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Influence of running-in in dry and wet conditions on the interfacial adhesion of tire tread rubber

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Abstract. To study the running-in effects on the adhesion properties of tire tread compounds, an approach is proposed based on theoretical analysis of experimental load-distance curves. The approach uses the Maugis model of adhesion in an axisymmetric elastic contact. Classical AFM tests with a ball of 450 nm glued to the probe are performed for two different surfaces of the tire tread rubber after running-in in dry and wet friction regimes and for the original rubber surface. It is shown that running-in in wet conditions decreases the surface energy of rubber. Also, running-in in both wet and dry conditions leads to softening of a surface layer of rubber.

1. Introduction

It is known that the friction of elastomers has two basic mechanisms – adhesion and hysteresis – and thus depends on the adhesion and mechanical properties of elastomers [1, 2]. But the process of friction, in turn, influences the properties of elastomer surfaces, depending on many factors, such as type of materials, regime of friction, surface relief. In this study, a highly elastic tribotechnical material – tire tread rubber – is tested in sliding contact with a regular rigid surface having macro- and microtexture in the conditions of dry and wet friction. Under the influence of friction, wear, and liquid medium (soap solution in distilled water) during running-in, the surface of rubber changes its adhesion properties. To evaluate these changes, we propose an approach based on theoretical analysis of experimental load-distance dependencies obtained by AFM before and after running-in.

Usually, to extract the adhesion properties of materials from load-distance curves, the classical models of Johnson-Kendall-Roberts (JKR) and Derjaguin-Muller-Toporov (DMT) are used. These models give the specific work of adhesion w by the following simple formulas, which use only one value obtained from the load-distance curve – the pull-off force F_1 [3]:

$$w_{JKR} = 2F_1 \cdot (3\pi R)^{-1}, \quad w_{DMT} = F_1 \cdot (2\pi R)^{-1}$$
(1)

where R is the radius of curvature of the probe tip. But these simplified models cannot be used in the entire range of parameters. In the JKR model, the radius of adhesion force action is assumed to be infinitely small. The DMT model does not take into account the influence of adhesion on deformation of the interacting surfaces. Later, more exact models of adhesion were developed, both numerical and analytical, which can be used in wider ranges of parameters [4-6]. In particular, the Maugis model

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suggested for the description of the sphere-plane elastic adhesive contact [4] and generalized for axisymmetric bodies of power-law shape [5] takes into account both finite radius of adhesion force action and mutual influence of adhesion and contact deformation. This model is used as a basis for the approach suggested in the present study for the calculation of the adhesion properties of elastomers.

As a measure of adhesion of rubber, we use the specific work of adhesion at the silicon-rubber interface. This quantity is defined by the surface energy of rubber which plays the key role in friction of elastomers against smooth rigid surfaces.

2. Theoretical model

Consider the interaction between the AFM probe tip, which is assumed rigid and whose shape is described by the parabolic function $f(r) = r^2 \cdot (2R)^{-1}$, where *R* is the radius of tip curvature, and a rubber specimen. The probe is acted on by the external load *F*, the distance between the surfaces *d* being registered (figure 1 (a)).



Figure 1. Illustrations for the theoretical model – scheme of contact between the probe tip and a specimen (a) and the adhesion pressure as a function of gap between the surfaces (b).

Let (r, φ, z) be the cylindrical system of coordinates whose origin lies on the surface of the specimen and the z - axis coincides with the axis of symmetry of the probe tip. The surfaces are in contact over the region $r \le a$. In the region $a < r \le b$, the adhesion attraction occurs between the surfaces defined by the adhesion pressure p_a as a function of the gap δ . This function follows from the potential of intermolecular interaction. For Lennard-Jones potential, it is given by curve 1 in figure 1 (b). In the present study, we use the Maugis model in which this function is approximated by a piecewise-constant function (curve 2 in figure 1 (b)) and is expressed by the relation [4]:

$$p_{a}(\delta) = \begin{cases} -p_{0}, & 0 < \delta \le \delta_{0} \\ 0, & \delta > \delta_{0} \end{cases},$$
(2)

where the characteristic radius of adhesion force action δ_0 and adhesion pressure p_0 are considered the adhesion properties of the interface. The specific work of adhesion w is the work done by the adhesion force when two surfaces are moved apart to infinity. In the case of model (2), the specific work of adhesion is given by the relation

$$w = \int_0^{+\infty} p_a(\delta) d\delta = p_0 \delta_0 \tag{3}$$

Mechanical properties of the rubber specimen are described by the model of an elastic half-space under axisymmetric loading, for which the normal displacement of the boundary u(r) is related to the

