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Sclero-topometry Metrology in Valorisation of Waste Oil for Micro-machining of Ductile Cast Iron

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Abstract

During the time, the specific characteristics and the efficient lifetime of oil progressively decrease, due to complex pollution, ultimately making the oil unsuitable for the initial applications. The strategy to regenerate and to valorise waste oils is investigated using improved combinations of sclerometric and topometric tests on ductile nodular cast iron. Tribo-abrasive tests are performed in critical conditions, with base oil, waste oil and regenerated oil, of similar viscosities in order to discriminate their interfacial performances. The forms of the scratch traces indicate wear resistance and tendency to elasto-plastic deformation. The mechanisms of deformation and frictional behaviours were evaluated using optical and Scanning Electron Microscopy and measured for various tribological conditions with tactile and optical profilometry. The Energy Dispersive X ray Spectroscopy completes the chemical superficial distribution of pertinent elements. The surface topography metrology is used to characterize the scratch profiles and to determine the volume of the displaced and removed material, as well as maximum pit height. The originality of this paper is that it is a unique approach specifically devoted to transformer oil concerning tribological conditions.

Keywords: Sclero-topometry, Waste oil, Scratch test, Ductile cast iron, Oil revalorization in machining

1. Introduction

Nowadays many foundry processes are focussed on the process of surfaces morphological functionalization which one of the fundamental descriptors is the roughness parameter (cf conclusion and perspectives). One of the aims of this study is a better understanding the phenomenon existing on the interface between the mould characteristics and the casted and machined piece, in order to simplify the manufacturing process, including

foundry, and makes them less time and cost consuming. A usual final stage of those foundry processes is the machining, with cutting as well as abrasive processes, which are offering a high added value to the final function of products. That is the reason of the study of the re-use of waste oils in the comprehension of the elementary interactions between an abrasive grain and a surface. Nowadays it is well known that the petroleum resources are decreasing, and according to the estimation they should be dry up until 40 years. That is why recycling is the major challenge of

worldwide society today, specifically for petroleum resources, and therefore, for derivatives like transformer oil. Most power transformers use a liquid to electrically insulate and remove heat from the core and windings. The contradictory characteristics from physical approach of that oil, requiring and insuring simultaneously a high thermal conductivity as well as an electrical resistivity, are specific for their application. Durability during the efficient lifetime is also one of fundamental performances. The characteristics are simultaneously influenced by electromagnetic and thermal fields [1] by complex mechanical constraint states and by dynamics interfacial conditions between different material categories (vegetal, organic as well as metallic compounds). Therefore the final performances are progressively decrease, due to complex wear processes.

There is a lack of global tribological approach and critical analysis of progressive transformer oil degradation due to its pollution. There are multiples pollution origins: defects of seal design [2], wear debris formation due to vibration and fretting of contacting bodies. On the one hand the insulating paper degradation results in the formation of particles, water and gases [3], on the other hand the oil oxidation result in the formation on acid compounds and sludge. [4]. These are at the genesis of transformer oil performance reduction. At this stage the regeneration and revalorisation strategy should be elucidated and studied. That aspect is principal aim of that research.

In present paper via sclero-topometric investigations; waste and regenerated transformer oil and classical new naphthenic base oil are compared in term of its superficial action on microscopic scale on ductile cast iron surfaces and therefore boundary lubricants performances at very high stress conditions producing plastic deformations [5]. Motivations for selected materials are simultaneously ecological, economic and scientific developed below.

1.1. Ecological approach

Each year about 100 000 tons of industrial used oil are product [6], but only 5 000 to 10 000 tons are revalorized in France, including of transformer oils. The waste oils can be submitted to different treatments:

- Energetic valorisation via combustible (80%)
- Product Valorisation via regeneration (20%)

It is known that the first solution is not as safe as the second, insofar as combustion of waste oil leads to the formation of compounds like nitrogen oxides and oxides sulphur that contribute to acid rain formations or to the formation of polycyclic aromatic hydrocarbons (PAHs), whose carcinogenic potential is well known.

The second solution is a technologically and economically optimised valorisation [7], which is not an easy task, principally due to the following reason.

The required properties of electrical insulating fluids are high dielectric strength, low viscosity, high chemical stability and etc. Polychlorinated Biphenyls (PCB) fit those criteria, therefore they were widely used as dielectric fluid in transformers from 1930 [8]. PCBs ($C_{12}H_{10-n}Cl_n$) are the family of chemicals formed by attaching one or more chlorine atoms to a pair of connected benzene rings. In 1977, the Environmental Protection Agency (EPA) began to impose bans on PCB manufacturing and sales and

on most PCB uses, due to the fact that their ability to accumulate in the environment and to cause harmful effects became apparent [9, 10]. PCBs are probably carcinogenic in humans (group 2A classification, International Agency for Research on Cancer) and are classified as Persistent Organic Pollutant. The Stockholm Convention on Persistent Organic Pollutants in 2001 recommends to remove the equipment polluted with PCBs by 2025 and to achieve disposal in an environmentally sound manner by 2028 [11].

