Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 15
ISSUE 1-2
March-April
2006

New approach to study tribological properties of polymer materials. A case of car windshield wipers

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Received 15.11.2005; accepted in revised form 15.02.2006

Methodology of research

ABSTRACT

Purpose: Existing approach to tribology of polymer materials does not take into account their composition and structure. This paper presents the original analysis of friction characteristics, enabling an insight from the material engineering point of view.

Design/methodology/approach: Apart friction also dissipation energy spectra have been analyzed. For transformation of friction fluctuations from the force into the frequency domain the FFT methodology has been applied. Parameters for evaluation of friction and wear have been proposed. Results of bench tests have been verified by examinations of wiper blades under conditions simulating real exploitation, adapting a system of car window cleaning. Power consumption by a driving engine has been monitored.

Findings: Various kinds of modifications, influencing composition and structure of polymer materials have been studied. Ageing and wear resistance have been found to be the most important from the point of view of working properties of blades. They influence the efficiency of water removal from a car window.

Research limitations/implications: Different blade designs have to be checked working under various conditions.

Practical implications: A correlation between tribological properties and structure of polymer materials has to be taken into account at the stage of their compounding and processing. The proposed method for testing car windshield wiper blades is more appropriate than standards used so far, enabling quantitative assessment of products.

Originality/value: The paper helps to understand materials engineering aspects of tribology. It is of potential value for producers of polymer parts.

Keywords: Resistance; Working properties of materials and products; Rubber; Materials design; Car windshield wipers

1. Introduction

Polymer materials, among them rubber, are multicomponent and multiphase systems. Their morphology depends on several factors, both of material engineering and technological nature [1]:

1. type of polymer matrix and its molecular and physico-chemical characteristics,
2. type of filler, size distribution and surface activity of its particles,
3. type of curing system and parameters of network being formed,
4. overall mix composition, and
5. efficiency of mixing (deciding mix homogenization and / or degree of filler dispersion and uniformity of its distribution).

Very often the problem of relationship between morphology of a system and physical properties of polymer materials is underestimated or even neglected. However, heterogeneity of material, connected with non-homogeneous mixing or segregation of its components, non-uniform dispersion of fillers, distribution

Table 1.
General composition of polymer materials being studied at the first stage

No.	Component	Mix no. 1	2	3	4	5	6	7	8
		Content [phr]							
1.	Polymer matrix (NR[1])	100	100	90	80	100	100	100	100
2.	Polymer matrix (EPM[2])	0	0	10	20	0	0	0	0
3.	Active filler (HAF[3])	0	36	0	0	0	0	0	9
4.	Inert filler (MT[4])	36	0	36	36	36	36	36	27
5.	Zinc oxide (ZnO)	10.8	10.8	10.8	10.8	10.8	10.8	10.8	10.8
6.	Curing system[5] (activators, accelerators, sulphur)	6.1	6.1	6.1	6.1	6.7	6.6	6.1	6.1
7.	Environmental protecting agents[6] (chemical and physical)	5.1	5.1	5.1	5.1	3.1	5.1	5.1	5.1
8.	Processing aids[7] (combination of salts of fatty acids and waxes)	0	0	0	0	0	0	1.5	0
9.	Internal lubricant[8] (FG, perfluoropolyether)	0	0	0	0	0	0	0	1

[1] natural rubber
[2] ethylene-propylene copolymer
[3] high abrasion resistance carbon black
[4] thermal carbon black
[5] composition of stearic acid, sulphenamides (CBS), thiurams (TMTD) and sulphur (S)
[6] composition of amines and waxes
[7] Aktiplast or Aflux (Rhein Chemie, Germany)
[8] Fluorogard (DuPont, USA)

Table 2.
General composition of polymer materials being studied at the second stage

No.	Component	Mix no. 1	2	3	4
		Content [phr]			
1.	Polymer matrix (NR[1])	100	100	100	90
2.	Polymer matrix (EPM[2])	0	0	0	10
3.	Active filler (HAF[3])	3	6	6	6
4.	Inert fillers (MT[4]+graphite)	33	30	30	36
5.	Zinc oxide (ZnO)	10.8	10.8	10.8	10.8
6.	Curing system[5] (activators, accelerators, sulphur)	6.1	6.1	6.1	6.1
7.	Environmental protecting agents[6] (chemical and physical)	5.1	5.1	5.1	5.1
8.	Internal ubricant[8] (FG, perfluoropolyether)	0	0	1	0
9.	Chlorine stabilizer[9] (Zn-Ca salts of fatty acids)	4	4	0	0

