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RESEARCH ARTICLE

Study of the corrosive effect of ozone on vulcanizates

Stanislav V. Mednikov✉, Pavel D. Kravchenya, Alexander S. Ponomarev, Oleg O. Tuzhikov
Volgograd State Technical University, Volgograd, 400005 Russia
✉ *Corresponding author, e-mail: mednikov17@yahoo.com*

Abstract

Objectives. The work sets out to model the ozone corrosion of vulcanizates as a percolation phase transition, similar in the scheme of development of continual percolation on a plane, during which the growing regions of the new phase form a single "spanning" cluster. In this case, the continuity of the sample is broken, being divided into two parts. In the presented model, the ozone corrosion process is divided into two stages. At the first stage, ozone corrosion of the material occurs mainly along the perimeters of already ozonized surface areas, which leads to their growth and subsequent merging. Upon contact of adjacent surface areas consisting of ozonolysis products loaded with two-dimensional tension, corrosion cracks begin to appear on the surface. At the second stage of the corrosion process, corrosion cracks that grow deeply into the material due to its stress state lead to the penetration of ozone into the internal regions of the sample. The article presents the results of computer-simulation and real experiments carried out on ozone corrosion of technical vulcanizates in a plane stressed state.

Methods. Computer simulation of the time dependence of the total contact length of the areas of ozone corrosion products and the initial vulcanizate was carried out using a C++ program developed by the authors. Real experiments were carried out on a TOM-1000 setup. Samples for research by the TOM (technical ozone resistance of materials[1]) method comprise thin disks, which are clamped along the contour and subjected to one-sided two-dimensional tension by compressed air pressure. From the side of the opposite plane, the sample is exposed to the ozone flow. The installation makes it possible to create in the sample a relative deformation of up to 100% increase in the surface area.

Results. Computer simulation allowed, in combination with direct measurements of the time dependence of ozone absorption, the dynamics of the destruction of vulcanizates in an ozone environment to be investigated. A numerical parameter of the ozone resistance of vulcanizates—the coefficient of ozone resistance—is proposed. This coefficient is almost linearly related to the time before the onset of cracking, but it is more accurate because it does not require visual observation of the ozonolysis process.

Conclusions. The results of computer simulation are in good agreement with the results of real experiments.

Keywords

hardness, corrosion, ozonolysis, percolation phase transition, aggressiveness of vulcanizates

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[1] TOM (technical ozone resistance of materials) is a method patented by the authors for testing vulcanizates for ozone resistance under flat stress conditions.

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НАУЧНАЯ СТАТЬЯ

Исследование коррозионного воздействия озона на вулканизаты

С.В. Медников✉, П.Д. Кравченя, А.С. Пономарев, О.О. Тужиков

Волгоградский государственный технический университет, Волгоград, 400005 Россия

✉ *Автор для переписки, e-mail: mednikov17@yahoo.com*

Аннотация

Цели. Озонная коррозия вулканизатов моделируется как перколяционный фазовый переход, аналогичный по схеме развития континуальной перколяции на плоскости, в процессе которой разрастающиеся области новой фазы образуют единый «перекидывающийся» кластер. Процесс озонной коррозии представляется разделенным на две стадии. На первой стадии процесс озонопоглощения происходит на границах корродированных участков с окружающей поверхностью вулканизата, поэтому поглощение образцом озона вначале возрастает, затем уменьшается по мере смыкания участков увеличивающейся корродированной поверхности. При соприкосновении соседних участков поверхности, состоящих из продуктов озонолиза, под воздействием двумерного напряжения на поверхности начинают появляться коррозионные трещины. На второй стадии коррозионные трещины проникают в глубь материала из-за его напряженного состояния, сопровождаясь проникновением озона во внутренние области образца. Изменение в процессе озонолиза общей длины линии контакта областей продуктов озонолиза с поверхностью, еще не подвергнутой озонной коррозии, отражает общее озонопоглощение корродирующего образца. Целью данной работы является экспериментальное исследование и компьютерное моделирование процессов зарождения трещин в образцах технических вулканизатов, находящихся в плоском напряженном состоянии, вследствие воздействия озона.