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normal pressure p(r) by the expression [7]

$$u(r) = \frac{4}{\pi E^*} \int_0^b p(r') \mathbf{K}\left(\frac{2\sqrt{rr'}}{r+r'}\right) \frac{r'dr'}{r+r'}, \qquad 0 \le r \le b$$
(4)

where $\mathbf{K}(x)$ is the complete elliptic integral of the first kind, $E^* = E \cdot (1 - v^2)^{-1}$ is the reduced modulus of elasticity of the rubber specimen. The conditions at the boundary z = 0 of the elastic specimen follow from the contact conditions and relation (2):

$$u(r) = -r^{2} \cdot (2R)^{-1} - d, \qquad 0 < r < a$$

$$p(r) = -p_{0}, \qquad a \le r \le b$$

$$p(r) = 0, \qquad r > b$$
(5)

Also, the condition following from the adopted model of adhesion (2) is satisfied:

$$\delta(b) = \delta_0 \tag{6}$$

where the gap between the surfaces δ is defined by the expression (see figure 1 (a)):

$$\delta(r) = r^2 \cdot (2R)^{-1} + u(r) + d \tag{7}$$

Besides, the condition of equilibrium for the forces acting on the probe tip is satisfied:

$$F = 2\pi \int_{0}^{b} rp(r)dr$$
(8)

The solution of the adhesion contact problem (4)-(8) problem was constructed in [5] for the case of the power-law function of shape, $f(r) = Ar^{2n}$. By setting $n = 1, A = (2R)^{-1}$, we get the following relations for the load applied to the probe

$$F = \frac{4E^*a^3}{3R} - \frac{2p_0a^2}{\gamma^2} \left(\arccos\gamma + \gamma\sqrt{1-\gamma^2}\right)$$
(9)

distance between the probe tip and the specimen

$$d = -\frac{a^2}{R} + \frac{2p_0 a}{E^* \gamma} \sqrt{1 - \gamma^2}$$
(10)

and the equation for the determination of the contact radius a:

$$\left[\frac{a}{R}\left(\frac{1}{\gamma^2}-2\right)+\frac{4p_0}{E^*\gamma}\sqrt{1-\gamma^2}\right]\arccos\gamma+\frac{a}{R\gamma}\sqrt{1-\gamma^2}-\frac{4p_0}{E^*}\left(\frac{1}{\gamma}-1\right)-\frac{\pi\delta_0}{a}=0$$
(11)

Relations (9)-(11) specify the dependence of the load F on the distance d via the parameter $\gamma = a \cdot b^{-1}$. These relations are applicable in the entire range of variation of the Tabor parameter [8]

$$\mu_{\rm T} = \left(\frac{Rw^2}{E^{*2}\delta_0^3}\right)^{1/3}$$
(12)

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Unlike the classical simplified models which are its limit cases for $\mu_T >> 1$ (JKR limit) and $\mu_T <<1$ (DMT limit) [4, 8]. The disadvantage of relations (9)-(11) is their complex parametric form which does not allow one to obtain simple computational relations similar to (1).

In this work, we propose a method based on using two values obtained from the load-distance curves – the pull-off force F_1 and difference $d_2 - d_1$ between the distance at which the force is zero and the pull-off distance (figure 2). These values are determined from the retraction AFM curves.



Figure 2. A retraction segment of the load-distance curve with denoted values used for the calculation.