Comparing with oxidative methods, reductive methods avoid the production of noxious chemicals and have potential application. Among them, photochemical, electrochemical, ultrasonic, supercritical, microbial, and radiolytic and thermal reductive methods have been reported [12].

1.2. Economical approach

Recently one can note the proliferation of specialized companies in waste oil collection, oil recycling and collection of hazardous waste fluids, but only few of them, due to lack of efficient market model and complexity of technological process, considers all aspects simultaneously from collection to valorisation. The regeneration and valorisation strategies increase the technological difficulties. It is one of the specificity of Daffos&Baudasse Co, which is specialized in the treatment of industrial oil.

One can state that concerning the production of ferrous and non-ferrous alloys, France is one of the worldwide leaders. About 40% of those parts belong to the category of ductile cast irons. That is a reason of the choice of the selected material for that study. Moreover the selected material for the experimental investigation (ductile cast iron) is widely cast and machined by Ferry Captain Co. The developed strategies promote collaborative scientific and technological efforts to satisfy the end users through optimised revalorisations of waste oils into functional products. It is one reason of the cooperation with Ferry Captain Co. That is part of the objectives of the present paper.

2. Material and methods

2.1. Petroleum products

The tests were performed with three types of oils:

- New naphthenic base oil and commercial lubricants.
- Waste oil, resulting from the mixture of collected used transformer oil. This oil was deliberately selected for its high PCBs pollution (about 150 ppm).
- Regenerated oils. This oil has been treated in order to remove the degradation products (acids, particles, sludges...) and the pollutants (PCB, water...).

For tribological reasons selected oils have comparable rheological behaviours with similar viscosities (about 10^{-5} m²/s at 313°K) in order to compare the physical chemical reactivity and influence more than the rheological role.

The oil samples were deposited on investigated surface in manner to fulfil totally interface composed of indenter and cast iron sample before the sclerometric test.

Experiments without lubricant have been carried out to reveal effect of lubricants.

2.2. Ductile cast iron

Ductile cast iron is a heterogeneous material [13, 14], also known as nodular cast iron or spheroidal graphite cast iron, it was invented relatively recently in 1943. Nodular cast iron is a currently used as construction material with a wide range of applications in engineering practice and particularly; cylinder liners, gears for mining & cement industry transition power systems, offshore, windmills, material conversion from cast steel to ductile cast iron, maritime industry etc. Nodular cast iron is obtained by adding magnesium just before casting, this encourage the graphite to form spheres or nodules [15]. Tribological behaviour of nodular cast iron depends on the different individual constituent properties, solidification process and therefore metallurgical structure, on their interactions with lubricants and the tribological system parameters [16].

2.3. Surfaces preparations

Material for experimental investigations was supplied by Ferry Capitain Co due to its expertise in foundry of ductile cast iron, master of its solidification and manufacturing. For taking into account structure gradient the selected samples from bloc 400x200x800mm were carefully sawed in the form of plates of 20x20x10 mm. The chemical analysis of selected material is 2,65%C, 1,94%Si, 0,37%Mn, 4,89%Ni, 0,43%Mo, 0,10%Cr, 0,019%P, 0,004%S (spheroidal graphite characteristics – according to standard EN ISO 945:1994: shape VII, size 6). According to EN 1563:1997 grade of ductile iron is EN-GJS-450-10 by added Ni. Average Hardness is 199HB. Then the of ductile cast iron samples was polished to remove minor surface imperfections, applying the expertise developed during study of morphological kinetics of abrasive process on different cast irons [17]. The metrological strategy based on polishing and morphological measurements is summarize in the cycle illustrated below in Figure 1.

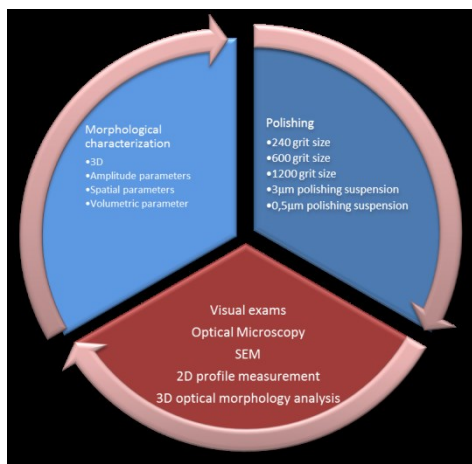


Fig. 1. Cycles of surface metrology of morphology characterisations during polishing process

The samples with homogeneous distribution of spheroidal graphite were carefully prepared as shown on Figure 2 (a). Size of graphite nodules of the selected nodular cast iron were predominantly ranging from 5 to 15 μm . The etched surface analyse with an optical microscope reveal the principal metallurgical structures, with the ferritic and pearlitic matrix (figure 2(b)).

2.4. Surface morphology measurements

The different surface metrology techniques, applied systematically to morphology characterizations of polished cast iron submitted to sclerometric investigations, are versatile:

- Light optical Microscope for observations performed on the selected material at different magnifications depending on different tasks. The examinations were conducted at 5, 10 and 20x magnifications to evaluate the first stages of surface preparations.
- Numerical microscope to do observations of the indenter tip with a numerical microscope Keyence. This technique allows the characterisation of the real tip radius of the indenter.