[1] natural rubber
[2] ethylene-propylene copolymer
[3] high abrasion resistance carbon black
[4] thermal carbon black
[5] composition of stearic acid, sulphenamides (CBS), thiurams (TMTD) and sulphur (S)
[6] composition of amines and waxes
[8] Fluorogard (DuPont, USA)
[9] Stabiol CZ (Cognis Corp., Germany)

profile and various structure of crosslinks, influences exploitation performance of engineering parts and goods e.g. tyres. It is not only of economical but also of ecological aspect. It has been calculated, that each year, only in Europe car tyres generate about 2 mln tons of rubber debris [2].

Till to now, the rheological "stick-slip" mechanism of friction, proposed by Schallamach [3] and its further development, elaborated by Moore [4] have been commonly accepted. They represent approach to tribology of polymer materials from the point of view of mechanical engineering. Based on our previous papers [5-7 and other literature studies 8-10] on morphology of polymer systems, especially devoted to phenomena of the surface segregation, filler agglomeration, as well as gradient of structure and density of crosslinks, we propose to analyze tribological characteristics of rubber from the point of view of material engineering. New approach to analyze tribological characteristics attempts to correlate experimental data with morphology of polymer materials. It showed to be more indicative in comparison to commonly used: coefficient of friction or abrasion resistance. Original parameters for evaluation of polymer compositions are proposed and discussed on an example of materials used for blades of car windshield wipers.

2. Experimental

2.1. Materials and modification

General compositions of polymer materials, designed for blades of car windshield wipers, at the first stage of our studies, are given in Table 1. They differ according to components constituting polymer matrix, kind and amount of fillers and a curing system. Details have not been provided, because of potential commercial application of the recipes.
Polymer mixes were prepared with a laboratory two-roll mixer (David Bridge Ltd., UK). Samples for further studies were cured in a steel mould at 160 °C during the optimum time (t_{90}) determined rheometrically (WG-05, Metalchem, Poland), according to ISO 3417.

At the second stage of our studies, further modifications to the compositions given in Table 1 were made. An amount and a kind of filler were optimized carefully, trying not to deteriorate the resistance of material to cut growth (ISO 133). Apart this, a commercial chlorine stabilizer was introduced to the mixes (did not concern the mixes no. 3 and 4, for which the surface segregation of components: the internal lubricant (FG) or the ethylene-propylene copolymer (EPM), protecting the base material from ageing was expected) in order to prevent the material from chlorine release after a post-curing surface chlorination. Compositions of the second series of mixes are given in Table 2.

Apart playing with composition of polymer materials (bulk modification) also the effect of a post curing chemical or physical treatment was studied.

Chemical modification

Some polymer compositions (see the above) were subjected to post curing chlorination, realized by immersion of wiper blades in chlorinating medium (water solution of sodium hypochlorite and sodium chloride of the total chlorine concentration of ca. 2 g/l) for the period 20–40 min. The degree of modification was controlled by determination of chlorine concentration in the bath.

Physical modification

The same polymer compositions containing carbon black, instead of chlorine treatment were subjected to simultaneous action of UV radiation and ozone, generated by a mercury lamp of 700 W of power, installed in a closed chamber of the volume of 0.05 m^3. The surface of polymer material, placed about 100 mm from the source, was treated with a radiation dose of 1 kW/m^2. The effect of modification carried out 1–24 hrs was studied.

2.2. Techniques

Composition of the surface layer

Experiments were carried out with a Bio-Rad FTS 175C FTIR spectrometer (Germany), equipped with a Split-Pea internal reflection spectroscopy (IRS) microscopic accessory (Harrick Scientific, USA), probing the surface layer of polymer materials with Si crystal. Spectra were run in the wavelength region 600-4000 cm^{-1}. Each time 64 scans with resolution of 4 cm^{-1} were collected at ambient temperature. In order to follow chemical changes to the surface of polymer materials accompanying modification, the characteristics absorption peaks originated from:
- chlorine or fluorine species,
- aromatic styrene ring,
- aliphatic CH_2 and CH_3 groups, or
- products of oxidation

were analyzed.