Методы. Компьютерное моделирование временной зависимости общей длины контакта областей продуктов озонной коррозии и исходного вулканизата осуществлялось с помощью разработанной авторами программы на языке C++. Реальные эксперименты по регистрации кинетики озонопоглощения озона шинными вулканизатами проводились на установке ТОМ-1000. Образцы для исследований по методу ТОМ[2] (техническая озоностойкость материалов) представляют собой тонкие диски, защемленные по контуру и подвергаемые одностороннему двумерному растяжению давлением сжатого воздуха. Со стороны противоположной плоскости образец подвергается воздействию потока озона. Установка дает возможность создания в образце относительной деформации до 100% увеличения площади поверхности.

Результаты. Компьютерное моделирование позволило, в сочетании с прямыми измерениями временной зависимости озонопоглощения, исследовать динамику процесса деструкции вулканизатов в среде озона. Предложен численный параметр озоностойкости вулканизатов — коэффициент озоностойкости, который практически линейно связан с временем до начала трещинообразования, однако более точен, т.к. не требует визуального наблюдения за процессом озонолиза.

Выводы. Результаты машинного моделирования находятся в хорошем согласии с результатами реальных экспериментов по регистрации кинетики озонопоглощения образцов шинных вулканизатов в процессе озонолиза, что свидетельствует в пользу правомерности модели двухстадийной коррозии вулканизатов в среде озона.

Ключевые слова

жесткость, коррозия, озонолиз, перколяционный фазовый переход, агрессивостойкость вулканизатов

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[2] ТОМ (техническая озоностойкость материалов) — запатентованный авторами метод тестирования вулканизатов на озоностойкость в условиях плоского напряженного состояния. [TOM (technical ozone resistance of materials) is a method patented by the authors for testing vulcanizates for ozone resistance under flat stress conditions.]

INTRODUCTION

The necessity for theoretical research on various aspects of material corrosion in aggressive environments is due to the diversity both of corrosive media and materials operating in these environments. In this work, we focus primarily on describing the behavior of mechanically stressed technical vulcanizates (rubbers) in an ozone environment, to which the established term "ozone corrosion" (ozonolysis) applies.

Resistance to the aggressive impact of ozone, which leads to stress cracking of rubber components, is one of the most important operational characteristics of these materials [1–3]. The universally accepted numerical criterion for ozone resistance of vulcanizates, as prescribed by the standards of many countries, is the "time to the appearance of the first corrosion crack on the surface of a vulcanizate sample under uniaxial load in an ozone environment" [4–5]. This moment in time is generally recorded visually. With the advancement of experimental techniques, dynamic testing methods which still rely on visual observation of the crack initiation moment have appeared in the practice of ozone resistance measurements [6]. However, the main focus in improving the ozone resistance of vulcanizates is currently directed toward developing new formulations and modifying existing ones [7–9].

Recently, the processes of crack formation and evolution in materials under ultimate loads have been analyzed in terms of geometric phase transitions (percolation) [10–14]. Without delving into possible microscopic mechanisms of crack initiation in ozone-aged vulcanizates, the present work considers ozone damage to vulcanizates as a geometric phase transition. The result of this transition is the nucleation of corrosion cracks in the surface layer of a material under a plane stress state.

The process of aggressive ozone action on the surface of rubbers in a mechanically stressed state begins in those areas where the surface structure is distorted by defects, such as the presence of filler particles, areas with uneven filler distribution in the material, or irregularities in the formed vulcanization network [15]. The surface density of such distorted sites, which initiate the interaction process between ozone and the vulcanizate, varies significantly depending on the vulcanizate production technology and its components [16]. Their initial areas are usually considered to be comparable to the sizes of filler components. Since ozone action occurs primarily at the boundaries of these "defective" sites with the surrounding surface, the areas of these sites increase as the interaction process between the vulcanizate and ozone develops. Accordingly, the increased rate of ozone absorption is proportional to the total perimeter of the areas of reacted sites on the ozonated surface. However, ozone diffusion into the bulk of the vulcanizate only affects the overall ozone absorption to a minor extent.

Subsequently, the isolated reacted areas begin to merge, leading to a decrease in their total perimeter and consequent reduction in the ozone absorption rate.

When the entire sample surface has reacted, further development of ozone corrosion occurs solely due to the diffusion of ozone into the vulcanizate. This initiates the formation and growth of cracks on the surface undergoing ozonolysis. Under stressed material conditions, this leads to crack opening and propagation, along with a continuous increase in the reactive area of the vulcanizate. Consequently, ozone absorption continues to increase until a through-crack forms in the sample.