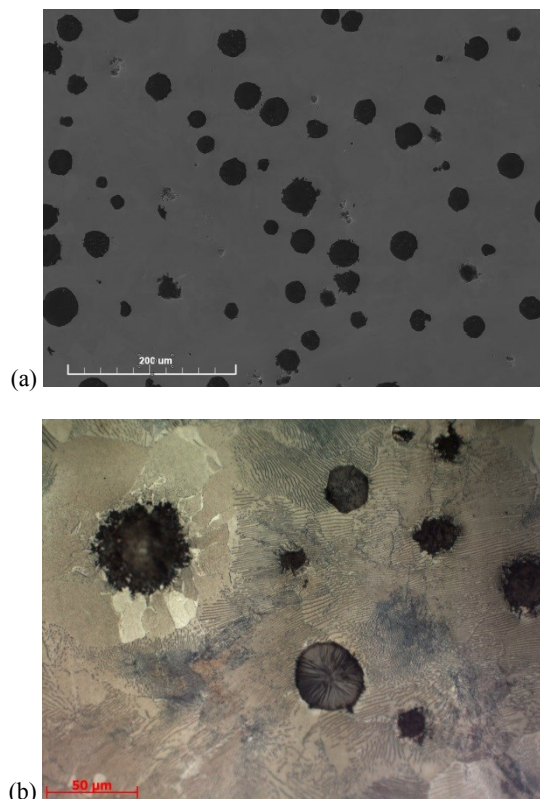


Fig. 2. SEM of large area showing homogeneity of spheroidal graphite of ductile cast iron (a) with 500x magnification (b) optical microscopic picture revealing principal metallurgical structures

- Scanning Electron Microscope: a standard SEM Vega-Tescan SEM (Czech) was used for all morphological

- observations after polishing process as well as residual sclerometric traces. Energy Dispersive X ray Spectroscopy (EDX) offered chemical analysis of different elements in complementary characterisation of surface metrology. Back scattered electron (BSE) method offered chemical topography of the surface.
- 2D Tactile profilometry with a tactile topometer Surfscan Somicronic (French) was essentially devoted to profilometry investigations of the scratches from residual sclerometric measurements in frame of surface metrology of rheological behaviour of ductile cast irons. A diamond stylus of $2\mu\text{m}$ tip radius was traversed at a constant speed of $10\mu\text{m/s}$ across the sample with a force of 10-3 N. Profiles were measured every 5mm, from the first to the fourth mm along the scratch. The vertical resolution of the measurement was 6nm.
 - Optical 3D topometer: with a 3D optical topometer, WYKO 9300NT Optical System, Veeco Instruments Inc., (US), was used to study the surface topography of the scratched samples. The vertical scanning interferometry (VSI) mode, based on interference fringe of the light reflected from a reference mirror and the light reflected from the sample surface, was used to determine the scratch surface topographies.

3. Sclerometric investigations

Sclerometry is one of the metrological techniques used to characterise the dynamic superficial rheological properties of solids in terms of its elastic, brittle and plastic deformation aptitude [18, 19, 20] of pure and coated materials [21, 22].

Acquired knowledge and experience with previous metrological device using fully computerized sclero - topometry described in details in [23] have oriented the authors to separate the techniques of sclerometry and topometry in order to increase the precisions of collected data.

Scratch tests were performed using a CSM instrument Tester (Swiss). A spherical micro-contact rounded Conical Rockwell diamond indenter, with $200\mu\text{m}$ tip's radius (R) and 120° angle was used in continuous mode. During sclerometric investigations, the ductile cast iron sample is horizontally translated at a controlled speed while the stationary diamond tip stylus is applied vertically under a specified normal load (F_n). In present paper are analysed the results of sclerometric investigations under the normal load varied between 1N and 23N to observe the effect of contact pressure on scratch trace deformations modes at (10mm/min) scratching speed. During the test the normal (F_n), the tangential (F_t) loads and the vertical indenter displacement were measured and recorded. Each test was replicated at least four times respecting a distance between scratches in order to avoid mutual interactions. Consequently, the sclerometric results are pertinent and representative of the average response over larger surfaces and of tribological as well as kinematic conditions.

The material behaviour, during sclerometric experiments for heterogeneous material, involves complex dynamic contact phenomena, elastic and large plastic deformations, in some cases fractures which are very complicated to be scientifically interpreted. Measuring the post scratch orthogonal cross-section of

residual groove in case of negligible residual stresses and visco - elastic relaxation, (B) represents a part of penetrated material by the indenter.