Microscopic examination

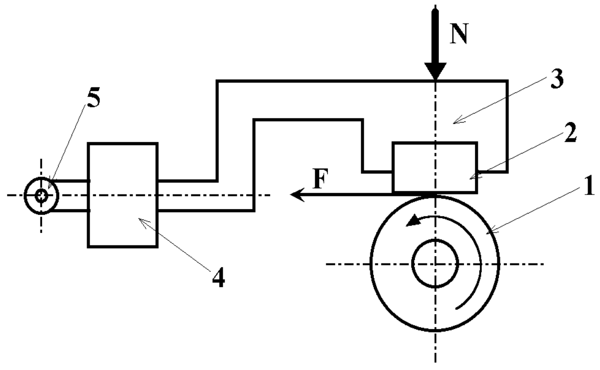
Quality of strip edges of car windshield wiper blades were examined with a Leica MZ6 (Switzerland) stereoscopic microscope under 40× magnification. Roundness and roughness of the edges were compared between virgin and exploited staff.
Additionally, in the case of chlorination and UV treatment the surface of polymer material was checked for presence of deep microcracks, disqualifying the modification.

Micromechanical properties

Hardness and mechanical moduli of polymer materials were determined with a Micromaterials Nano Test 600 apparatus (UK), applying the procedure of spherical indentation with 10 % partial unloading. R=5 μm stainless steel spherical indenter probed the surface layer of material with the loading speed of dP/dt=0.2 mN/s, reaching depths up to 6000 nm. More information on the instrumentation can be found elsewhere [11].

Evaluation of friction characteristics of materials

Friction was determined with a block-on-ring T-05 tribometer (ITeE, Poland) – Fig. 1.



1–specimen (ring), 2–counterface (block), 3–holder, 4–force sensor, 5–joint

Fig. 1. Scheme of the T-05 instrument

The ring of 35 mm in diameter (1), made of polymer material rotated against the flat block (2), made of glass, with the rotational speed of n=1.0 rps (equivalent sliding speed calculated for the contact surface was v= 12 cm/s) and the normal load of 10 N, at ambient temperature (20^{+5} °C). The tribometer was equipped with a multi-channel electronic PC measurement unit – Spider 8 (HBM, Germany) for data acquisition. During experiment lasting 2 hrs the friction force was collected with the frequency of 1200 Hz in 2000 scans, each containing 4096 experimental points. Example of the friction trace together with determined parameters are presented in Fig. 2.

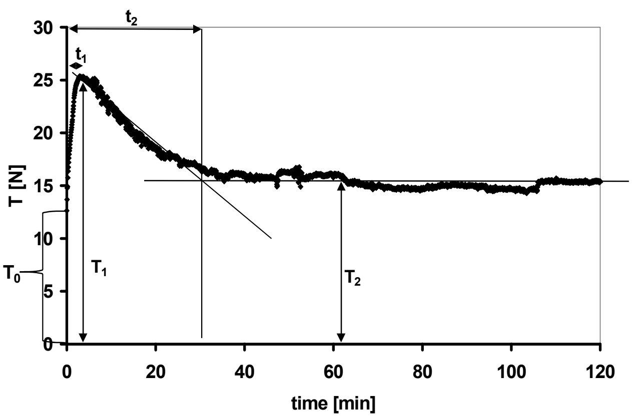


Fig. 2. Typical "dry" friction characteristic of the materials studied

The following parameters were calculated:
T_0 – starting value of the friction force; T_1 – maximum value of the friction force; t_1 – period of time before abrasion starts; T_2 – the force during "stable" friction; t_2 – time for stabilization of the friction force.

Friction characteristics were transformed into frequency domain 0-600 Hz, applying the Fast Fourier Transformation to friction force fluctuations. Discrete levels of energy, dissipated by polymer samples during friction, were determined [12]. Example of energy characteristics for polymer material is given in Fig. 3.

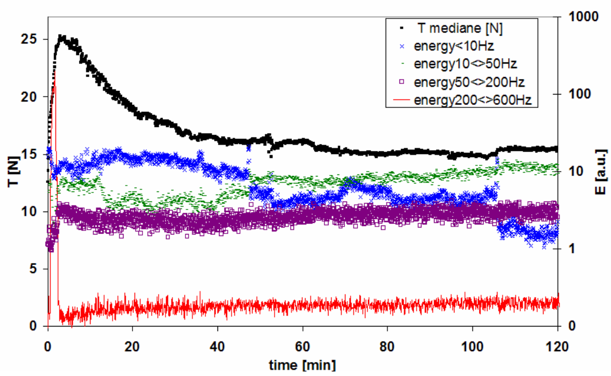


Fig. 3. Typical "energetic" transformance of the "dry"friction characteristic for the materials studied

The high frequency spectrum showed to be the most indicative for abrasion, despite the highest part of energy being dissipated in low frequency deformations. The sharp peak visible on the energy spectrum is placed a little bit in front of the friction force maximum, indicating on the beginning of abrasion process.