We solve equation (11) for *a*, choose the positive root of two, and substitute it into equations (9) and (10). As a result, the load and distance are expressed as functions of three unknown parameters - $F = F(\gamma, p_0, \delta_0)$ and $d = d(\gamma, p_0, \delta_0)$. These functions are applied for two points of the load-distance curve – the pull-off point and the point of zero load. At the pull-off point, the load attains its extremum value F_1 , i.e., the following relations are satisfied:

$$\left. \frac{\partial F(\gamma, p_0, \delta_0)}{\partial \gamma} \right|_{\gamma = \gamma_1} = 0 \tag{13}$$

$$F(\gamma_1, p_0, \delta_0) = F_1 \tag{14}$$

where γ_1 is the unknown ratio of the radii of the contact and adhesion regions at the pull-off point. At the point of zero load, we have

$$F(\gamma_2, p_0, \delta_0) = 0 \tag{15}$$

where γ_2 is the unknown ratio of the radii of the contact and adhesion regions at the point of zero load. Also, the following equation for the distance difference $d_2 - d_1$ is satisfied:

$$d(\gamma_2, p_0, \delta_0) - d(\gamma_1, p_0, \delta_0) = d_2 - d_1$$
(16)

The system of equations (13)-(16) is to be solved numerically by the iteration method for unknowns $\gamma_1, \gamma_2, p_0, \delta_0$, provided that the values *R* and E^* are known. After the adhesion properties p_0 and δ_0 are determined, the specific work of adhesion *w* is calculated as their product in accordance with (3).

Unlike formulas (1) of the classical models, the method suggested takes into account the influence of the modulus of elasticity on the pull-off force, and it does not assume any limitations on the value of the Tabor parameter μ_{T} (12).

3. Materials and equipment

As a material for specimens, we choose a tire tread rubber of car winter tire, which is produced from a compound based on mix of natural rubber (NR) and cis-butadiene rubber (CBR). Three groups of

rubber specimens are tested. The first group (N1) of rubber specimens are tested in the conditions of wet friction in 20% solution of liquid soap in distilled water. The second group (N2) of specimens are subjected to dry friction tests in the conditions similar to those of the first group. The third group (N3) of specimens is the original rubber after vulcanization. Rubber specimens are produced as rings of 7 mm height with internal radius 41 mm and external one 55 mm. The rings are glued to a steel substrate. As a counterbody for friction tests, a laminated plywood is used. The photograph of the counterbody, its surface microroughness, and geometric characteristics of texture are presented in figure 3.



Figure 3. Photograph of the counterbody (a), its surface roughness with the parameters $S_a = 1.4 \,\mu\text{m}$, $S_z = 6.2 \,\mu\text{m}$ (b), and 3D texture of a typical area of the plywood surface (c).

3.1. AFM technique

Experimental curves of approach and retraction were obtained by the atomic force microscope Integra Prima (NT-MDT, Russia). The photographs of the microscope and a rubber specimen under investigation are presented in figure 4.

We use the commercially available silicon cantilevers FMG01_Bio with the stiffness $1.5 \text{ N} \cdot \text{m}^{-1}$. At the end of the probe (cantilever), a spherical ball of silicon oxide with 900 nm diameter is attached. The force sensor calibration was performed by a series of shots into the polished amorphous sapphire whose stiffness is orders of magnitude higher that the stiffness of the rubber surface under investigation. It is assumed that when the sapphire is indented at large depth, the stiffness of the cantilever coincides with the stiffness of the sapphire. The depth of indentation of the ball into a rubber specimen is 2000 nm, and it varies slightly because of surface roughness of the rubber, particularly that after tribological tests.



Figure 4. Photographs of the AFM (a) and its probe over a specimen being tested (b).



Figure 5. Photograph of a rubber sample 1, which is fixed in the tribometer holder, where 2 is the counterbody.

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3.2. Tribological tests technique

Tests were carried out on the UMT-2 Tribometer (CETR Inc., USA) with ring-disk scheme of contact. A friction pair is shown in figure 5. A ring-shaped rubber specimen 1 is fixed in the self-adjustment holder of the tribometer. The disk-shaped counterbody 2 is fixed axisymmetrically on the stage rotated by the stepper motor. For tests in liquid medium, a lubricant container is set upon the stage, inside which the counterbody is fixed by a three-pin system. The working principle of the tribometer and the experimental technique are given in detail in [9]. Tests were performed under specific loads P = 0.1...05 MPa and sliding velocities V = 0.01...100 mm·s⁻¹ at room temperature $T = 23 \pm 3$ °C.