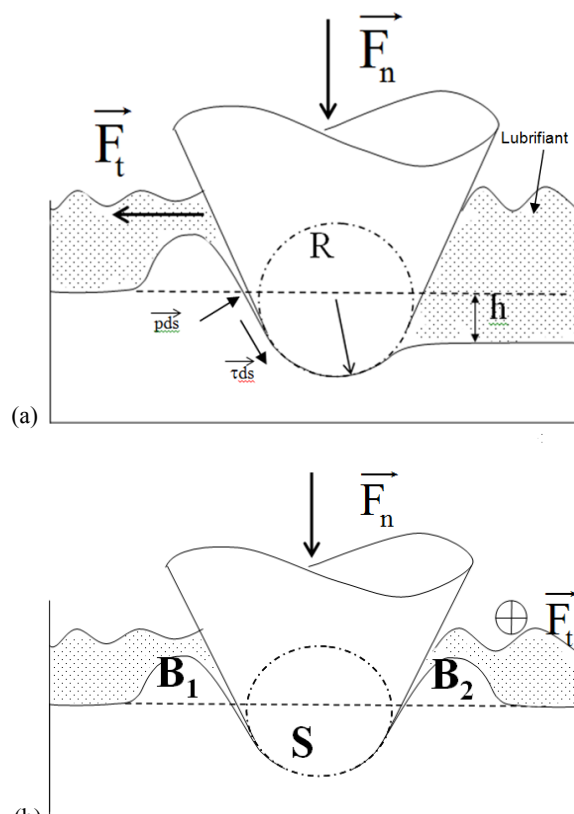


Fig. 3. Schematic view of scratch groove; (a) the indenter in direction of its displacement showing frontal prow of deformed material, (b) orthogonal cross-section view of the indenter with the laterally displaced material.

The prow of visco - elasto plastically deformed material pushed in front of indenter is contributing to combined effect of expelled from orthogonally to the surface penetrating indenter and laterally displaced material to form on the side mater deformation sections (A_1 and A_2) illustrated in Figure 3b. This phenomenon is due to the complex deformation process of the ductile cast iron [24]. Therefore in some cases the part of the material can be transferred to the lateral ad frontal prow while the rest of the material is removed from the surface as cutting chips. The morphology of the scratch traces depends on different parameters: the frontal area of the indenter, the efficiency of the removal process, tribological and kinematics conditions, etc... [25]. Those mechanisms in case of ductile cast iron are also governed by the distribution of graphite nodules. In dry sclerometric contact conditions, with the increase of normal load, the deformation mode changes from ploughing to wedge formation until the cutting [26]. This phenomenon is not observed in conditions of presented investigations.

4. Results and discussion

4.1. Reproducibility of sclero - topometric investigations

In order to verify accuracy, errors, precision, reliability of sclerometric investigations as well as pertinence of selected area of ductile cast iron, the experiments have been reproduced 4 times for different kinematic and lubricant conditions.

The profile analysis in Figure 4 delivers some metrological information of the plastically deformed surface, through the measurement of the indented (or cut section) and the displaced parts of the sclerometric traces [27]. For selected heterogeneous material, the ratio of the penetrated surface to the part of ploughed section of material varies from 0 to 1.4 what is not completely astonished due to graphite cavities of very low hardness. The 3D analysis, which offering a global evaluation of the ratio of plastically penetrated volume to displaced volume to piles, varies from 1.2 to 2.5.

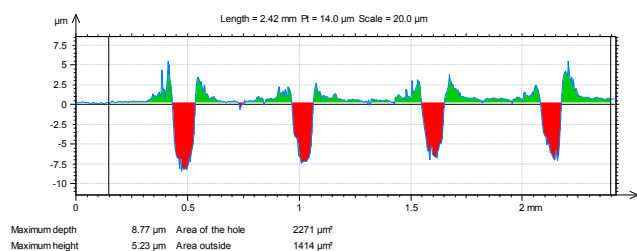


Fig. 4. Profiles of sclerometric traces obtained at 10mm/min under 23N and with waste oil show average ratios of ploughed peaks sections to penetrated section

The 3D topometer seems to be the more suitable analytical method for measuring the morphology of the scratch traces because it shows the surface distribution of the spheroidal graphite [28].

In present part presented results are concerning waste oil. The morphology obtained with the 2D tactile profilometer and the 3D optical topometer reveal a very good reproducibility of the scratch profiles. In order to confirm this first observation, a lot of parameters were measured for the four traces. One of them is the fluctuation of the tangential load (F_t), which was measured from the first to the fourth mm of the scratch (the begin and the end of the scratch have specificities that are not studied in that paper). In severe conditions, with a normal load of 20N and a speed of 10mm/min, the fluctuation of the tangential load is lower than 6%, what indicate very satisfying sclerometric reproducibility, insofar as tests were performed with a heterogeneous material. The Figure 5 shows morphological 3D views of sclerometric traces.