Evaluation of performance of car windshield wiper blades

A heavy duty Mercedes motor for windshield wipers was driving the arm (1) working on the rear window (2), both taken from a Daewoo Lanos. Together with a commercial garden sprinkling system (3), adapted to cooperate with the system, they were controlled by computer, working under an elaborated test procedure. Picture of the stand is presented in Fig. 4.

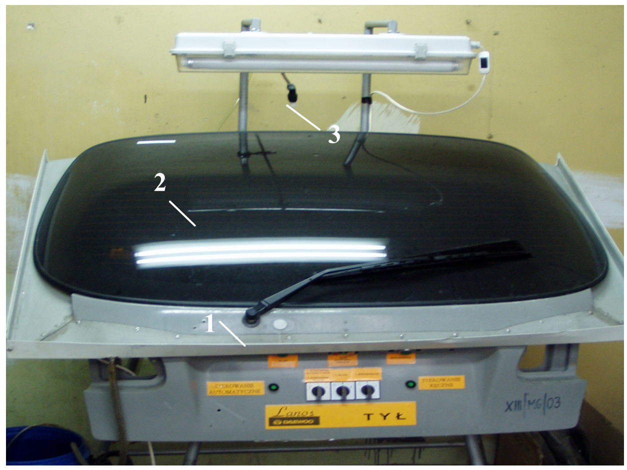
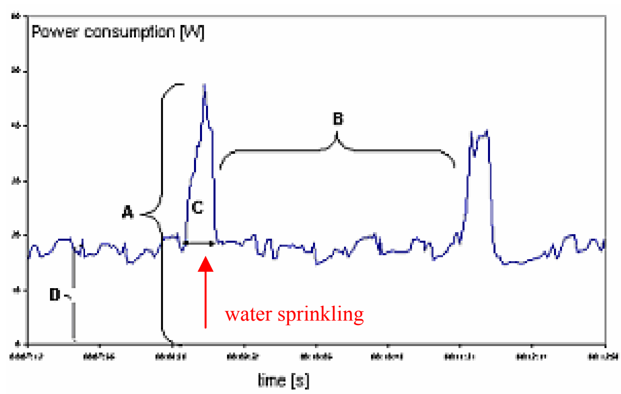


Fig. 4. Picture of the stand for evaluation of exploitation behaviour of car windshield wipers (see the text for figures)



A-amplitude of power consumption during "dry" friction no water lubrication),
B-period needed to water removal from a car window,
C-period of "dry" friction (water completely removed)
D-level of power consumption during "wet" friction

Fig. 5. Typical exploitation characteristics pattern of the car wipers studied (FWS – every 20 s; FAR = 0.5 Hz)

Characteristics of the motor power consumption during time, characterizing behaviour of windshield wipers under designed (software) and computer controlled exploitation conditions:
- frequency of water sprinkling (FWS), and
- velocity of a wiper (determined by frequency of the driving arm rotation - FAR)

were registered.

Fragment of a typical short time exploitation characteristic of a car windshield wiper, together with parameters proposed for its evaluation are presented in Fig. 5.

Apart a short time analysis (0.5 h), the car windshield wiper blades were also run applying a longer test, lasting ca. 36 hrs.

3. Results and discussion

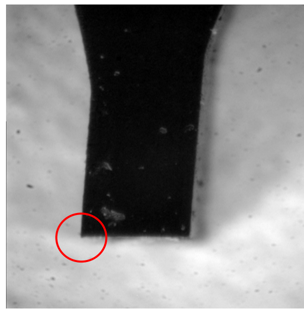
Requirements for car windshield wipers concern effective removal of water, snow, dust and dirt from a car window in order to maintain good visibility for a driver, what is prerequisite from safety reasons. It is desired to be achieved no matter atmospheric conditions, temperature and speed of driving. However, the market additionally demands from windshield wipers to work effectively for at least one year (one season in the case of special applications), simultaneously expecting reasonable, low price. Despite many patents in the area of design of car windshield wipers, shape of blades practically has not been changed very much since many years. Base of a blade, being mounted in a driven arm, used to be connected with a strip – being in frictional contact with a glass window, by a narrow bridge, enabling the latter to maintain the contact when direction of movement changes. Optimal construction makes challenge for material engineering. Material for blades of car windshield wipers should:

1. preserve elasticity in the broad range of temperature, enabling continuous contact with a glass counterface of various curvature,
2. have hydrophilic surface, as well as,
3. preferably low friction and resistance to abrasive wear, especially under "dry" conditions of duty against glass.

