

On wear Behaviour of a Composite Class with Micro-Nano Adding Particles and PA Matrix

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The paper presents results on wear after testing a class of composites (PA + 1% black carbon and different 5 concentrations of micro glass spheres) and discussions about damaging processes within the superficial layer, grouped in two categories: one favouring the material remove and the other the reduction of wear intensity. The analysis points out that tribological behaviour should be interpreted only by putting together information about wear, friction, temperature in contact, pair of materials in contact in order to obtain sustainable results that could be useful in engineering design.

Keywords: micro-nano particles, polyamide, wear, IR thermogravimetric, SEM

As the tribological performance is not uniquely defined [8, 20, 26], materials with low wear rate and friction coefficient are desired, often as coatings assuring the tribological protection, the support material bearing the load. Unfortunately these materials desired for their tribological properties, do not have properties required by engineer designers as mechanical strength, stiffness, creep strength, thermal and electrical conductivities, low thermal expansion coefficient, low chemical reactivity [6, 9, 27].

Adding materials as fibers and powders substantially improve mechanical and tribo-logical properties [4, 5, 7, 24, 21, 42]. It was reported that adding CuS, CuF₂, CaS, and CaO reduces wear of polyamide and PEEK [1, 2] but others as CuAc and CaF₂ increase the wear rate. More irregular the shape of the particles, the possibility of an intense abrasion increases, decreasing the wear resistance. For this point of view the nanoparticles seem to behave better. Further-more, as the ratio area/volume is greater, this is favourable for improving the bond between particles and the matrix they are embedded into [32, 33, 35]. Incorporating inorganic nanoparticles and fibers generate synergic processes (fig. 1), not entirely studied and explained, upon the tribological behaviour [3, 27].

The concept of improving tribological behaviour of polymeric composites [14, 26] relies on reducing the material adhesion to the mating surface and increasing the hardness, stiffness and compression strength using fibers (aramide [8, 9], glass [34] and organic as PTFE [15]) and inorganic powders (graphite, metallic oxides,

ceramics etc.) [2, 4, 12, 17, 19, 21]. Some researchers appreciate the continuous long fibers are more efficient as compared to the short ones, having a lower wear, but arguments and results are not generally valid, but only for particular tests and applications. One of the mechanisms for reducing the friction coefficient is the generation of a transfer film on the mating surface [1, 25, 30]. Sometimes it is more advantageous to have a softer adding material (like PTFE) in a more rigid polymer and fibers in order to obtain good tribological results. Some adding materials increase the thermal conductivity of the composite (PTFE + bronze) [1, 29] are able to reduce thermal regime. Not all the adding materials are benefic for improving the tribological behaviour of polymeric composites. Wear resistance increases if the adding material reacts and generates products with stronger bond between the transfer film and the mating surface [5, 8, 33], while others decrease wear resistance by generating discontinuities and voids into the material. Thus, it is important to understand initiation, development and degradation of transfer films and their dynamics, these being involved in wear mechanisms and chemical and mechanical interactions of transfer films are complex, interconditioned [10, 22, 30].

The barrier between nano and micro analysis is hard to trace as the efficient manufacturing technologies for the nanocomposites are still expensive at industrial scale; Larsen [15] is one of the specialists admitting that in polymeric composite there could be pointed out adding materials at both scales, nano- and micro-, and the line between these scales is very volatile.

It is noteworthy that the problem with dispersive mixing is that nanoparticles commercially available usually exist as agglomerates [8, 9, 16, 35, 36], difficult to disconnect by the limited shear force during mixing. This is true even when a coupling agent is used, since it can only react with the nanoparticles on the surface of the agglomerates that will maintain their friable structure in the composite and thus, hardly being able to provide improvements.

A class of composites

What is a class of composites? The definition is similar to that found in several references [3, 4, 8, 26, 30], being named as a family or a group of composites, but generally a class of composites is characterized by a set of characteristics kept constant or in a particular range, the

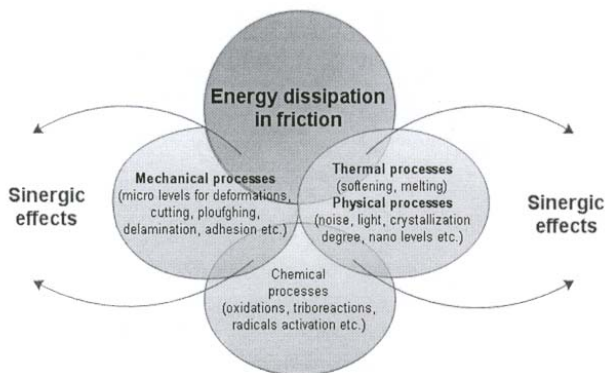


Fig. 1. Interactions among processes generated by energy dissipation in friction

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matrix or / and the adding materials, thus having similar or directioned properties, etc., but also involve processing or similar applications. A class of composite, when analysed, has to have clear criteria to fulfill, in order to be clearly defined and to use the results for directioning the properties of these group of materials or for selecting one or several corresponding better for the taken-into-account application.