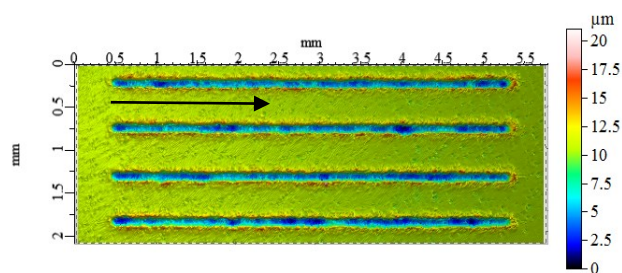
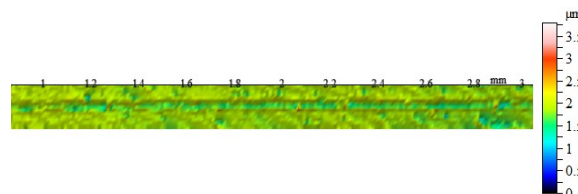
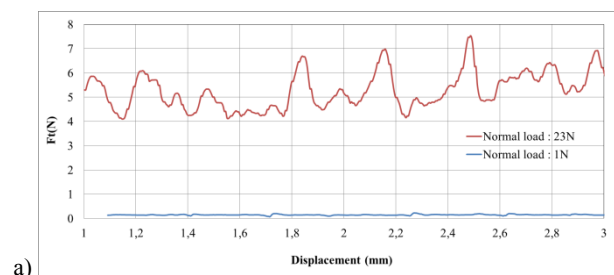


Fig. 5. An example of 3D top view of four sclerometric traces on ductile spheroidal cast iron showing satisfying experimental reproducibility. Conditions: spherical ($R=200\ \mu\text{m}$) diamond indenter, normal load ($F_n=23\text{N}$), displacement speed ($V=10\text{mm/min}$, arrow indicating direction), waste oil lubricant

4.2. Discrimination aspect of tribological conditions

In order to realize the dynamic and tribological characterization of the lubricants, the evolution of the tangential load is measured as a function of the displacement at various speeds and normal loads. In the present paper the results for a speed of 10mm/min and under a variable normal load from 1 to 23 N are discussed. The figure 6 presents an example of evolution and the corresponding 3D trace morphology obtained with the waste oil at 10mm/min and under a normal load of 1N and 23N showing discriminating effect of load. For a low normal load (1N), the indenter "slides" on the surface without producing any visible damage. In that case the friction coefficient is close to zero. For higher normal load, the amplitude of the F_t fluctuation revealed the metallurgical and rheological heterogeneity of the cast iron. Indeed the graphite cavities have a lower hardness than the cast iron matrix.



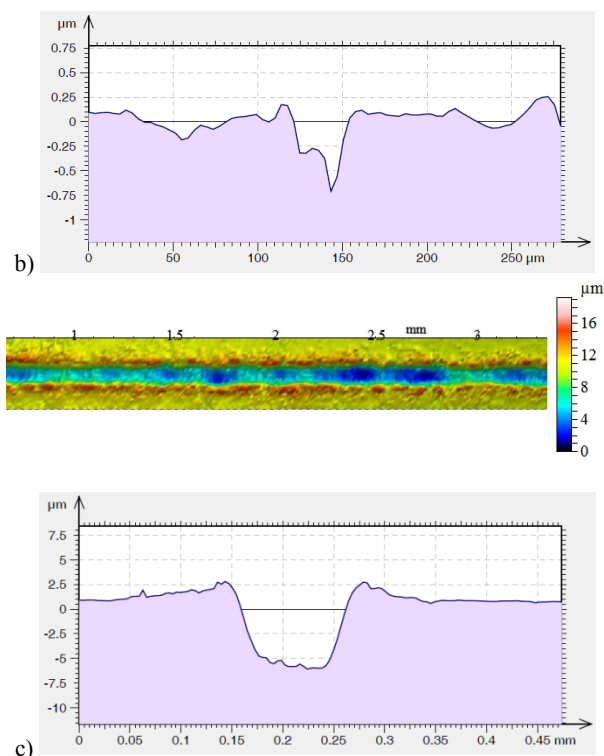


Fig. 6. Evolution of F_t - tangential force lubricated with waste oil as a function of displacement on 10mm/min under normal loads; a) $F_n=1N$. Morphological 3D top view of residual traces with corresponding extracted profile b) $F_n=1N$ in and c). $F_n=23N$

4.3. Lubricants effect discrimination

In order to discriminate the performances of lubricants most pertinent surface metrology in term of morphology and rheology via sclerometric investigations results are presented.

• Qualitative morphological analyses

It is known that morphology of surfaces influences wettability of deposited liquid, that aspect is not deliberately presented and discussed in the present paper, however this aspect of surface metrology is systematically investigated and as far as possible modelled [29].

The influence of lubricants in relation to dry contacts [30,31] shows some evident morphological effects on sclerometric traces, cf. Figure 7 respectively without lubricant (a), with waste oil (b), and regenerated oil (c). The non-lubricated contact cf. Figure 7(a) shows larger width and deeper traces with visible lateral ploughed parts material.

One can easily state that regenerated oil reduces considerably damages and offers smoothest trace's morphologies than with waste oil. Therefore waste oil as well as regenerated oil reduced interfacial share between ductile cast iron and diamond spherical penetrator.