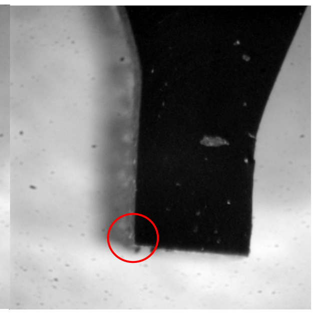
These demands are very difficult to fulfill, especially when have to be combined with low cost, requiring cheap materials and simple technology of manufacturing to be applied. Infrared (FTIR) and thermogravimetric (TG) analyses of various car windshield wiper blades available on the market revealed that more than 90 % of all products are made of sulphur vulcanizates of natural rubber (NR) or its mix with styrene-butadiene rubber (SBR) usually filled with ca. 30-40 phr of low activity carbon black. In order to solve the problem of hydrophobic character of hydrocarbon rubber more than 70 % of the examined samples revealed application of post chlorination to the surface of blades. Among the analyzed staff a windshield wiper of graphitized strip was also present. This kind of modification was undoubtedly applied to reduce friction. In order to improve exploitation properties of car windshield wipers we decided to analyze possible mechanisms behind their failure, looking for the best way of modification.

3.1. Abrasion resistance of material

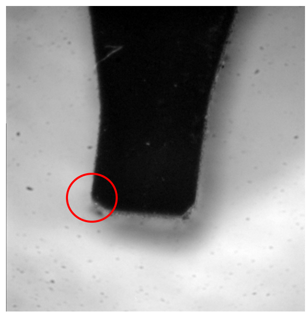
Windshield wipers which cannot effectively remove water from a car window, leaving traces of water or mist, were examined with an optical microscope. Special attention was devoted to edges of their strips, which should be "sharp", assuring good contact with a cleaned glass – Fig. 6.



A -"good", new wiper (sharp edges)



B -"bad", new wiper (rough edges)



C -"bad" old wiper (round edges)

Fig. 6. Abrasion or damage of sharp edges of wiper strips

The pictures above clearly indicate either on roughness (B) or roundness (C) of strip edges to be responsible for failure in performance of the car windshield wipers being examined. The former is associated with damage, the most likely happened to a strip during its removal from a mould (a technological background), whereas the latter is a result of material abrasion (a consequence of exploitation).

In order to improve abrasion resistance of the base material its recipe was modified – Table 2. A part of low activity carbon black (MT) was replaced by a high abrasion resistance (HAF) one. The amount of HAF had to be individually adjusted for the given polymer matrix in order to preserve overall elasticity of the material. Proper balance between amounts of MT and HAF grants durability of a flexible bridge and good, continuous contact between strip edges and a car window, as well as low abrasion resistance of polymer material. Tribological effects of modification are illustrated by some examples of friction and energy characteristics of the materials – Fig. 7, 8.

Partial replacement of MT by HAF carbon black significantly lowered the coefficient of friction due to reduction of energy dissipation, especially the part of high frequency (>50 Hz), by the polymer material. Stress generated by low frequency deformations is less likely to accumulate to the level dangerous from the point of view of material cohesion, producing wear.