Table 1
PROPERTIES OF THE PA MATRIX [23]

Characteristic	Value
Upper service temperature, [°C]	110...180
Thermal conductivity [W/m·K]	0.25
Thermal expansivity, [K ⁻¹]	90·10 ⁻⁵
Tensile modulus [MPa]	3,300
Tensile strength [MPa]	82

The class of composites being the subject of this paper is characterised by [23, 16]:

- polyamide (PA) + 1% black carbon matrix,
- micro glass particles of spherical shapes (abbreviated in this paper with MGS), thus high hardness ratio between the two components of the composites (PA and MGS),
- large dispersion of the adding material (fig. 4) and an investigation on a large concentration range (0% – for the polymer, 2.5, 5, 10, 20, 30, 50%wt MGS) for tribological applications.

New technologies for composite manufacturing are initially modelled with dedicated soft [39], then tested in laboratory equipment, pursuing a future implementation at industrial scale [37, 38, 23, 40].

In polymeric composites the barrier between micro- and nano- is hard to trace as the efficient mixing technology [15] of the constituents is still expensive at industrial scale. Polyamide was selected as matrix in many applications and researches [4, 10, 18, 21, 23, 35] for reasons taking into account its manufacturing availability for being mixed with very different adding materials, for its mechanical, physical and tribological set of properties, but sometimes, polyamide is considered a basis to start a research for improving a particular application, being positioned in comparison with other promising polymers as matrix [8, 9, 10, 28, 36].

The properties of the polymer used as matrix is given in table 1 [23] and SEM images of adding material (fig. 2) reveal a large dispersion range of sphere dimensions, allowing them being named a micro-nano adding mixture.

Even if composite modeling and simulation of their behaviour in different conditions have been progressed [12, - 14, 26, 31, 34], experiments remained necessary as the actually processed in composites are hard to be modeled in such a complex way to approach their actual particular behaviour, especially for tribological point of view.

Experiment methodology

Failure mechanism in composites could be modelled but should be confirmed by experimental work [41]. It is hard to estimate by modelling the consequences of local discontinuities upon tribological behaviour, i.e. the influence of local agglomeration in a non-regular composites as it happened for composites with low concentrations of MGS.

Dry regime [3, 4, 24, 25] has advantages as cost decrease, simplicity, cleanness, easiness for applying, a specific range for work temperature and application where fluid or boundary lubrication is not allowed. Among the problems of dry regime there are the compromise low strength/ stiffness, high friction coefficient and / or wear,

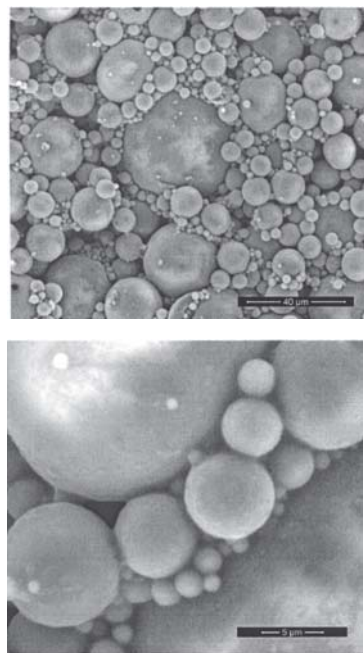


Fig. 2. SEM images of micro glass spheres, pointing out a wide range of dispersion for sphere diameters, from 50 µm to sub-micronic scale (about 400...900 µm)

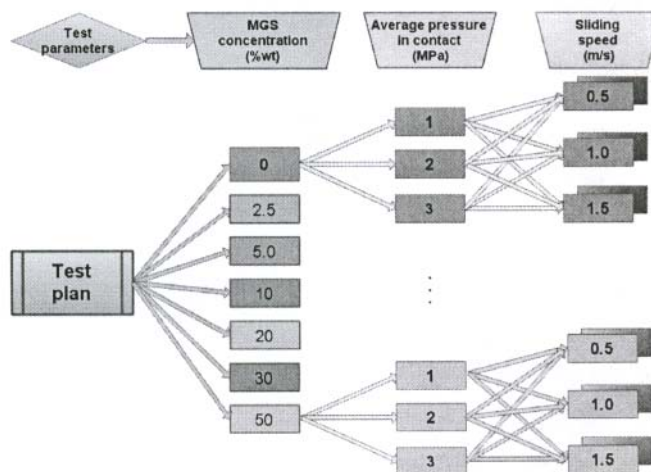


Fig. 3. Test plan [16]

limitations of the environment parameters (temperature and/or composition changes).

Tests were performed on a CETR unit and the surfaces topographies were registered by PRO500 3D (stylus) profilometer in the laboratory of Dunarea de Jos University of Galati. Pins were made of commercially available steel grade 51VMn11 (Sa=0.4...0.6 µm, 40...43 HRC). The test parameters in dry regime were: sliding speed (0.5 m/s, 1.0 m/s and 1.5 m/s), average pressure (1 MPa, 2 MPa and 3 MPa, respectively) and micro glass spheres (MGS) concentrations: 2.5, 5, 10, 20, 30 and 50% (wt), considering 0% for the polyamide + 1% black carbon.

Results and discussions

Wear Analysis

Specialists [8, 20, 24, 28, 34] pointed out that wear mechanisms of polymeric composites are similar to those in metallic materials: micro-cutting, micro-ploughing and (de)lamination of micro-plates (similar to the rippling process on highly loaded metallic surfaces). The presence of adding materials generates less intense wear as micro-ploughing and micro-cutting, but is favourable to generate micropores and microcracks.