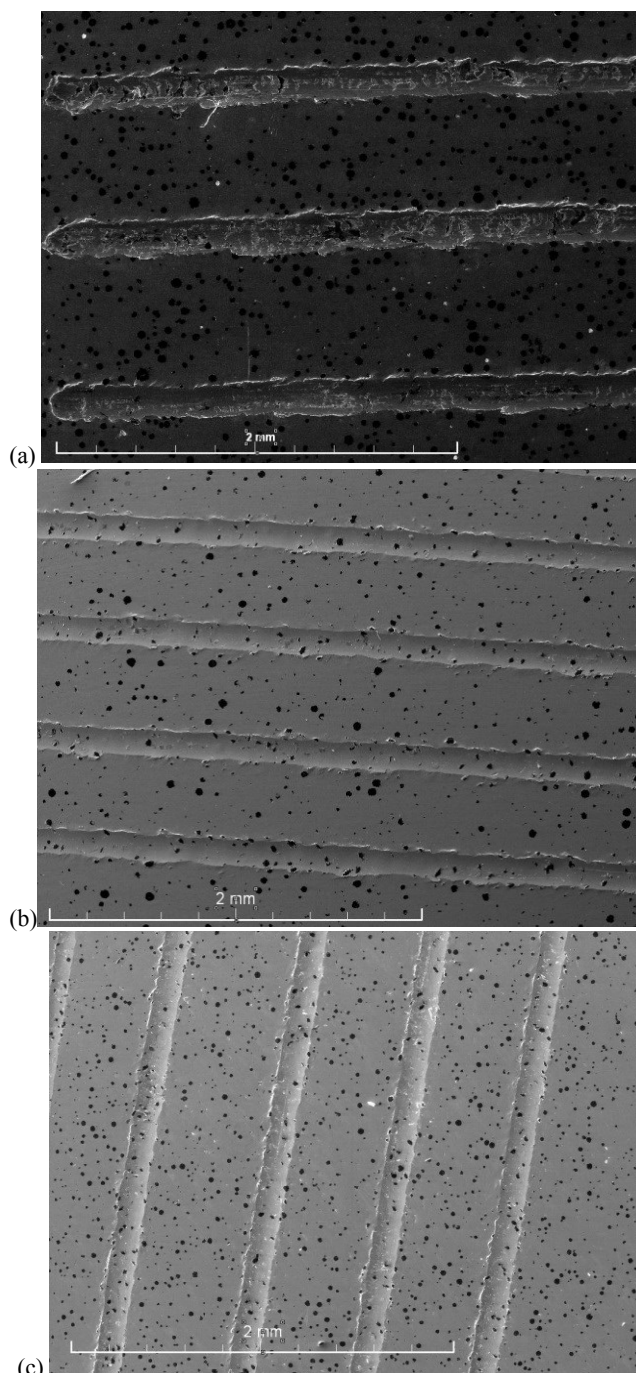


Fig. 7. SEM morphological views of sclerometric traces at 10mm/min and under 23N load; (a) without lubricant, (b) waste oil and (c) regenerated oil

Looking in details the discrimination effects on sclerometric trace's morphologies, influenced by interfacial conditions, it is straightforwardly revealed the differences between dry and lubricated contact. Cf. Figure 8 without lubricant (a), waste oil (b), and regenerated oil (c).