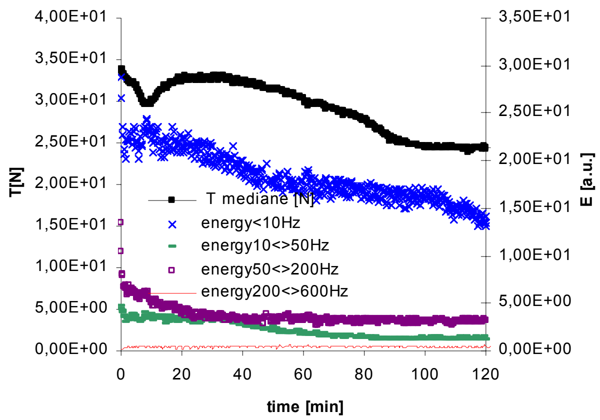


Fig. 7. Friction and energy characteristics of the base material (Table 1/mix no. 1), subjected to "dry" friction

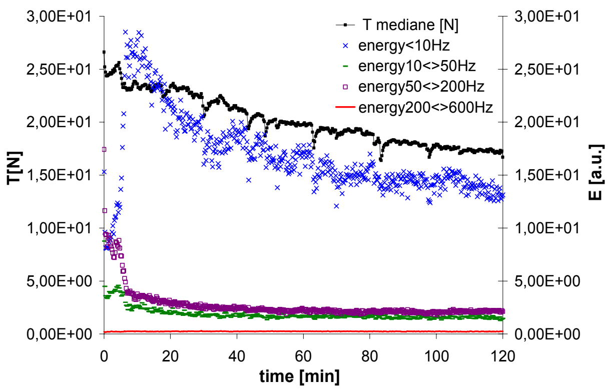


Fig. 8. Friction and energy characteristics of the modified material (Table 2/mix no. 3), subjected to "dry" friction

3.2. Effect of chemical modification

Apart an increase of water wettability and ageing resistance, the most popular chlorination produces surface hardening of material, as indicated by microindentation data – Fig. 9, what facilitates lowering of friction. Domination of the hysteretical component of friction over the adhesional one for elastomers, was reported in our previous work [13].

However, protective action of the modification decreases with time of exploitation, due to release of chlorine atoms from the surface layer of polymer material, as indicated by chemical microanalysis. It produces bulk brittleness of the material, what results in increase of its abrasion, leading eventually to loosing ability of water removal by a car windshield wiper.

Simultaneous application of UV radiation and ozone was reported in the subject literature [14] as a very effective way to increase polarity of the surface of hydrocarbon polymers. Apart this, the modification produces significant lowering of mechanical parameters of the surface layer of polymer material – Table 3. H/E ratio decreased what points on softening of its surface. Macroscale examination did reveal "smoothening" and elimination of characteristic surface tack, what could be responsible for improvement of exploitation performance of the material. It seems likely that graphitization of the surface layer took place [15], as indicated by preliminary Raman spectroscopy studies.

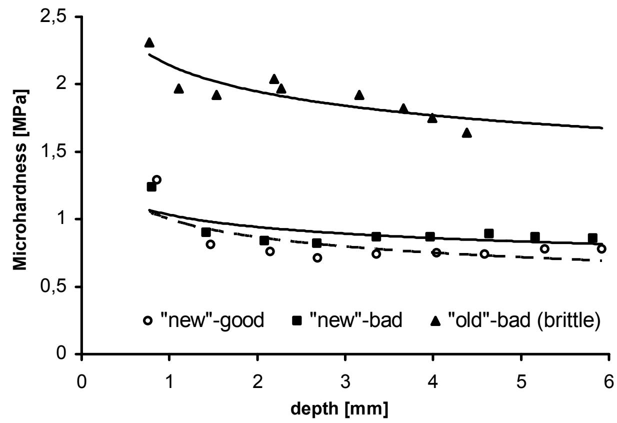


Fig. 9. Hardness profiles of materials subjected to the surface chlorination

Tribological effects of the modification are illustrated by friction and energy characteristics of polymer material – Fig. 10.

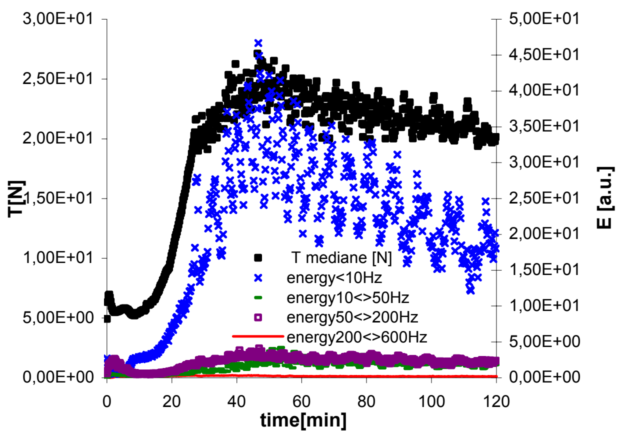


Fig. 10. Friction and energy characteristics of the materials exposed to UV radiation (time of treatment 4 hrs), studied under "dry" conditions

However, probably due to low abrasion resistance of the modified layer the effect showed limited durability under too severe "dry" test conditions. More encouraging results were obtained for "wet" test conditions. This way of the surface modification of car windshield wiper blades is protected by the Polish patent declaration.