4. Results and discussion

Results of the tests are presented in figure 6 as plots of the coefficient of friction μ vs. sliding velocity V at various nominal pressures ranging from 0.1 to 0.5 MPa for two friction regimes. Analysis of the obtained experimental data shows that the curves have a peak, more noticeable for the dry friction regime (figure 6 (a)), which is accounted for by the adhesion mechanism of friction [10].



Figure 6. Coefficient of friction μ vs. sliding velocity V for dry (a) and wet (b) regimes of friction, where 1 is for P = 0.1 MPa, 2 is for P = 0.2 MPa, and 3 is for P = 0.5 MPa.

In the considered range of loads, the coefficient of friction decreases as the normal load increases. This is accounted for by the adhesion properties of rubber surface. It was shown in that in the presence of adhesion in dry contact, the coefficient of friction decreases as the load increases [11, 12]. In wet contact, the contribution of adhesion into friction is reduced, therefore the effect of the load on the coefficient of friction is less significant. Still, peaks associated with adhesion are seen in figure 6 (b), which is due to the artificial texture applied on the surface of the plywood. The texture makes the lubricant flow out from the interface, and thus the adhesion mechanism works even in wet conditions.



Figure 7. Experimental curves of retraction of the probe from the specimens surface (dots) and their approximation by calculation (solid lines), where curve 1 corresponds to the surface after wet friction, curve 2 to that after dry friction, and curve 3 to the original surface after vulcanization.

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AFM tests of the rubber surfaces were carried out before and after tribological tests in wet and dry regimes. Each specimen was subjected to not less than 10 tests on different spots located at 50 µm from each other. In figure 7, typical load-distance retraction curves are shown for three tested surfaces of rubber specimens in the range of the probe displacements from d = -100 to the value of d at which the maximum tensile force F is registered. From these curves, the values of F_1 and $d_2 - d_1$ were determined to be used as input data for solving the system of equations (13)-(16) to calculate the adhesion properties p_0 and δ_0 . As one more input parameter, we first used the Young modulus of rubber E = 6.9 MPa obtained by the classical test on uniaxial compression. But the results lead to high discrepancy between the calculated load-distance curves and experimental data for negative loads, which could not be reduced by any selection of the adhesion properties p_0 and δ_0 but it was eliminated well by choosing the modulus of elasticity E. This is supposedly explained by nonlinear elastic properties of rubber. However, our calculations show that in the range of small positive (≤ 100 nm) and negative depths of indentation of a ball with radius R = 450 nm, the rubber properties can be approximated well by linear elasticity with its own modulus, which we consider particularly justified for comparative tests. Thus, we calculate the modulus of elasticity by approximating experimental data with the parametric function (9)-(11) by the least square method at each iteration of the solution of system (13)-(16). In doing so, the elastic (E) and adhesion (p_0 , δ_0) properties are calculated simultaneously. The calculated curves are presented in figure 7. Note that simultaneous extracting of the elastic and adhesive characteristics from load-distance curves was first suggested in [13].

Three experimental curves were selected for each rubber specimen, the corresponding values of F_1 and $d_2 - d_1$ presented in table 1 (specimen N1 is after wet friction, N2 is after dry friction, N3 is the original surface). Also, the calculated adhesion pressure p_0 , radius of adhesion force action δ_0 , and modulus of elasticity *E* are presented. The specific work of adhesion *w* averaged for three curves is calculated. For comparison, the specific work of adhesion determined by the JKR and DMT formulas (1) is given, as well as the Tabor parameter calculated by formula (12)