Thus, the interaction of ozone with the vulcanizate surface can be divided into two temporal stages: before and after the moment of the first corrosion cracks appearing on the surface.

The first stage—the process of growth and subsequent merging of corroded areas on the vulcanizate surface—is analogous in its development scheme to continuous percolation on a plane. This is an abstract geometric phase transition that concludes with the formation of a "spanning" cluster, which divides the surface into two parts [17, 18]. For example, when sequentially punching holes of a certain predetermined diameter into a conductive plate, whose positions are chosen randomly, the electrical conductivity of the plate decreases along a curve characteristic of second-order thermodynamic phase transitions [17]. By continuing this procedure, a chain of interconnected holes is eventually created which cut the plate into two parts in some location. At this point, current through the plate ceases due to its electrical conductivity approaching zero. The region of the plate at which it becomes divided into two parts is called the "spanning" cluster.

Unlike a thermodynamic phase transition (e.g., ferromagnetic–paramagnetic), the onset of a percolation phase transition depends on time, whereas in the former case it depends on the temperature of the substance's phase.

The difference in our model lies in the fact that it does not involve the sequential addition of new phase regions (holes), but the sequential increase in the size of existing new phase regions (ozonolysis products). These regions initially exist as an isolated system of structural defects in the vulcanizate. With a merging of the growing ozonolysis product regions, crack formation occurs on the ozonated surface because the material is in a stressed state, and the second stage begins—the propagation of corrosion cracks into the sample bulk.

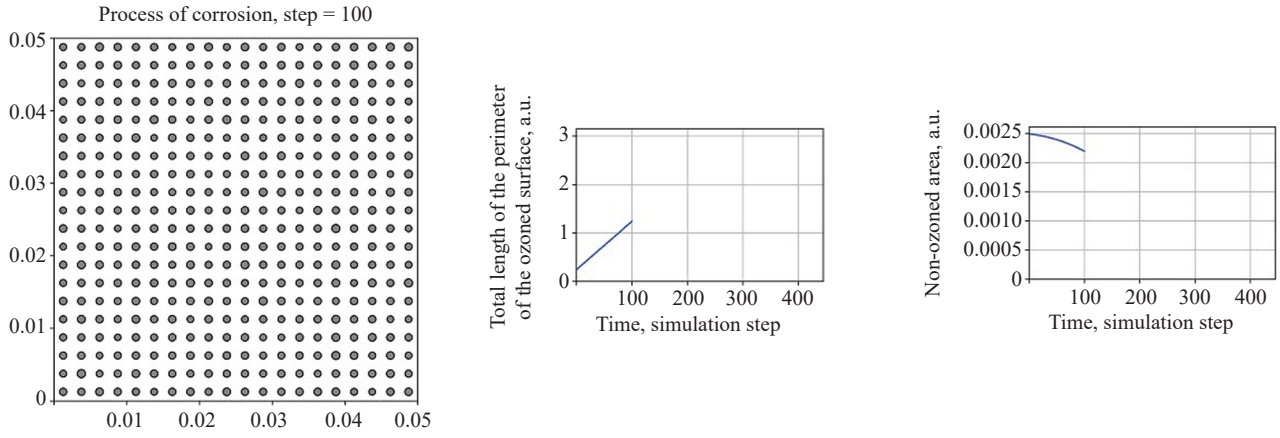
EXPERIMENTAL

To confirm the percolation nature of the change in the area of the vulcanizate surface having reacted with ozone, we conducted machine modeling of the change over time of the non-corroded surface area of a sample as a function of the number of modeling steps (i.e., time).

Consider a square plate with a certain area *S*. Place a square grid of equally spaced circles with initial radii r_0 on the plate. These circles model defects in the original surface structure of the vulcanizate that initiate ozonolysis along their perimeters. At each modeling step, the perimeters of the circles increase by a certain amount. Then, calculate the new circle radii, the total area occupied by the circles, the area not yet reacted with ozone, and the total length of all circle circumferences in the grid (the total perimeter of the boundary with the unreacted surface). Within this model, the ozone absorption rate is considered proportional to the total length of this combined perimeter.