Table 3.
Micromechanical properties of the surface layer of polymer material subjected to UV radiation

No.	Mechanical properties	Time of treatment						
		0	15 min	30 min	1 h	2 hrs	4 hrs	14 hrs
1.	Microhardness, H [MPa]	8.0	7.5	6.5	6.0	4.0	3.0	4.0
2.	Young modulus, E [MPa]	25.5	28.0	28.5	28.0	17.0	16.0	22.0
3.	H/E	0.31	0.27	0.23	0.21	0.24	0.19	0.18

Table 4.
Parameters of power consumption during short (0.5 h) exploitation test determined for some car windshield wipers studied

No.	Material	Power consumption during "wet" friction, D [W]			Amplitude of "dry" friction, A [W]	Peak area, C [W×min]	Time for removal of water, B [s]
		median	average	deviation			
1.	Table 1 / mix no. 1	20.4	20.0	2.8	35.7	1.23	23
2.	Table 1 / mix no. 2	18.1	18.4	2.6	27.1	0.80	18
3.	Table 2 / mix no. 3	17.5	17.3	1.9	22.9	0.40	18
4.	Table 2/mix no.1 /chlorinated	15.8	15.9	1.3	21.0	0.40	12
5.	Table 2/mix no.2 /chlorinated	18.5	17.6	2.1	22.2	0.40	14
6.	Table 1 / mix no. 8	17.1	17.2	2.4	21.2	0.30	24
7.	Table 2 / mix no. 4	16.6	16.8	1.4	21.5	0.40	20
8.	Table 2/mix no.3/UV 4 hrs	17.1	17.0	1.7	22.2	0.30	14
9.	Commercial product no. 1	17.8	17.5	1.3	21.2	0.30	18
10.	Commercial product no. 2	17.6	17.8	2.0	37.6	2.00	18
11.	Commercial product no. 3	18.3	16.7	1.7	18.4	0.20	43

3.3. Effect of physical modification

Application of commercial chlorine stabilizers (used commonly for stabilization of polyvinyl chloride - PVC) in an amount of 1-5 phr, at the stage of preparation of polymer composition, practically eliminated release of chlorine from the modified materials, even after one year of exploitation of car windshield wipers. It has been confirmed by chemical microanalysis data and microindentation profiles, which do not reveal any trace of the material ageing, contrary to the data presented in Fig. 9. Tribological characteristics of the material exposed to the test of accelerate ageing (70 °C/one weak), do not vary significantly from the "fresh" one. This idea of modification, considerably extending lifetime of car windshield wipers, is also protected by the other Polish patent declaration.

Another way of bulk modification tested was based on our previous observations, concerning surface segregation taking place in polymer blends [5, 16]. The surface segregation, the most likely of low molecular weight fraction, of ethylene-propylene copolymer (EPM) blended with NR or SBR, was confirmed by FTIR spectra. Apart physical protection from ageing, segregated layer plays a role of some kind of lubricant, lowering friction of the materials – Fig. 11 and 12, what is especially important under "dry" conditions of friction.

Lubricating effect observed in the case of NR/EPM blends (Fig. 11), resembling the effect of UV radiation, was less durable in comparison to the effect produced by Fluorogard (FG), which is of significantly lower molecular weight than ethylene-propylene copolymer. The former produces much thinner surface skin, which is gradually worn off during friction, whereas lack of the latter is systematically completed by another molecules easily migrating from the subsurface layers of higher concentration [6]. It justifies significant difference in tribological behaviour of materials in the beginning of test, which however diminished when friction had stabilized.

3.4. Evaluation of car windshield wipers

It has been found, that available standard procedures proposed for evaluation of the efficiency of water removal from a car window by windshield wipers do not reflect their real performance under exploitation conditions. The tests are generally too short, and qualitative parameters proposed for evaluation of wiper work efficiency are not representative. Extending the period of examination makes possible to draw more reliable conclusions on suitability of the applied corrections to composition and overall modifications of the material. Some results of examinations are summarized in Table 4, in a form of exploitation parameters. Apart compositions given in Tables 1 and 2, one can also find some commercial products evaluated for comparison.