NN	Massurad values		Calcula	ted adhes	ion and	Averaged work	Work of a	dhesion by	Tabor
	Measure	wieasureu values		elastic properties		of adhesion	JKR and DMT models		parameter
	F_1 , nN	$d_2 - d_1$, nm	$p_{\rm 0}$, MPa	$\delta_{\scriptscriptstyle 0}$, nm	E , MPa	$w, J \cdot m^{-2}$	W_{JKR} , $J \cdot m^{-2}$	w_{DMT} , J·m ⁻²	$\mu_{ ext{T}}$
1	320.4	445.1	7.768	19.39	1.530				
	280.6	465.5	6.101	21.64	1.253	0.1399	0.1413	0.1054	8.35
	293.0	439.3	7.562	18.13	1.428				
2	463.1	702.3	8.205	25.00	1.117				
	473.5	517.2	10.020	22.25	1.807	0.1946	0.2081	0.1562	8.54
	415.1	670.3	8.156	23.97	1.368				
3	463.1	702.3	19.761	9.98	5.935				
	473.5	517.2	19.040	9.49	5.575	0.1919	0.1927	0.1445	8.17
	415.1	670.3	20.151	9.82	5.636				

Table 1. Results of calculation of the p_0 , δ_0 , E and w for the three rubber specimens.

Since the surface energy is directly related to the specific work of adhesion w at the interface, it can be concluded that after dry friction, the surface energy of rubber increases insignificantly, while after wet friction it considerably decreases in comparison with the surface energy of the original surface. These results are in good correlation with the results of the tribological tests which show the decrease in the friction losses when the rubber is tested in 20% solution of soap in water (figure 6). Thus, the coefficient of friction in wet conditions decreases not only due to a decrease in the real

contact area but also due to a decrease in the surface energy of the rubber. This effect reduces the contribution of adhesion mechanism, which in most cases dominates the friction of elastomers.

The calculated values of the modulus of elasticity E correspond to the range of small compressive and tensile deformations occurring in a thin surface layer of a specimen, and they differ from its bulk Young modulus E = 6.9 MPa. Thus, running-in in both dry and wet regimes leads to a surface layer of rubber becoming effectively softer. The results of comparative tests suggest that it is softening of a surface layer rather than change in roughness or nonlinearity of the material. Also, the effective radius of adhesion force action δ_0 becomes larger than that of the original surface.

The calculated value of the specific work of adhesion w lies between of the values obtained by JKR and DMT models but it is closer to the JKR limit. This result is explained by high values of the Tabor parameter $\mu_{\rm T}$ which correspond to soft materials with relatively high surface energy [8].

5. Conclusion

Series of tribological tests were carried out in the conditions of dry and wet friction. The results show that the dependence of the coefficient of friction on the sliding velocity has a distinguished peak associated with the adhesion mechanism of friction. Introducing the lubricant (liquid soap solution in water) into contact reduces the contribution of adhesion into friction. Increasing of the normal load leads to decreasing of the coefficient of friction, irrespective of the sliding velocity and conditions of lubrication. This behavior is typical for molecular mechanism of friction of elastomers.

To study the adhesion properties of the rubber surface after running-in in different conditions, an approach was proposed to evaluate the adhesion characteristics of materials based on the load-distance curves obtained by atomic force microscopy. The method uses the Maugis model of adhesion in contact of elastic axisymmetric bodies and it is applicable in the entire range of the Tabor parameter. The results obtained by AFM and their theoretical analysis shows that friction in water solution of liquid soap considerably decreases the surface energy of rubber, while friction against the dry surface slightly increases the surface energy of rubber in comparison with that not subjected to friction tests.

Since the suggested approach, unlike the classical JKR and DMT formulas, takes into account the modulus of elasticity as a model parameter, the change of stiffness of a surface layer of rubber due to running-in was also possible to evaluate. It was obtained that as a result of running-in, the rubber surface softens, the presence of lubricant having no significant influence on its level.

The obtained values of the specific work of adhesion for rubber are compared with the results obtained by JKR and DMT models. The approach suggested allows one to obtain not only more exact values of the specific work of adhesion, but also to evaluate the characteristic values of the adhesion pressure and radius of the adhesion force action, which can be used as input parameters in modelling the adhesion component of friction between rubber and a rigid surface at the microlevel [12, 14].

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