wear for different values of the load.

Fig. 7 Width of the wear for sliding velocity 1.31 m/s

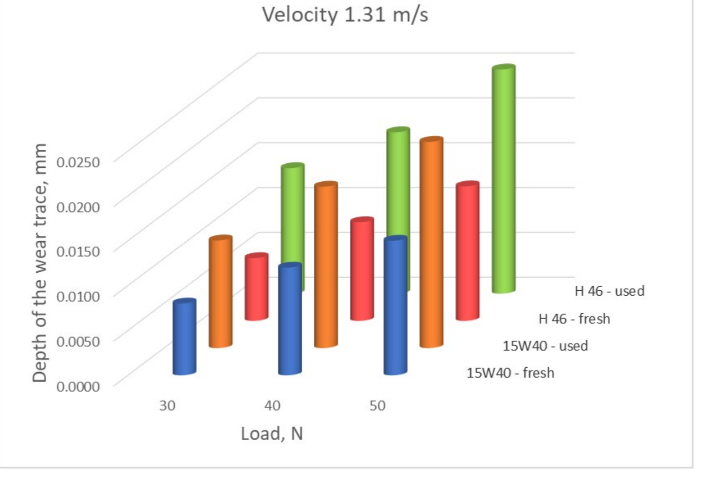


Fig. 8 Depth of the wear for sliding velocity 1.31 m/s

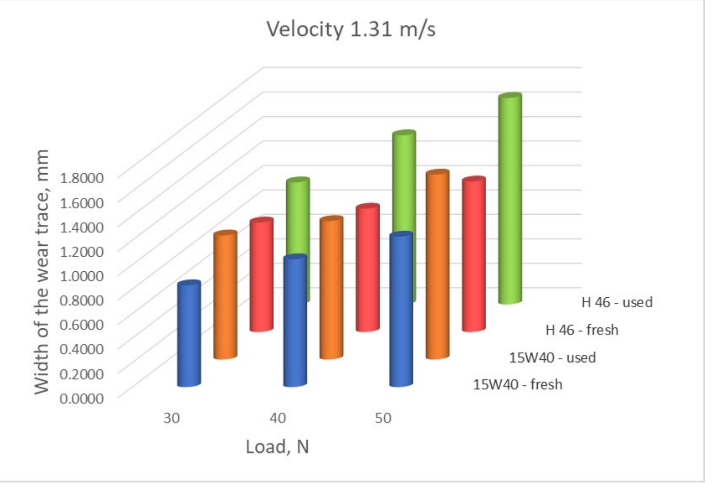


Fig. 9 Width of the wear for sliding velocity 1.83 m/s

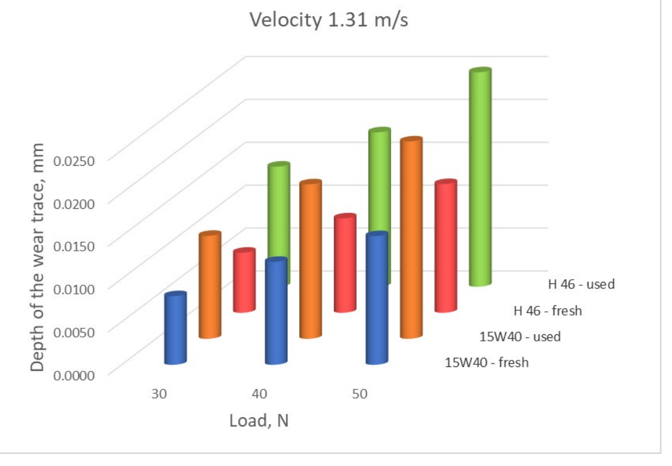
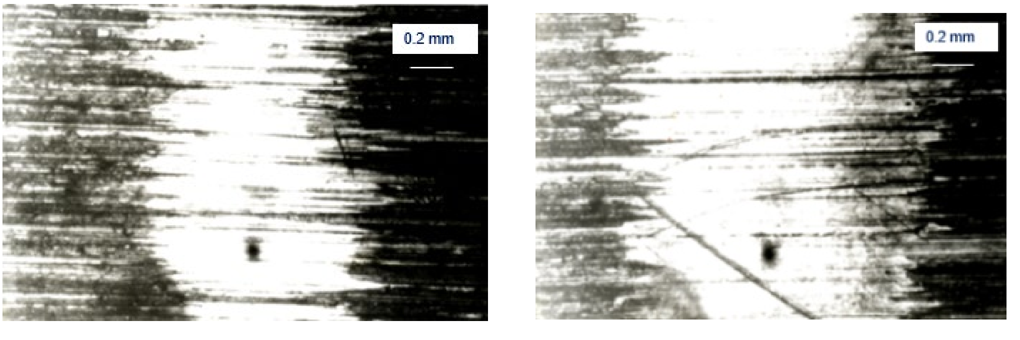