Analysing the results obtained on pin-on-disc tests, several of tested composites (PA + 1% black carbon +

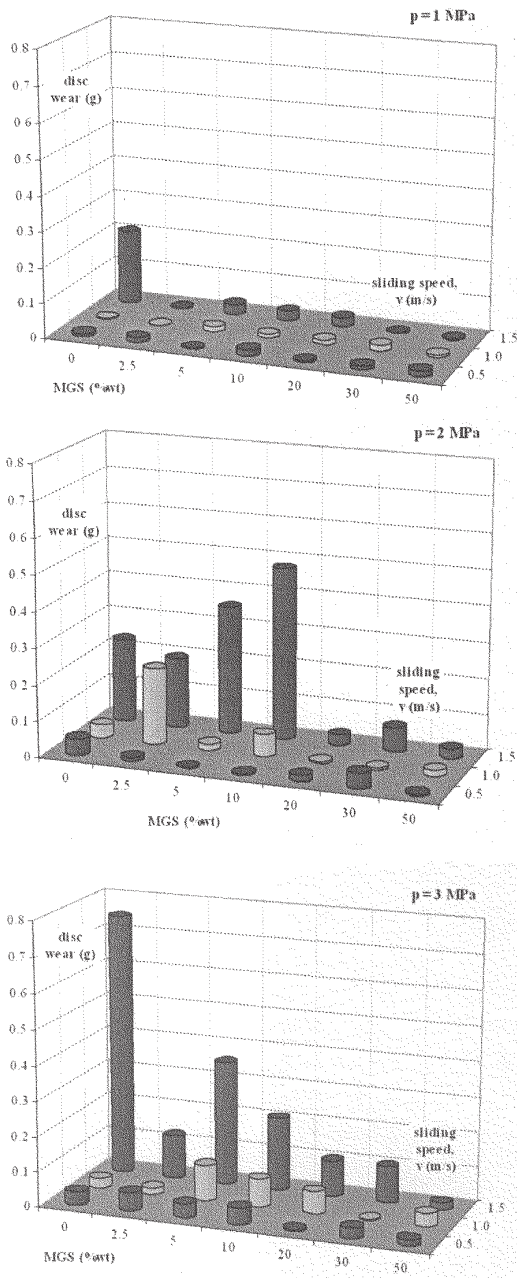


Fig. 4 Wear of the discs

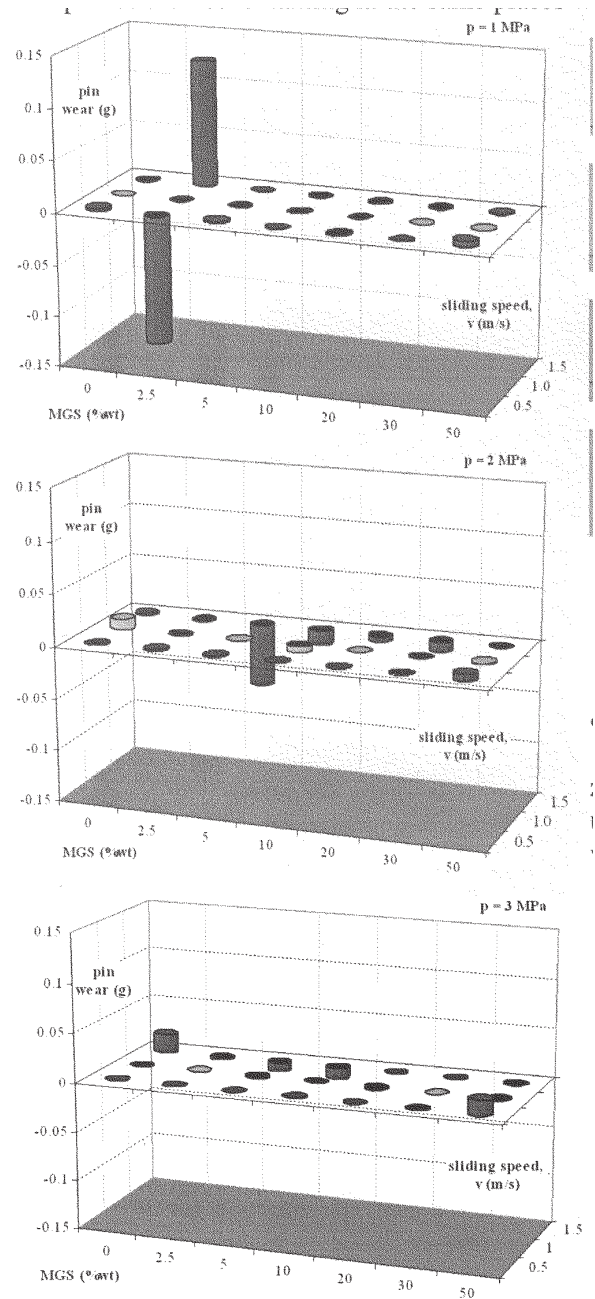


Fig. 5. Wear as mass loss of the steel pin

MGS) may be recommended for sealings and other machine elements with sliding motion.

