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# Investigation of relation between durability of adhesive joints of soft polymeric materials and roughness characteristics of glued surfaces

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## 1. Introduction

The real contact area of glued surfaces has a big influence on strength and durability of adhesive joints. This indicator depends on geometrical dimensions of the glued surfaces as well as on the roughness of the surface due to the fact that the properly chosen glue covers the surface as well as penetrates the irregularities that appear on the surface after it has been processed with abrasive materials. Relations between durability of adhesive joints and indicators of the roughness of abrasive materials are analyzed in this paper. The master curve method was used for processing the experimental data on durability.

The master curve method is the result of the existence of definite analogies, and the temperature-time analogy is the most widely researched one [1, 2]. Initially it was applied for the description of viscoelastic properties of polymers, and later it was used for the analysis of strength properties of polymers and adhesive joints [3-8].

Macroscopically tests confirm that external factors of the same or even different origins, for example, short-term high temperatures or long-term low temperatures, influence mechanical properties of polymers and must be acknowledged [9] as a direct proof of the existence of this and other analogies, keeping in mind that complex structure of polymers (and particularly of adhesive joints) cannot be defined strictly mathematically.

When applying the temperature-time analogy, master curve is received from experimental curves by parallel shift along the time axis by the value of $a_T$ ($a_T$ is called the shift factor), which is determined by the Williams - Londell Ferry (WLF) equation

$$lga_T = -\frac{c_1^s\left(T - T_s\right)}{c_2^s + T - T_s} \qquad (1)$$

where $c_1^s$ and $c_2^s$ are coefficients; $T$ is temperature; $T_s$ is reference temperature.

When the temperature-time analogy is valid, the expression of WLF equation does not depend on the choice of $T_s$. So it is possible to match empirically received values of $a_T$ with the WLF equation in two ways: by choosing two parameters $c_1^s$ and $c_2^s$ or one parameter $T_s$.

The first method is based on the fact that $c_1^s$ and $c_2^s$ acquire universal values: $c_1^s = 8.86$ and $c_2^s = 101.6$. Then the value $T_s$ often satisfies the condition $T_s = T_g + 50^\circ$ ($T_g$ is glass transition temperature). But this is not always observed. This phenomena is especially characteristic for adhesive joints [7]. So it is not possible to state that reference temperature $T_s$ is known after choosing values for $c_1^s$ and $c_2^s$ that are equal to the universal values.

When master curves are plotted in the second way, the value of $T_s$ is selected freely for the reasons of rationality. Then $c_1^s$ and $c_2^s$ are calculated according to empiric values of $lg\, a_T$ using the least-squares method. Such method of drawing the master curves is especially effective for the analysis of durability because choosing the minimum experimental temperature as the reduction temperature extends the time interval considerably.

Besides the temperature-time analogy other analogies were also investigated where the influence of strains, vibrations, and moisture (instead of temperature) on deformational properties of polymers was determined [10]. The existence of temperature-concentration analogy was determined when analyzing the strength of adhesive joints [11]. When applying two analogies in parallel or in series a possibility appears to draw the complex master curve that allows to forecast the investigated indicator depending only on three factors.

In this paper the durability of adhesive joints was investigated with dependence on three factors: temperature, load, and grains size of abrasive material which is used to roughen the glued surface. The aim of this work is to produce a complex master durability curve used to forecast the durability depending on the three before mentioned factors.

Grain size of the abrasive material that is used to roughen the surface influences the characteristics of surface roughness. These characteristics may be of two types: statistical and fractal, depending on positions from which the profile of the roughened surface is treated. Statistical profile characteristics are constructed, when the profile of the roughened surface is considered to be the realization of a random process. Also it is considered that the random process satisfies the preconditions of stationarity, ergodicity, and normal distribution. But these preconditions are not always satisfied, and this stimulates to develop other models of the surface profile. Nowadays, methods of fractal geometry [12] are used to define rough surfaces and their quantitative fractal characteristics. One of the most important is fractal dimension $D$ that is calculated from the following equation

$$D = \frac{ln\left(N\left(\delta\right)\right)}{ln\left(\frac{1}{\delta}\right)} \qquad (2)$$

where $\delta$ is the length of segments that cover fractal curve;

$N(\delta)$ is the number of such segments.

In [13-15] the methods of fractal geometry were used to define characteristics of metal surfaces roughness. But fractal characteristics of profilogramms have not yet been applied for the investigation of roughened surfaces of soft polymeric materials. Moreover, the fractal dimension has not been related with the durability of adhesive joints.

## 2. Experimental methodology and results

The experimental research objects were adhesive joints of monolithic black rubber and textile material. Butadiene-styrene rubber (the density and hardness according to the Shore scale were $\rho$=1.25 g/cm$^3$, $H$=7 respectively) and double-layered kersey were used for adhesive joints. Samples of the researched materials were glued with polyurethane glue (with concentration of 15%).

Before gluing the surfaces of rubber samples were processed with strips of abrasion paper of various grade numbers. The following grade number abrasives were used in this study: P24, P40, P60 and P100. The bigger grade number of the abrasive corresponds to the smaller size of the abrasive grain. Table 1 presents all abrasives of various grade numbers and the corresponding average abrasive grain size.