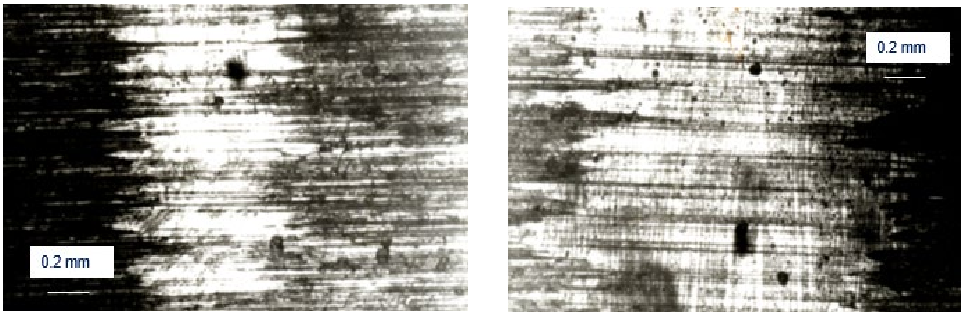
Fig. 10 Depth of the wear for sliding velocity 1.83 m/s

Differences between the fresh and used oils can be observed more easily using metallographic microscopy. Figures 11 and 12 show the images of the superficial damages for surfaces, which were obtained with NEOPHOT 21 microscope.



a. Fresh lubricant b. Used lubricant

Fig. 11 Wear trace for 15W-40 oil



a. Fresh lubricant b. Used lubricant

Fig. 12 Wear trace for H46-EPS/1 oil

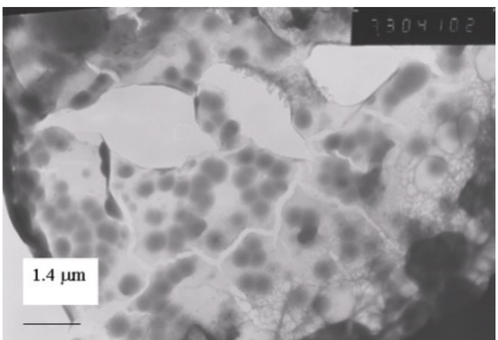
Tables 5 and 6 present the electron microscopy results obtained for 15W-40 oil, in fresh and used state, while Tables 7 and 8 present the similar results for and H46-EPS/1 oil.

The particles identified in fresh and used oils represent various microscopic solid components (wear particles, additives etc.), resulting from the normal process of oil degradation during operation.

Unfortunately, the same particle type could not be identified in fresh and used oil, due to the large number of tests and acquired images required.

The obtained results constitute the core of a database regarding the solid particles that can be identified in oils.

Table 5: Structural particles visualization in 15W-40 fresh oil

Phase	Characteristics
δ - CaP_2O_6 (X) (ASTM 15-0185)	Particles are nanometrical, having spherical and/ or ellipsoidal shape. Dimensional values range is for particles system (2 – 28) ± 0.01123 nm. Particle average is ~ 14.88 nm.
Structure	
 	
Phase	Characteristics
Hexagonal ZnO (ASTM 80-0075)	Particles have round boundaries; they are not uniform as dimension. Their values range between (20 – 138) ± 0.0821 nm and the average is 58 ± 0.01254 nm.