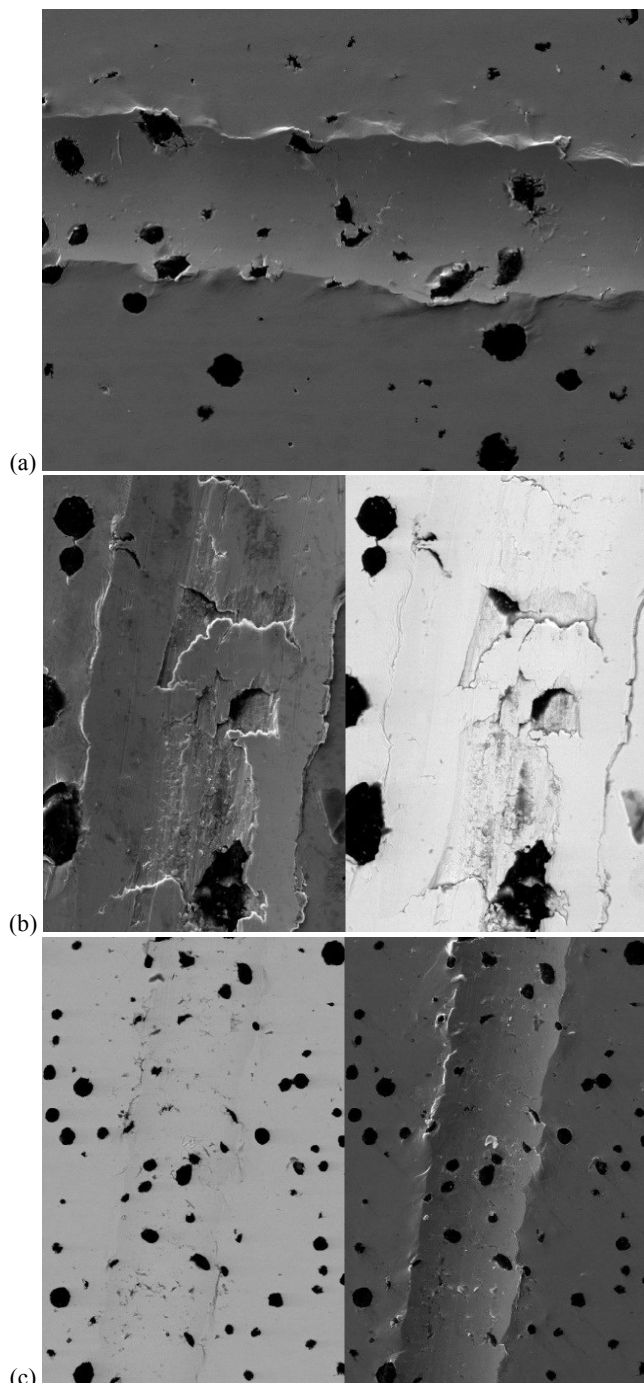


Fig. 8. SEM and BSE morphological views of sclerometric traces at 10mm/min and under 23N; (a) load without lubricant, (b) waste oil and (c) regenerated oil

The lack of lubricant (cf. Figure 8 a), increasing interfacial shear and internal adhesion of ductile iron (despite of diamond non reacting material), produces smear effect inside the trace. BSE image contributes to better understanding of different constituents

influence in damage process confirming by smoothness of bottom of the furrow.

Comparing sclerometric traces lubricated with waste oil (cf. Figure 8 b) and traces lubricated with regenerated oil (cf. Figure 8 c) it is easy to observe that there is a thinner width in case of regenerated oil. Therefore depth of penetration as well as morphology indicates totally different modes of damage due to the scratch. Particularly the BSE images of lubricated contact showed a more homogeneous mode than in case on non-lubricated contact where adhesion phenomena are probably totally eliminate.

• Quantitative surface metrology of sclerometric traces

Systematic measurements of a great numbers of sclerometric traces at different kinematics, dynamics and tribological conditions allow reaching a great number of assumptions. Thanks to the Mountains map software, the section profile of the scratch in real sclerometric conditions are analysed (cf Figure 9). For the measurements, the volume of the scratch penetration and the volume of ploughed material are separated by the P plan which is the least square plane on all points outside of the contour defined outside of the deformed area.

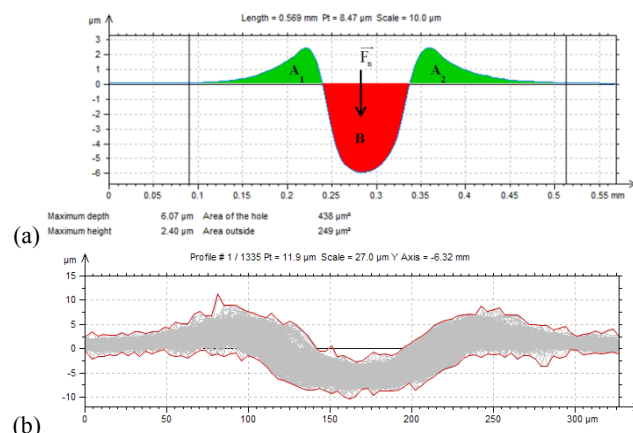


Fig. 9. Anamorphous profile's sections (a upper and cumulative lower with scale for real sclerometric conditions and corresponded deformed areas (A1, A2) for lateral pads and (B) for groove lubricated with waste oil under 23N normal load

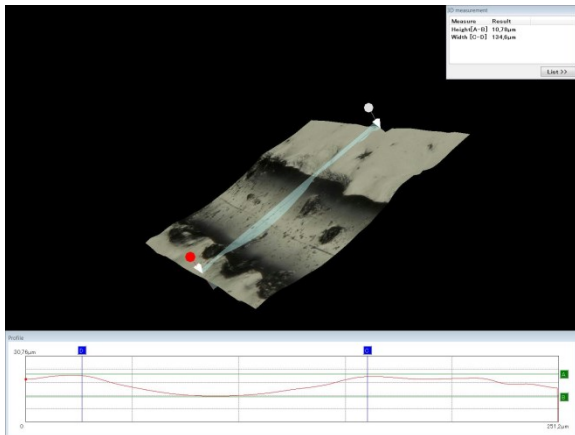


Fig. 10. Fragment of scratch trace of ductile cast iron lubricated with waste oil under 23N normal load

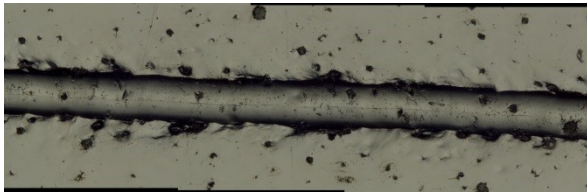


Fig. 11. Long range of scratch groove of 130 µm width, lubricated with waste oil under 23N normal load thanks to stitching showing plastic deformation of lateral pads

The discrimination methodology, thanks to sclero-topometric surface metrology, is first depicted on local level of spheroidal nodules in ductile iron, with the measuring of S_v parameter (which is the maximum pit height: depth between the mean plane and the deepest valley) for various oils. This local parameter is not sufficient to characterize completely the interactions between the lubricant and the graphite, that is why a further study on the material displaced volume is done.