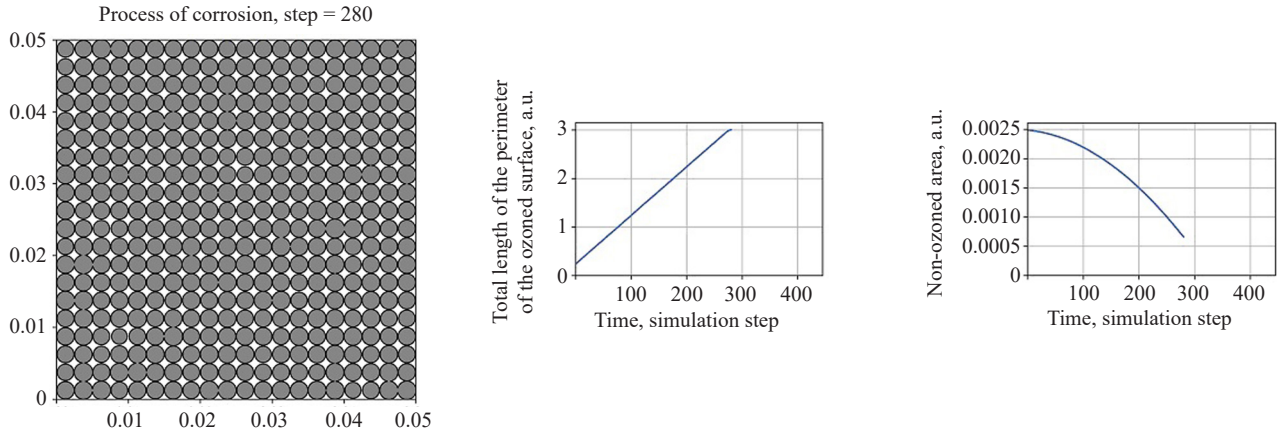
During the program's operation, the areas of the circles increase. Eventually, the circles begin to overlap, and the total length of the perimeter where the ozone-reacted surface contacts the still unreacted surface decreases. The program continues until the total area occupied by the reacted surface equals the total area of the plate *S*.

Figures 1–4 show the simulation stages for four time points, corresponding to step numbers: 100, 280, 320, and 400. In Figs. 1a–4a, the dark areas correspond to the current sizes of the reacted areas on the vulcanizate surface (hereinafter referred to as clusters). Figures 1b–4b show the time dependencies of the total length of cluster perimeters, while Figs. 1c–4c show the time dependencies of the total surface area not yet reacted at that moment. All quantities on the axes are given in arbitrary units.



(a) (b) (c)

Fig. 1. Simulation on a time interval of isolated clusters



(a) (b) (c)

Fig. 2. Moment of mutual contact of corroded clusters, corresponding to the maximum rate of ozone absorption