Wear could be relevant if both elements of the friction couple are analysed by the same wear parameters. Figures 4 and 5 give wear values for both discs and pins. The results given in these plots are the average of two tests done in theoretically identical conditions, including normal load ($\pm 5\%$), sliding speed ($\pm 5\%$), laboratory temperature ($19 \dots 21^\circ\text{C}$) and air relative humidity ($36 \dots 42\%$), sliding distance (10,000 m), the same radius of the pin axle to the disc rotation center. Discs and pins were weighted by the help of a digital balance, the mass of each one have been measured as average of three consecutive measurements, before and after a test.

Pins were weighted as they are after being detached from the tribotester device, meaning that all deposits remained on them are included (figs. 5 and 6). The adherent material on the pins is easy to be detached and pins were carefully handled.

Analyzing disc wear values, the following could be noticed:

- for 1 MPa wear seems to be less influenced by MGS concentration, except the polymer and middle values (5%,

10 and 20% MGS); polymer exhibit 4...5 times higher wear as compared to tested composites;

- at $p=2$ MPa wear remains low only for $v=0.5$ m/s, increasing for $v=1.0$ m/s only for low concentration of MGS, but having high values at $v=1.5$ m/s for the polymer and MGS concentration till 10% inclusively;

- after testing at $p=3$ MPa, wear increases obviously for $v=1.5$ m/s for composites with low and medium concentrations, the polymer having a value 8...10 times higher than that obtained for composites with 20...50% MGS.

Literature had given an optimum value of maximum 10% of adding material for obtaining an acceptable tribological behaviour [8, 21], but for this composite class low wear values were obtained for 20...30% and even 50% MGS. Why? Because processes within the superficial layers are particular for these composites.

The authors consider that the large dispersion of spheres diameters and high concentration of MGS allow fixing the polymeric matrix, among spheres of different sizes, even if these spheres are not remaining in the same places within the superficial layers.

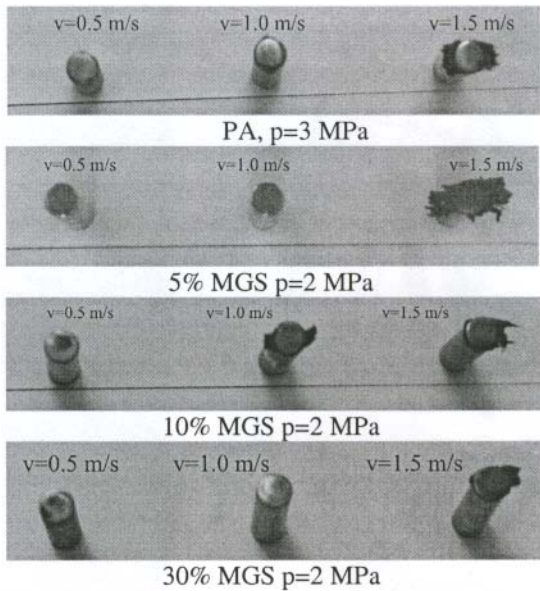


Fig. 6. Images of pins immediately after testing and without removing material either from contact zone or around it (sliding direction is left to right in all these photos)

Temperature monitoring

Monitoring temperature of contact zone by a thermographic camera was a useful method in understanding why wear was so low for higher concentration of MGS. Thermographical images obtained by the help of a thermo-camera and the temperature evolution in 3 points on the

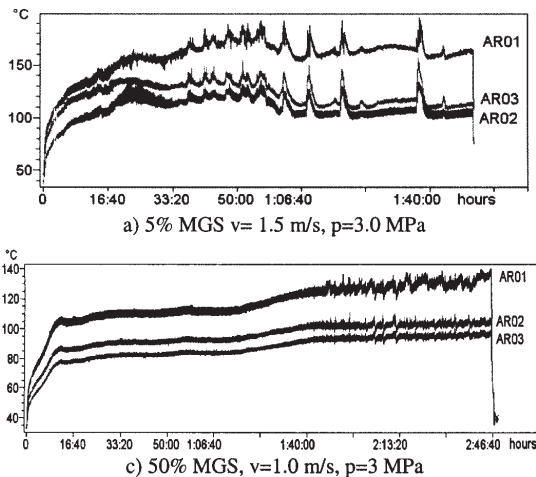


Fig. 7. Images obtained by the help of a thermographical camera

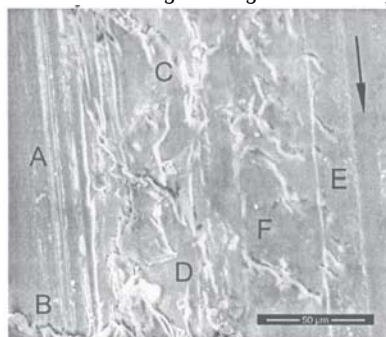
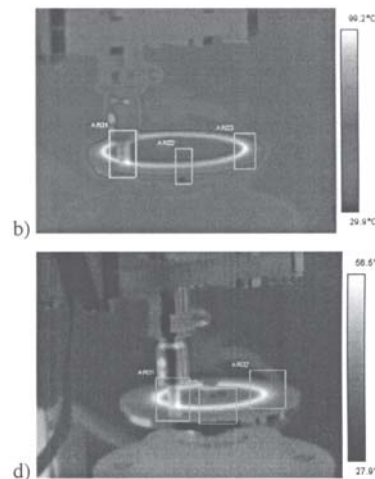
wear track (fig. 7a, c and e), demonstrating that for these test parameters and tribotester (including radius of pin axle to the rotation disc center), the polymer at the surface is at least soften, even melted near the pin contact, validating the model of viscous or melted film for polymeric composite – metallic material, as described in [26], even if this paper deals with “reverse” case of the tri-botester: pin made of steel and disc made of polymer or polymeric composite [16].