The best tribological parameters, from the materials being evaluated, were obtained for the commercial product no. 3 – having a graphitized strip, however this time some transfer of graphite to the glass counterface and its accommodation in the surface microroughness was detected, what was responsible for poor removal of water from the window by the wiper. It means, that taking into consideration only friction and energy characteristics of car windshield wipers is not enough to make the best choice for the most suitable material. From the exploitation point of view however, apart ability to effective water removal from a car window (reflected by the parameter "B", for which the lower the value of B the better), the other parameters proposed should not be too high. The lower the value of A the less energy is required to drive a car windshield wiper, what seems to be more important than values of C and D parameters, additionally deciding abrasion resistance of material. The higher their values the more energy is accumulated in the material, gradually intensifying its abrasion. The data presented in Table 4 demonstarte how the appropriate material engineering approach can improve tribological and exploitation performance of goods made of polymer materials. Some of the modifications work, leading to the materials of better characteristics / parameters in comparison to car windshield wipers available on the market.

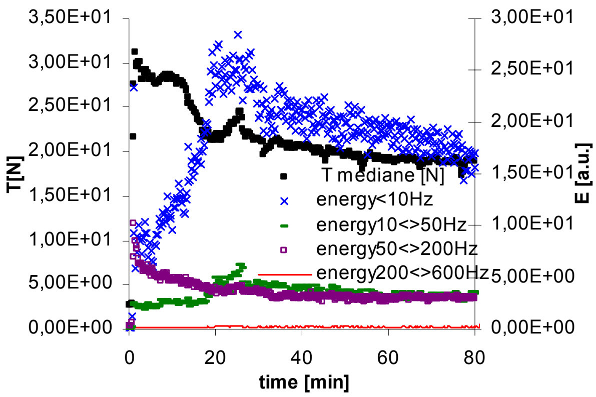


Fig. 11. Friction and energy characteristics of the polymer blends (Table 2/mix no. 4), studied under "dry" conditions

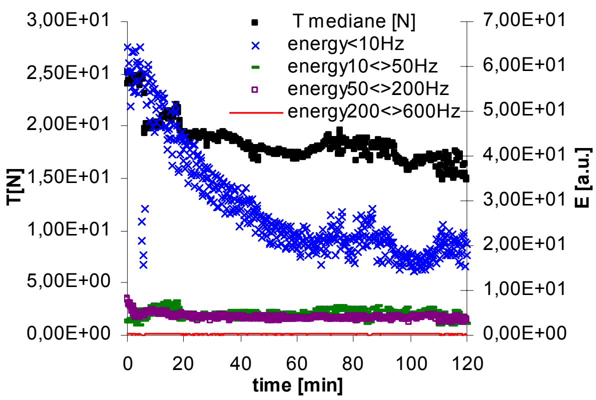


Fig. 12. Friction and energy characteristics of the polymer blends (Table 1/mix no. 8), studied under "dry" conditions

4. Conclusions

New approach to characterize tribological properties of polymer materials [17], based on application of FFT to friction force fluctuations, makes possible to estimate the beginning of wear and to identify the level of energy being dissipated during friction. Elastomer material of car windshield wiper blades revealed that high frequency deformations (200-600 Hz) are responsible for abrasion, whereas low frequency ones (0-10 Hz) decide the level of energy loss.

Tribological properties of polymer materials are not only dependent on mechanical and rheological properties but are also related to their composition and structure. Special attention should be devoted to the surface layer, which different nature in comparison to the bulk of polymers is very often underestimated or even neglected. This fact should draw a special attention to possibility of tailoring exploitation properties of polymer materials already at the stage of their compounding, processing or eventually by application of a simple chemical or physical post treatment. A case of car windshield wipers is a good example.

The data presented let to follow evolution of particular modification and to justify new experimental methods to determine and original approach to evaluate tribological and exploitation performance of: material and car windshield wipers respectively. The proposed parameters let friction, abrasion, as well as ability of a wiper to water removal to be correlated with composition and structure of the polymer material being made of.

Acknowledgements

The work has been carried out under the frame of the scientific project no. 4 T08E 064 24, financed by the Polish Ministry of Science and Informatization. Close cooperation with the MOTGUM Ltd., the Polish manufacturer of car windshield wipers is greatly acknowledged.

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