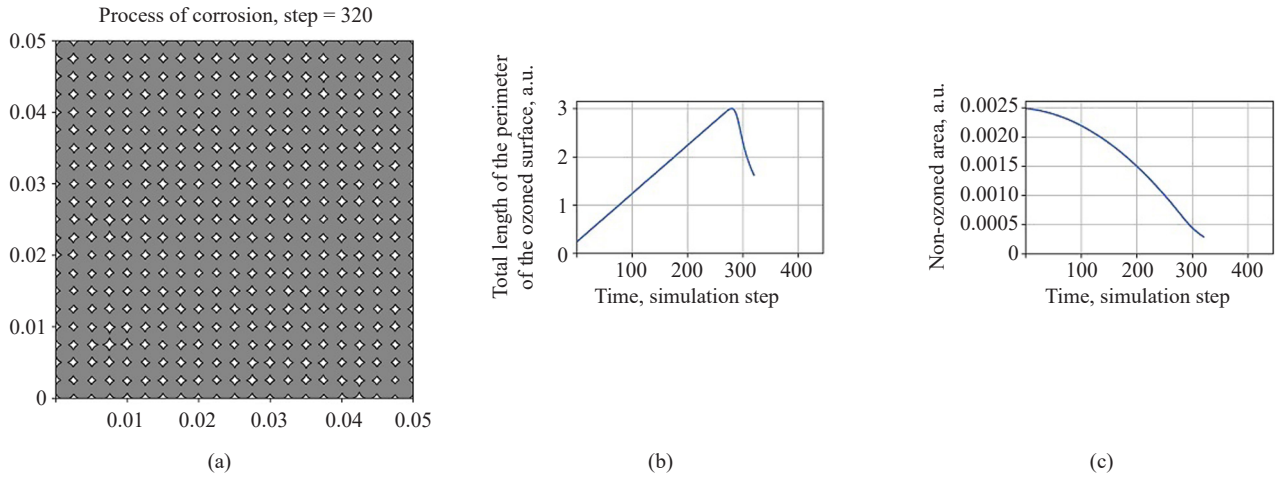


Fig. 3. Simulation on a time section of the connected clusters of the reacted surface

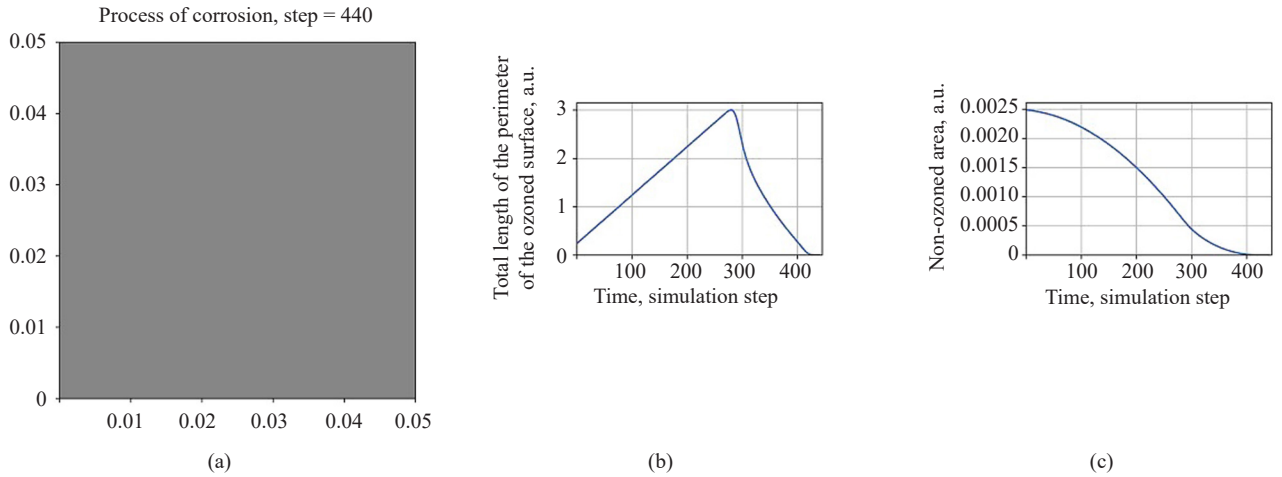


Fig. 4. Completion of the simulation. The entire surface is corroded

As the clusters grow in size, their total perimeter increases, and consequently, the ozone absorption rate increases (Fig. 1). Up to the 280th modeling step, the change in area over time resembles percolation behavior, characteristic, for example, of experiments recording current in a conductive strip as holes are punched with randomly chosen coordinates [19, 20], or in a system of parallel conductive buses as they are cut in random order [21]. Within this model, the maximum ozone absorption rate corresponds to the moment when the total perimeter of all growing clusters reaches its peak, observed when they contact each other at the 280th modeling step (Fig. 2). In continuum percolation theory, this moment corresponds to the formation of a "spanning" cluster [20]. In the conditions of our model, the total perimeter of boundaries with the unreacted surface subsequently decreases, corresponding to a reduction in the ozone absorption rate (Fig. 3). The simulation stops when the entire surface has been corroded (Fig. 4).