In figures 7b, d and f one may notice that temperature near contact are 110...150°C meaning that the polymer matrix is at least soften, but very probably even molted in the contact and that high temperature is maintained high on the wear track (see temperature evolutions for zone AR02 – diametrically opposed to the contact zone and for zone AR03 – at 270° after contact on the circular wear track, fig. 7a, c and e).

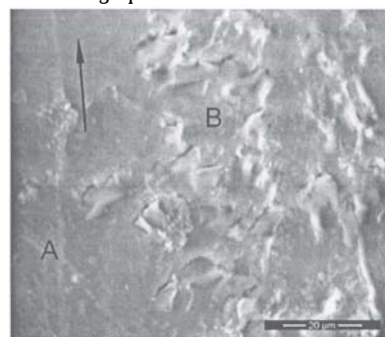
SEM Analysis

Analysing many SEM images of the superficial layers from both discs and pins the authors could identify wear processes characterizing the test regimes. In this paper regime zone means a zone of points (p, v) that had similar evolutions within the superficial layers. Adhesive wear was a dominant component of the damaging processes within the superficial layers especially for the polymer for all sliding speeds and normal load.

The following images are relevant for pointing out particular behaviour characterizing the damaging of the superficial layers of this class of composites. Under each



a) Abrasive and adhesion wear, p=1 MPa, v=0.5 m/s



b) Adhesive wear p=1 MPa, v=1.5 m/s

Fig. 8. Wear on the disc made of PA]

8 a): A – zone with abrasive traces; B – rough edge generated by detaching a polymer volume that was first deformed, laminated and maybe adherent to the mating surface; C – small spots of adhesion; D – a wear debris re-attached to the composite disc; E – zone with fatigue micro-cracks; F – zone with fatigue cracks

8 b): A zone with less “action”, meaning that the contact load is not uniformly distributed; – B – zone with adhesion wear, the polymer being tear-off as micro-flakes

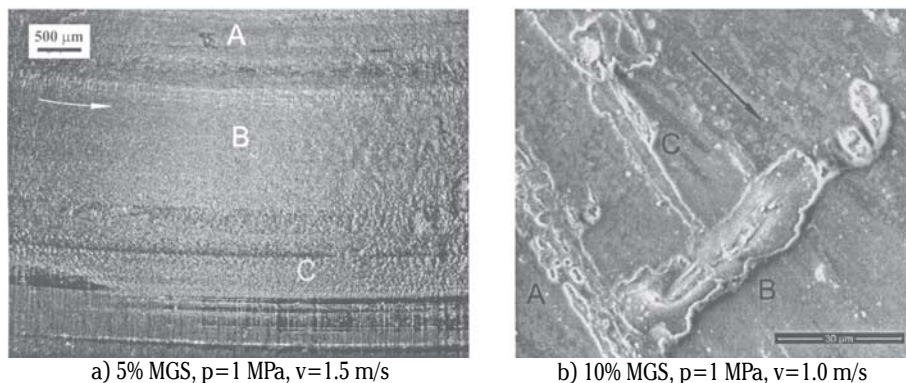


Fig. 9. Delamination process on discs made of composites PA + 1% black carbon + MGS

image they are explained and a letter indicates the local zone on the image where the process is more visible. In analyzing these images one may pay attention that these processes could overlap, or/and they could be dominant only in micro-local areas on the wear trace of the discs or pins.

On the contact track zones with intense adhesion (C and D zones in fig. 8a) alternate with zones with abrasion traces (zones A and E in fig. 8a). From figure 8b one may notice an intense adhesion on zone B (local adhesion, the result being tearing-off small triangular flakes). Analysing these two SEM images one may notice that increasing sliding speed from 0.5 m/s (fig. 8a) to 1.5 m/s (fig. 8b) the processes within the superficial layer of the polymer had dramatically changed.

At $v=0.5$ m/s different wear types pointed out are (fig. 8a): A – abrasive traces, B – rough cut (tear) of a micro-zone already adhered to the pin, C – microzone with intense adhesive wear, D – re-adhesion of the wear debris previously detached and laminated, E – a micro zone with fatigue micro-cracks almost perpendicular to the sliding direction, F – zone with alternating zones of adhesion and fatigue cracking. At $v=1.5$ m/s it was noticed a random alternation of zones with intense adhesion and ones with less intense damage of the superficial layer.

Delamination characterizes the polymer surface but also it takes places when micro-spheres are ploughing the polymer surface when they are pulling by the pin movement. Sometimes when the micro sphere is torn off from the matrix and then dragged in a neighbour zone, the

shape remaining into the polymer is favourable to develop rolling or delamination (fig. 9a and b).

9a): A – spalling, B – intense delamination (similar to the rippling process on metallic surfaces), C – less intense delamination and discontinuously abrasive traces towards the lateral limits of the contact.