If one is interested in influence of interfacial shear conditions on volume of generated trace from sclerometric test as a function of the applied load on the penetrator, it can be experimentally established that surface metrology of superficial rheology of ductile cast iron dominates the phenomenon. The volume of trace penetration (B in Figure 9) is measured over the entire trace. The evolution of the penetrated volume is presented as a function of the normal load (cf. Figure 12). The measured volumes of traces penetrations are quite similar in those experimental conditions. For the low load the volume are identical for the three oils, however small differences appear between the lubricants from a normal load of 10 N. That traduce on the one hand, that the experimental conditions and specifically the choice of the lubricant do not influenced the volume of penetration.

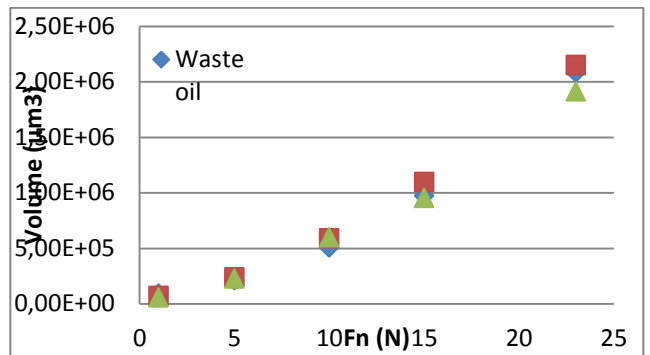


Fig. 12. Evolution of volume of traces penetration (B see Fig.9.) at 10mm/min and under different normal loads (1 to 23 N); with waste, new and regenerated oil

However, the observed differences are not significant enough to allow establishing discrimination between the tested lubricants. On the other hand the shear conditions due to different type of lubricants influenced the laterally displaced (ploughed) material outside of the furrow expressed in volume of $(A_1 + A_2)$ as shown in Figure 13.

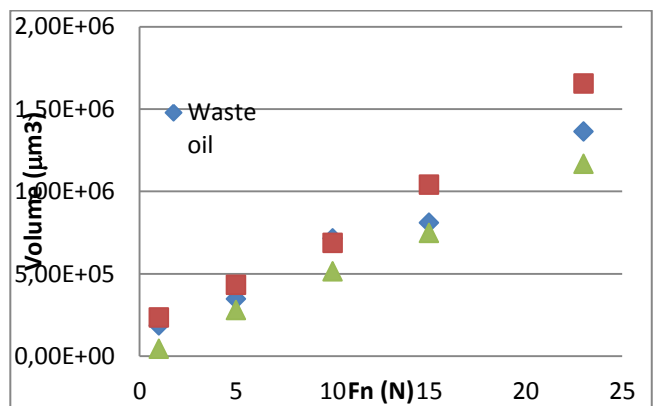


Fig. 13. Evolution of volume of ploughed material ($(A_1 + A_2)$ see Fig.9.) at 10mm/min and under different normal loads (1 to 23 N); with waste, new and regenerated oil

The deformation mode of the ductile cast iron depends on the type of used lubricants. What is really understandable is that new oil reduces more efficiently shear between diamond penetrator and ductile cast iron and throws out more of material than used or regenerated oils. What is astonished is that waste oil induces higher volumetric repels than regenerated one. All the measurements increase discrimination validity, feasibility and reliability of sclero-topometric tests.

5. Conclusions and perspectives

The study of the micr-machinability in case of lubricated polishing process with more or less waste and regenerated oils of functionalize surface [32] is done through sclero-topometric investigations. The developed strategy is well adapted to qualify

the oil influence in very severe conditions of strain and stress states. As it has been shown sclero-topometric characterisation in case of surface properties metrology can be helpful tool for selection and/or regeneration of oils predestined to manufacturing, treatment and lubrication. Indeed the measurements allow the characterization of the tested surface in terms of rheological behaviour.

The developed strategy of surface metrology can be used to determine the approach of regeneration and valorisation taking into account functional aspects of lubricants interacting with the contacting bodies in context of physicochemical, morphological and rheological characterisations with particular focus functionalize surface morphological parameters [32].

Nomenclature

A (1&2)	Parts of lateral section of displaced material,
ADEME	French Agence de l'Environnement et de la Maîtrise de l'Energie
ATSDR	U.S. Agency for Toxic Substances & Disease Registry
B	Part of the scratch penetration
BSE	Back scattered electron
EDX	Energy Dispersive X ray Spectroscopy
EPA	U.S. Environmental Protection Agency
F_n	Applied normal force [N]
F_t	Resulting tangential force [N]
PAHs	Polycyclic Aromatic Hydrocarbons
PCB	Polychlorinated Biphenyls
R	Tip's radius of sclerometer penetrator [μm]
S_v	Maximum pit height: depth between the mean plane and the deepest valley [μm]
SEM	Scanning Electron Microscope
UNEP	United Nation Environment Programme
V	Displacement speed of indenter [mm/min]

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