The rate of ozone corrosion and consequent ozone resistance of a vulcanizate can be most accurately calculated by analyzing the kinetics of ozone absorption by its surface. We investigated this process using the Technical Ozone Resistance of Materials (TOM) setup, developed at Volgograd State Technical University (Russia) [22, 23], for testing the ozone resistance of vulcanizates under conditions of a plane stress state. Ozone exposure is applied to one surface of a thin, flat disc-shaped sample, which is clamped along its perimeter and deformed on the opposite side by compressed air pressure. The TOM setup allows for the recording of the amount of ozone absorbed by the sample (i.e., reacted with the vulcanizate) during ozonolysis by measuring the ozone concentration in the ozone-air mixture to which the sample is exposed, at both the inlet and outlet of the reaction chamber. Figure 5 shows typical results of recording the time-dependent change in the ozone absorption rate (mol/s) during testing of a series of tire vulcanizate samples on the TOM setup. Comparing

the graphs in Figs. 1–4 and Fig. 5, a good agreement between the model and the results of the real ozonolysis process of a vulcanizate is observed. The formation of the first corrosion cracks, i.e., the increase in the reaction-active surface area, for this vulcanizate composition begins around the 24th second of the ozonolysis process (Fig. 5). As seen from the graph, the ozone absorption rate subsequently begins to increase again, indicating the onset of the second stage of ozonolysis that involves the propagation of corrosion cracks into the bulk of the sample.

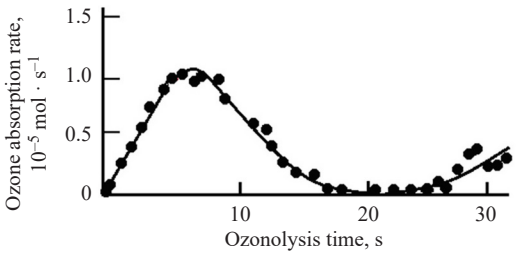


Fig. 5. Typical time dependence of the ozone absorption rate of tire vulcanizates

From a practical standpoint, it is of interest to investigate the dynamics of the deterioration (degradation) of the physicomechanical properties of a material during the first stage of ozonolysis, which concludes with the formation of corrosion cracks on its surface.

RESULTS AND DISCUSSION

The main parameter recorded during testing of vulcanizates for ozone resistance using the TOM method is the pressure of the air acting on the sample in the pressurization chamber of the setup. Ozonolysis leads to a decrease in the sample's stiffness and its subsequent deformation, which is recorded as a pressure drop. A typical view of the time dependence of pressure in the pressurization chamber during sample ozonolysis is presented in Fig. 6 [24]. The moment of crack initiation is registered by the TOM method as the beginning of the linear segment on the time dependence of pressure decrease in the setup's pressurization chamber.

A sample that has been clamped along its perimeter and subjected to compressed air pressure from the pressurization chamber side represents an elastic element, which is generally nonlinear. The deformation of this element can be characterized by the deflection w_0 at its center (the apex of the dome). For small deflection values, it can be considered a linear elastic element—a spring with stiffness k—and Hooke's law can be applied to it:

$$P \cdot B = k \cdot w_0, \quad (1)$$

where P is the current pressure of the compressed air on the sample; B is the effective area of the sample, depending on its diameter and the design features of the TOM setup; w_0 is the deflection of the apex of the sample dome loaded by the compressed air pressure from the pressurization chamber.

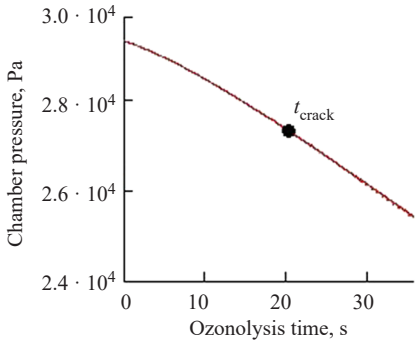


Fig. 6. Time dependence of pressure in the injection chamber. t_{crack} is the moment of the onset of crack formation in the sample

In the first stage of ozonolysis, ozone exposure affects a thin surface layer of the material. According to current understanding, the decrease in sample stiffness occurs only due to the deterioration of the physicomechanical properties of this layer. Let us model the sample as two elastic elements connected in parallel, having thicknesses h_1 and h_2 and stiffnesses k_1 and k_2. Stiffness k_1 decreases as the area of the surface not yet reacted with ozone diminishes, while stiffness k_2 decreases much more slowly during ozonolysis. At the start of ozonolysis, the material of the surface layer is identical to the rest of the sample material. Therefore, the ratio of the initial stiffnesses of the surface layer k_1^0 and the remaining material k_2^0 equals the ratio of their thicknesses, i.e.,

$$k_2^0 = \frac{h - h}{h_1} \cdot k_1^0, \quad (2)$$

where h_1 is the thickness of the surface layer; h is the thickness of the sample.