9b): A – local adhesion along sliding, B – local deformation caused by pushing a bigger sphere in the sliding direction (the sphere was already drawn in the image), C – delamination and adhesive wear.

Fatigue wear is visible under different aspects: spalling especially for low MGS concentration (fig. 9a) because the dispersion of MGS are not so good and within the composites there are local agglomerations of MGS that are exfoliated when fatigue is enough intense to produce cracks into the polymer matrix that propagate around the MGS conglomerates, finally detaching a macro flack from the surface (similar to the process happening on the thermo-chemical treated steels).

Fatigue is visible as small cracks almost perpendicular to sliding direction, both on discs (fig. 8a) and on transferred films on pins (fig. 13a). Cracks initiated on the transferred film on the pin are growing and at a given moment the transferred film is fragmented into bigger wear debris, this being reflecting in increasing the friction coefficient [16].

The authors also identify a type of tribocorrosion as for higher speeds ($v=1.0$ m/s and $v=1.5$ m/s) near the wear tracks there were small particles of brown colour meaning

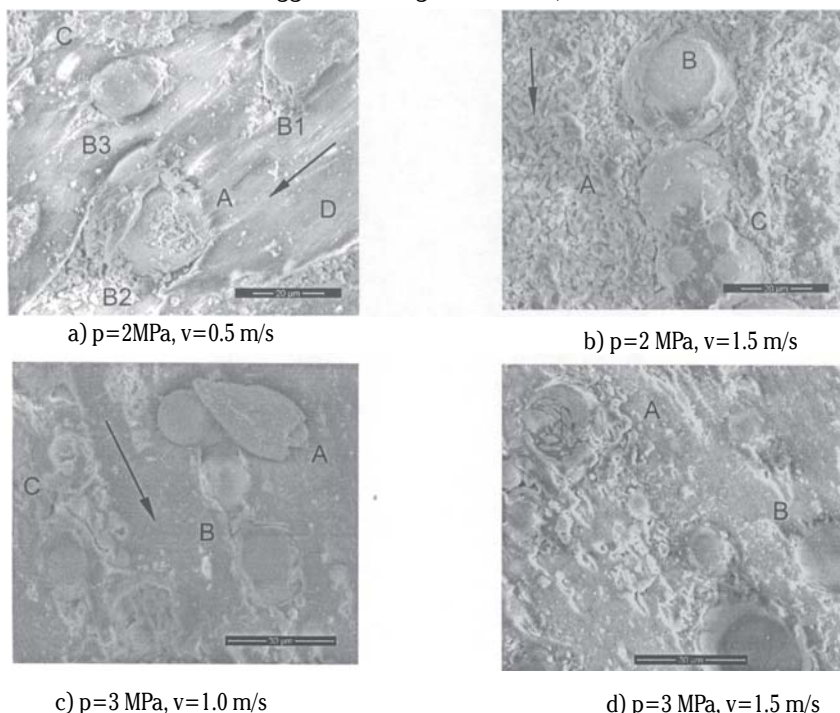


Fig. 10. Third body wear. Detaching small wear debris, very hot that would react with the environment and are stick again on the superficial layer. Tested composite: Pa + 1% black carbon + 50% MGS

a chemical reaction of polymer due to high temperature and due to exposing small particles to air when throwing them out the hot contact. But some of these small particles, even they bear chemical reactions, remain stacked on the wear track, generating the so-called "third body wear" (fig. 10b).

10a): A – the polymer is enough soften to be spread (like butter on bread) on the prominent micro spheres of the surface, B2 and B3 – similar processes (but not identical) in front of the microspheres: the microspheres are pressed and intermittently rolled into the matrix by the normal load and they deform it, generating small cavities in front of the micro-spheres; in time, due to the visco-elastic behaviour of the matrix, they could be partially covered by the polymer, also trapping wear debris previously detached; B1 – the microsphere is pushed into the matrix causing a bump of polymer in front of it

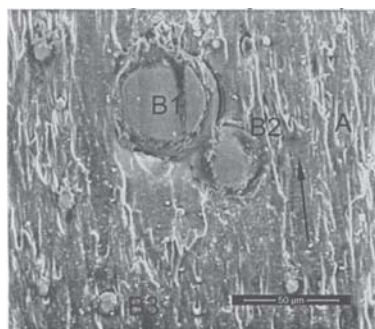
10b): A – very small debris resulted from polymer detaching and lighten colour indicates a change of their composition, especially because of high thermal field, they are previously detached, fragmented, they react with the environment (very probably – polyamide oxidation) but they are stucked to the soften/melted surface, locally generating a "third body friction"; B – bigger spheres are covered by non-uniform thin layers of polymer, a process favouring friction reduction, or with small wear debris pressed in contact; C – an MGS agglomeration due to their relative movement into the matrix surface and/or to their adhesive bonding among them due to the very thin films attached to them

10 c): A – a rolled conical wear particle, with very small MGS remained into it; B – the polymeric layer on the surface is viscous enough to be spread both in the sliding direction and laterally to cover the bigger spheres; C – very intense fragmentation and deformation of the polymer layer due to a local MGS agglomeration

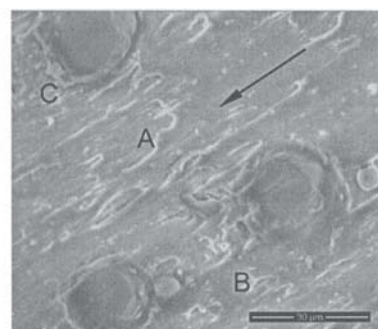
10 d): A – a bigger sphere that was rotated into the superficial layer and it was repeatedly covered with thin layer of polymer; when the sphere is again loaded, this layer is fragmented (if it has the conditions of becoming solid) or it becomes thicker by covering the previous one with a new molted thin layer.