Structure

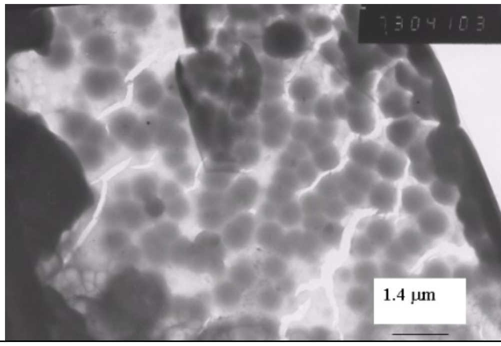
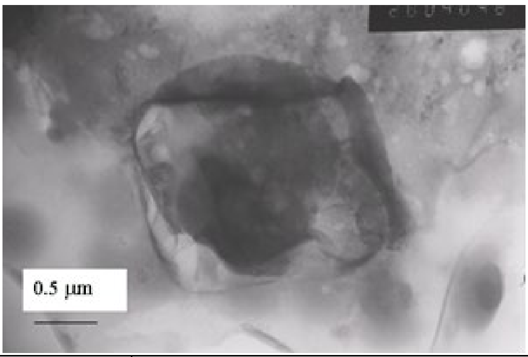


Table 6: Structural particles visualization in 15W-40 used oil

Phase	Characteristics
Rhombohedral V (ASTM 15- 0185)	Particles have a poliedralle shape with well crystallized round bounderies. Their values range between (70 – 122) ± 0.224 nm. and the average is 84 ± 0.0415 nm.

Structure



Phase	Characteristics
$VO(OH)_2$ monoclinic (ASTM 10-0377) + traces of additives (soap, polymers, etc.)	They suffered dimensional transformations especially during operation. There are micrometric particles with precipitates of average value ~ 0.18µm.

Structure

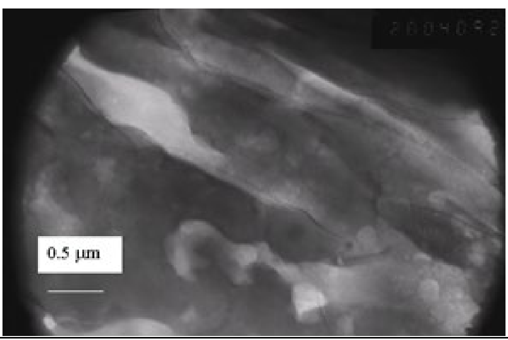


Table 7: Structural particles visualization in H46-EPS/1 fresh oil

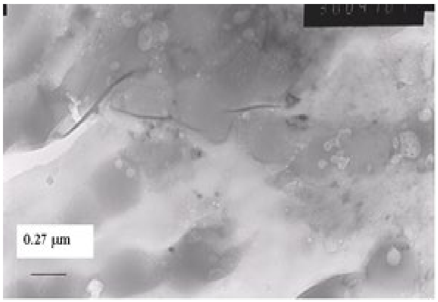
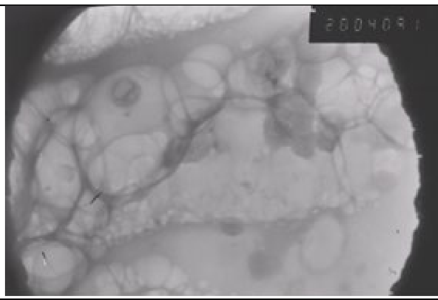
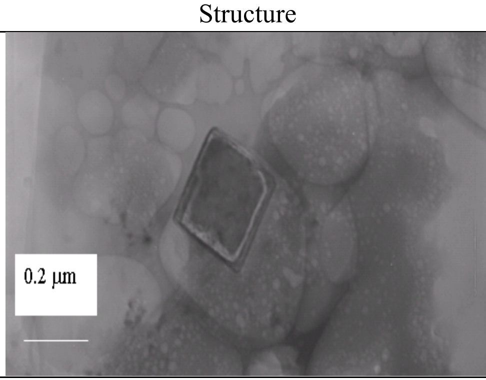
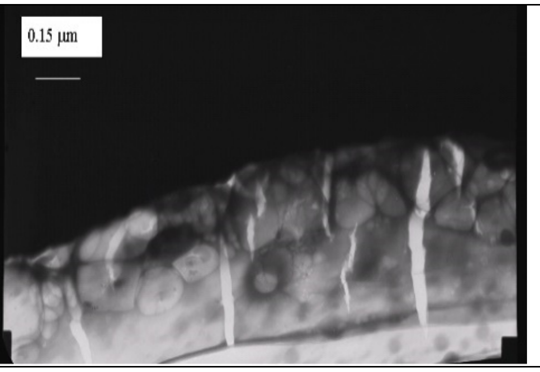
Phase	Characteristics
Amorphous phase	The amorphous phase presents particles with well-determined form (soap, polymer), with dimensions ranging between 50 and 100 nm, and an average value of ~ 82 ± 0.257 nm.
Structure	
	