Since the total perimeter in our model is assumed to increase linearly with time (until the clusters begin to merge), the total ozonated surface area increases quadratically with time. Therefore, the time-dependent law for the decrease in stiffness of the surface layer can be written as:

$$k_1(t) = k_1^0 - k't^2,$$

where, k' characterizes the rate of decrease in the stiffness of the sample's surface layer during ozonolysis. Its reciprocal, therefore, serves as an objective criterion

for the ozone resistance of the sample material (during the first stage of ozonolysis). The total stiffness of the sample is:

$$k_{tot}(t) = k_1^0 - k't^2 + k_2(t). \quad (3)$$

Here, $k_2(t)$ accounts for the slower process of ozone diffusion through the surface layer into the depth of the sample.

For small sample deflections (less than 0.4 of the sample radius), a dependence of pressure P in the pressurization chamber on the maximum deflection w_0 (at the apex of the deformed sample's dome) was obtained in the form:

$$P(t) = \frac{P_0 V_0}{B w_0(t)}, \quad (4)$$

which represents Boyle–Mariotte's law (the ozonolysis process of vulcanizates on the TOM setup is isothermal). Here, P_0 and V_0 are the pressure and volume of compressed air in the pressurization chamber at the start of ozonolysis, while B is the effective sample area introduced earlier.

Let us write Hooke's law (1) in the form:

$$P(t)B = k_{tot}(t)w_0(t) = \left[k_1^0 - k't^2 + k_2(t)\right] \cdot w_0(t). \quad (5)$$

Expressing the deflection w_0 from (4) and substituting it into (5), we obtain the time dependence of pressure change in the pressurization chamber due to the decrease in sample stiffness on the time interval from the start of ozonation to the moment of the first corrosion crack formation:

$$P(t) = \sqrt{\frac{k_{tot}(t)P_0V_0}{B^2}} = \sqrt{\frac{\left[k_1^0 - k't^2 + k_2(t)\right]P_0V_0}{B^2}}. \quad (5a)$$

The best agreement of the obtained relationship with experimental results on the TOM setup is observed under the assumption of a linear law for the decrease in stiffness $k_2(t)$, i.e.:

$$P(t) = \sqrt{\frac{\left[k_1^0 - k't^2 + k_2^0 - k''t\right]P_0V_0}{B^2}}. \quad (6)$$

The negative root in (6) corresponds to negative gauge pressure in the pressurization chamber. In the TOM setup, this sample loading option is not used.

Neglecting the change in stiffness k'' during the first stage of ozonolysis, we can rewrite Eq. (6) as:

$$P(t) = \sqrt{\frac{\left[k_1^0 - k't^2\right]P_0V_0}{B^2}}. \quad (6a)$$

Equation (6a) allows for the calculation of the constant k' from experimental results obtained using the TOM setup, which determines the moment of crack initiation, t_{crack}, and the initial sample stiffness k_0. The initial stiffness is calculated using Eq. (1), based on the condition that at $t = 0$, $P = P_0$. The set of experimental results constitutes a two-dimensional array "time t – pressure P," which, according to Eq. (6a), expresses a quadratic dependence of pressure on the sample versus time during the first stage of ozonolysis. To obtain k' values from this data, any suitable computational environment, for example, MATHCAD, can be used.

Further, the thickness of the surface layer damaged during the first stage of ozonolysis can be calculated from the experimental results. For the series of tire vulcanizate samples used in this work, the thickness of the surface layer damaged during the first stage of ozonolysis was approximately 50 μm.

Figure 7 shows the relationship between the ozone resistance coefficient we propose and the standard ozone resistance parameter—the time to crack initiation in the ozonated vulcanizate.

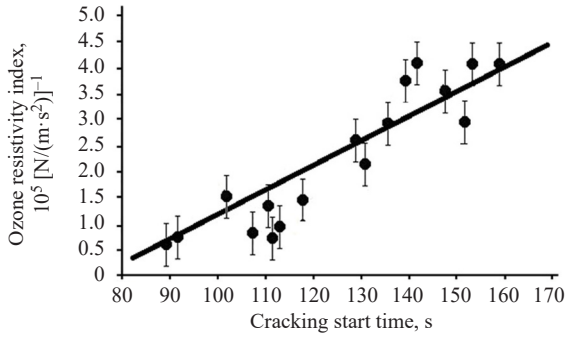


Fig. 7. Relationship between the ozone resistance coefficient and the time before crack formation in tire vulcanizate samples

The graph is based on the results of statistical processing of ozone corrosion experiments conducted on a series of 17 tire vulcanizate samples. The ozone concentration in the ozone-air mixture at the inlet of the reaction chamber was 12 mg/L ± 10% in all experiments.