Viscous or/and melted film friction was pointed out by the help of SEM images, but also this model of friction is also sustained by the high temperatures around contact and high temperatures (but 10..20°C lower than those around the contact) on the whole wear track on the composite discs (fig. 7), these values being enough to maintain a soften or even melted thin polymer layer (especially in contact).

It was noticed a particular process of rolling the bigger spheres into the soften matrix, thus they being covered with thin polymer "blankets", process that allows reducing friction as hard surfaces of spheres are not get into direct contact with the hard surfaces of the pins (figs. 10 and 11). When surfaces leave the contact, they become colder, the thin polymeric layers are solidified and when they get again in contact these thin blankets are broken, fragmented, but new viscous thin films adhere on the spheres and the final aspects are those in figure 10a.



a) 20% p=2 MPa, v=1.5 m/s

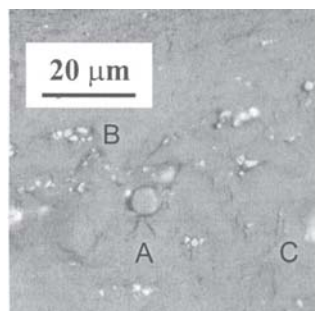


b) 10% MGS, p=3 MPa, v=1.5 m/s

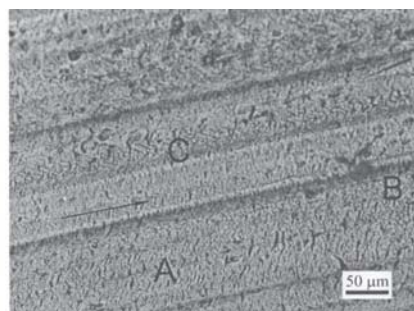
Fig. 11. Viscous or/and melted film friction

A –zone with melted layer of very small thickness (maybe sub-micronic) but enough to change tribological behaviour; B1 and B2 – microspheres embedded into the soften matrix and covered with thin layers of polymer

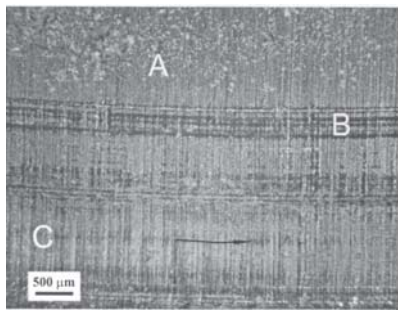
A – zone with local thin melt polymer due to testing regime; B – micro-spheres of different sizes make the polymer to non-uniformly flow among them; C – the polymer shape in front of microsphere demonstrates the microsphere complex movement of sliding and intermittent rolling.



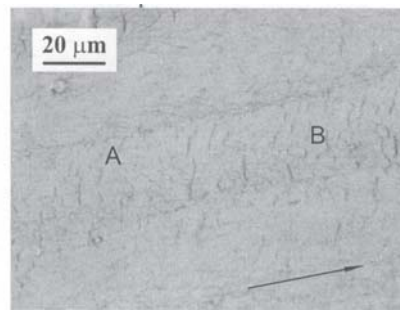
a) 2.5% MGS, v=0.5 m/s, p= 1 MPa



b) 2.5% MGS v=1.5 m/s, p =1MPa



c) 5% MGS $p=1$ MPa, $v=0.5$ m/



d) 5% MGS, $v=1.0$ m/s, $p=1$ MPa

Fig. 12. Images of the wear tracks, obtained by the help of an optical microscope

12a): A – cracks produced in the polymeric matrix due to fatigue (the microsphere is repeatedly loaded, temperature field is low due to the low sliding speed); B – agglomerations of MGS revealing that dispersion is not of good quality at such a low concentration of MGS; C – traces of abrasion and lamination.

12b): A – thermal fatigue of the superficial layer that could be recognized by the narrow and deep micro-cracks perpendicular to the sliding direction; B – abrasive trace and pits of different sizes denoting that MGS were torn-off; C – locally flows of the polymer revealing maybe

intermittent adhesion with the counterpiece.

12 c): A – wear debris laterally thrown out from contact, their lightened colour evidencing chemical reactions due to high temperature and greater surfaces exposed to environment; B – abrasive wear; C – central zone of wear track with local, alternative zones with lamination along sliding direction.

12 d): A and B - local melting, rolling, fatigue with different intensities; the image also reveals that dispersion is not of good quality as MGS are nor visible on the entire image.