Phase	Characteristics
Mo_2S_3 (ASTM 12-0692)	The particles measure between 50 and 80.4 nm and have an average value of 65.2 ± 1.55 nm.
Structure	
	

Table 8: Structural particles visualization in H46-EPS/1 used oil

Phase	Characteristics
Al_9Cr_4 cubic (ASTM 02-1193) + Mo_2S_3 traces (ASTM 12-0692)	Al_9Cr_4 particles are platelet with rounded edges that tend to polyhedral form. There are micrometrics with average particles size ~ 0.28 ± 1,5 µm. There is a clear evidence of Mo_2S_3 traces.
Structure	
	

Phase	Characteristics
Orthorombic FeC (ASTM 03-0400)	Particles have round boundaries, their values range between (52 – 89) ± 0.3215 nm and the average is 70 ± 0.042 nm.
Structure	
	

4. DISCUSSIONS

From a rheological point of view, a decrease in viscosity can be noticed for the degradated oil, by 35.45% for the 15W-40 oil and 6.12% for the H46-EPS/1 oil. So, it can be observed that measuring rheological properties offers consistent information concerning the degradation state of oil.

Analyzing the tribological results, one can observe:

- Trace wear (width and depth) increases with increasing load and sliding velocity;
- For all the cases studied, the wear mark is higher - in the case of used oil - compared to that of fresh oil;
- For all the analyzed cases it was found that the wear trace is higher for the H46-EPS/1 oil than for the 15W-40 oil. The possible explanation is the presence of antiwear additives in the15W-40 oil, while the same additives are missing in the H46-EPS/1 oil.
- The wear trace is a very accurate indicator of level of oil degradation.

Regarding the electronic microscopy testings on fresh and used lubricants, there were identified different types of particles, such as: non - crystalline phases, incipient crystallizations, polycrystalline phases, monocrystals etc.

Also, information was obtained on particle shape, particle defects, dimensional repartition of particles by phases (using SAED), values range and average by particle. It can be observed that the level of impurities and the period of the used oil depend on the the exploitation conditions, the quality of the oil, the construction and the technical state of the equipment.

5. CONCLUSIONS

As an experimental scientific research in the field of technical sciences, this alternative method is particularly important, due to its thematic approach and the degree of novelty it brings.

The proposed method can diagnose the state of degradation of lubricants, with high precision and accuracy. The wear degree of the friction couples lubricated with used lubricants is much more important than the same in the case of fresh lubricants. The use of structural analysis of particles by electron microscopy allows the identification of the crystallographic structure and the nature of the smallest particles, even in the nanometric field.

The final purpose of this microscopic study was to create a database with the physical-chemical properties of the solid particles that can be identified in oils.

The method can be applied using a small quantity of oil, is very efficient from the economic point of view and the results can be directly used by the beneficiary.

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