As can be seen from the graph, the ozone resistance coefficient correlates well with the previously mentioned standard criterion for vulcanizate ozone resistance—the time to the first corrosion crack formation in the sample. However, it avoids the subjective aspect of the standard method, which uses visual observation to record the time of corrosion crack formation.

CONCLUSIONS

1. Crack formation arising on the surface of vulcanizates in a mechanically stressed state during corrosive exposure can be considered in terms of a percolation phase transition, similar in nature to continuous percolation.

2. A model is proposed for the decrease in mechanical stiffness during ozonation of a sample deformed by one-sided pressure. The sample is considered as a system of two elastic elements connected in parallel, whose stiffnesses decrease during the first stage of ozonolysis at significantly different rates. The model is in good agreement with experimental results obtained on the TOM setup.
3. The numerical indicator of vulcanizate resistance to aggressive ozone exposure proposed in this work—the ozone resistance coefficient—is practically linearly related to the currently accepted ozone resistance parameter, the time to crack initiation. However, the numerical indicator is more accurate as it does not require visual observation of the ozonolysis process development.

Authors' contributions

S.V. Mednikov—development of the application of percolation theory in the theoretical model of ozone corrosion of vulcanizates, participation in the development of the methodology for machine modeling, participation in the discussion of results.

P.D. Kravchenya—development of the computer program for modeling vulcanizate corrosion, execution of computer simulations, participation in the discussion of results.

A.S. Ponomarev—conducting experimental studies on the ozone resistance of tire vulcanizates using the TOM setup, participation in the discussion of results.

O.O. Tuzhikov—development of the theoretical model of ozone corrosion of vulcanizates, participation in the development of the methodology for machine modeling, participation in the discussion of results.

The authors declare no conflicts of interest.

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About the Authors

Stanislav V. Mednikov, Cand. Sci. (Phys.-Math), Associate Professor, Department of Physics, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: s_mednikov@vstu.ru. Scopus Author ID 57212473929, RSCI SPIN-code 1584-9501, https://orcid.org/0000-0001-6621-7550

Pavel D. Kravchenya, Cand. Sci. (Phys.-Math), Senior Lecturer, Department of Electronic Computers and Systems, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: kpd_@mail.ru. Scopus Author ID 36628612400, RSCI SPIN-code 1584-9501, https://orcid.org/0000-0002-1934-5792

Alexander A. Ponomarev, Head of the Laboratory, Department of Computer Engineering, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: alpo5404@gmail.com. https://orcid.org/0000-0002-3659-7094

Oleg O. Tuzhikov, Dr. Sci. (Eng.), Associate Professor, Head of the Department of General and Inorganic Chemistry, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: tuzhikovoleg@mail.ru. Scopus Author ID 12645529200, RSCI SPIN-code 8142-5915, https://orcid.org/0000-0001-6316-8896

Об авторах

Медников Станислав Владимирович, к.ф.-м.н., доцент, кафедра физики, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: s_mednikov@vstu.ru. Scopus Author ID 57212473929, SPIN-код РИНЦ 1584-9501, https://orcid.org/0000-0001-6621-7550

Кравченя Павел Дмитриевич, к.ф.-м.н., старший преподаватель, кафедра «Электронно-вычислительные машины и системы», ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: kpd_@mail.ru. Scopus Author ID 36628612400, SPIN-код РИНЦ 1584-9501, https://orcid.org/0000-0002-1934-5792

Пономарев Алексей Сергеевич, заведующий лабораторией, кафедра «Вычислительная техника», ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: alpo5404@gmail.com. https://orcid.org/0000-0002-3659-7094

Тужиков Олег Олегович, д.т.н., доцент, заведующий кафедрой общей и неорганической химии, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: cand@vstu.ru. Scopus Author ID 12645529200, SPIN-код РИНЦ 8142-5915, https://orcid.org/0000-0001-6316-8896

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