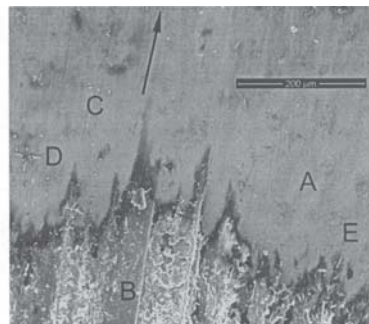
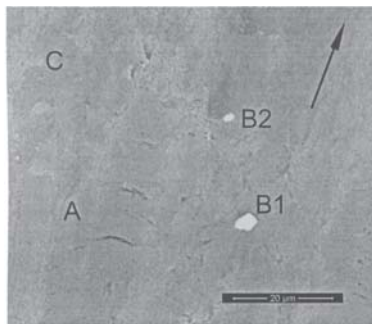


Fig. 13. Pin after the test 20% MGS $v=1.0$ m/s, $p=3$ MPa

a) central zone of a pin; A – micro-cracks in the transferred layer on the steel pin, pointing out that fatigue occurs on the transferred film too; B1 and B2 – MGS of different size transferred together with the polyamide; C – this zone evidences overlapped transferred layers, as transfer was non-uniform and repeatedly produced

b) in front of the pin; A – smooth zone due to the polymer; B – fragments detached from transferred layer are pushed in front of the pin, being obviously adhering, laminating and rolling processes with different intensities; C – an agglomeration of very small MGS into transferred layer; D – another agglomeration of MGS; E – fragmentation of successively transferred layers

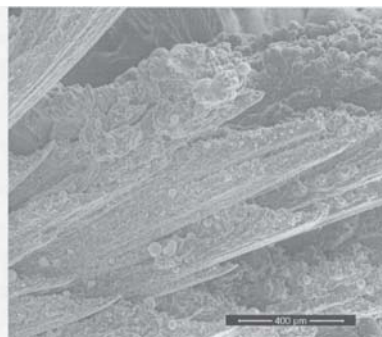
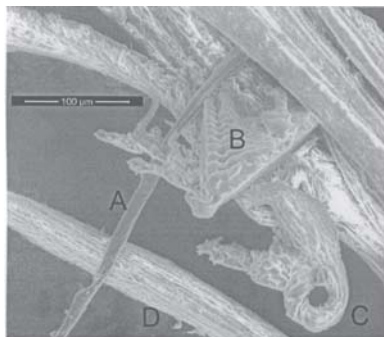


Fig. 14. SEM images of wear debris. Same testing conditions: $p=1$ MPa, $v=1.0$ m/s

a) wear particle from a disc of PA + 1% black carbon: A – evident rolling, B – lamination, C – curling due to rapid cooling in air, D – a wear particle of thin yarns partially rolled, obtained by mechanical interlacing and bonding due to high temperature; particles are longer, as compared to those obtained from composites

b) detailed of a wear particle detached from a disc made of PA + 1% black carbon + 20% MGS; due to very small MGS the particle is more rigid, the rupture zone has a brittle aspect and rolling are visible only in small isolated zones; wear particles have brittle and similar aspects for almost all tested composites

The transferred film on the pin contact zone is quite thick (fig. 13) and fatigue cracks perpendicular to sliding direction are developed. There are only few very small MGS that were transferred together with the polymer; the transfer film lost a large quantity of black carbon as its colour is more lightened. In front of the pin there are polymeric irregular deposits showing both adhesion and abrasion forms; wear debris are detached and then captured again.

Conclusions

After studying the particular processes taking place within the superficial layers of discs and transferred films on the steel pins, the authors could group them into two categories:

- characteristics favouring the mass loss:
 - thermal and mechanical characteristic of the matrix (but this could be also an advantage as it allows the tribosystem functioning with viscous or melted films, as given in a model described in [26], (figs. 9 and 10),
 - at low MGS concentration, clusters or agglomerations of MGS cause local spalling (fig. 9a),
 - small particles are evacuated in small wear debris outside the wear track (fig. 12d),
 - high sliding speeds for polymer and composites with low MGS concentrations (fig. 4);
 - characteristics favouring the wear reduction:
 - the large dispersion of MGS does not allow the bigger spheres being taken off (pull out) from the superficial layers,
 - the spherical shape of adding material allows complex movements into the polymeric matrix of the MGS (fig. 10), without increasing friction as in the case of micro-fibers,
 - a middle value of the MGS concentration (20 and 30%wt) and even 50% for high sliding speeds, but having a higher friction coefficient [16],
 - a particular type of three-body friction (as described in [11, 28]), as small wear debris are captured and re-captured among the bigger MGS (fig. 10b and d),
 - no micro sphere was noticed as been broken, fractured or hardly scratched as compared to composites with polymer matrix but filled with fibers [5, 8, 9, 26],

Introducing the notion of class of composites this exploratory research could be focused on evaluating changes in tribological behaviour that would be used for selecting an appropriate material from a class of materials with numerous components.

The test results validated the model with viscous film but further studies have to be done as the composite or even the polymer loss is, qualitatively speaking, not so high as evaluated when testing pin made of polymer or composite on steel disc, as reported in [28].

Wear results are only a component of the tribological behaviour of a tribosystem, thus it has to be analysed together with other parameters, including friction coefficient and thermal fields, during all time interval that should be close to the possible application one.

The advantage of "scanning" by tests a large range of concentration for the adding material consists of knowing how one or a set of tribological parameters could evaluate when this concentration varies. Tests on a large sliding speed range could also reveal processes that could be directioned in solving design for current applications, as for instance using composites with high concentration of MGS at elevated temperature for sealing or guiding, having low friction and wear, even in dry regimes.

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