Table 1
Relation between abrasive grade number and abrasive grain average size

| Abrasive grade number according to *FEPA* | Average size of abrasive grain *r*, mm |
|---|---|
| P24 | 0.698 |
| P40 | 0.382 |
| P60 | 0.260 |
| P100 | 0.149 |

Profilogramms were obtained from the roughened surface perpendicular to the abrasion direction using testing-machine – profilograph "Hommelwerke T500" (Germany; minimal measurement limit is 0.2 μm). 10 profilogramms that match the same abrasive grade number were produced for each surface. Fig. 1 presents profilogramms of surfaces processed with different abrasives.

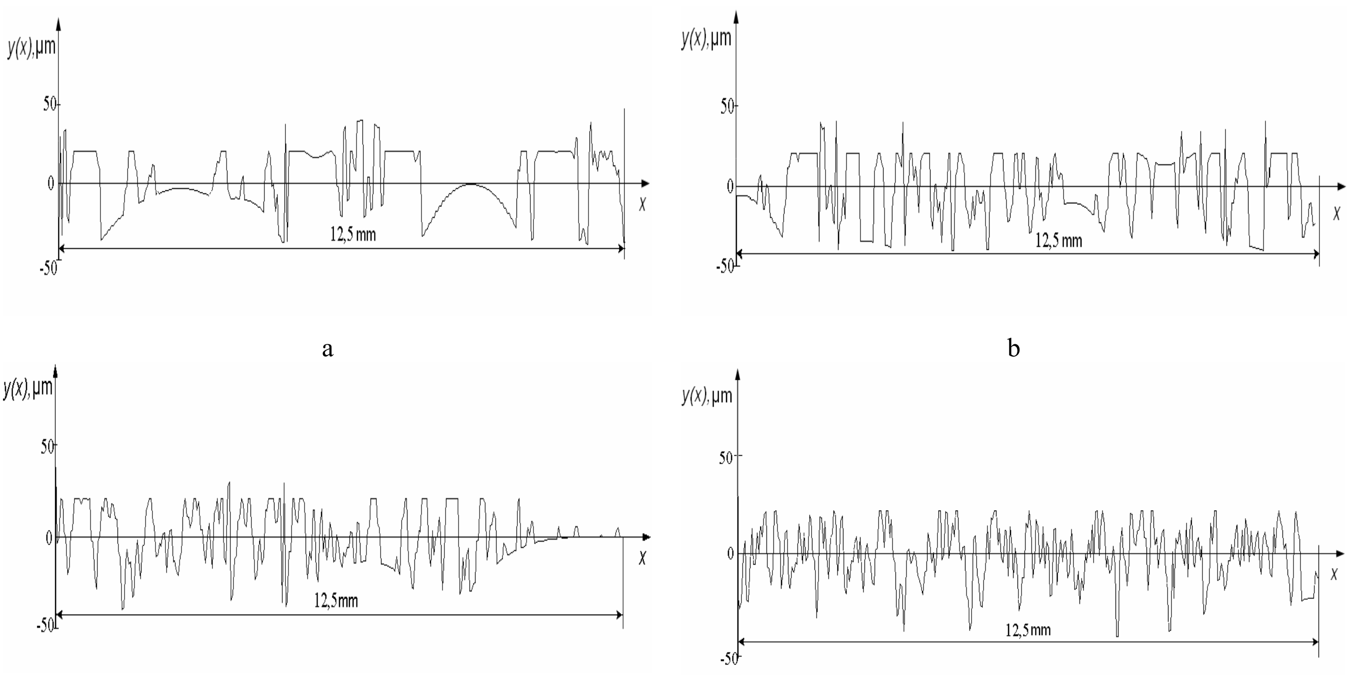


Fig. 1 Typical profilogramms of roughened surfaces that match different abrasive grade numbers: a - P 24; b - P 40; c - P 60; d - P 100

Then the samples of the analyzed materials were prepared for gluing. After application of the first and the second layers of the glue the samples were left to dry in room temperature for 30 and 60 min for the first and the second time, respectively. After that the adhesive film was heat reactivated at the temperature $T = 80$ºC for the duration of 1 min. After heat activation the samples of the researched materials were placed in contact and pressed for 1 min under the pressure of 0.25 MPa.

The size of the produced adhesive joints is $10 \times 20$ mm, and the size of the glued part is $10 \times 10$ mm.

The durability test was performed after 24 h after adhesion. The adhesive joints were tested in a thermal chamber of a modified machine used to measure creep under constant temperature $T$ (precision $\pm 1^\circ$) and constant load $P$. The samples were kept in all temperatures for 900 s. The adhesive joints were tested under the following temperatures $T$: 303, 308, 313 and 318 K. The following values of load $P$ were used: 1, 1.2, 1.5, and 1.7 kN/m.

We used mean arithmetical values of durability $\tau$ for our calculations (each experimental point was produced from 5 - 10 samples depending upon the spread of indices). In this paper durability $\tau$ is the time during which the sample with the glued part area of 1 cm$^2$ is de-laminated and disintegrates. The data on durability is expressed in seconds.

Results of the experimental test of durability of adhesive joints are given in Fig. 2.

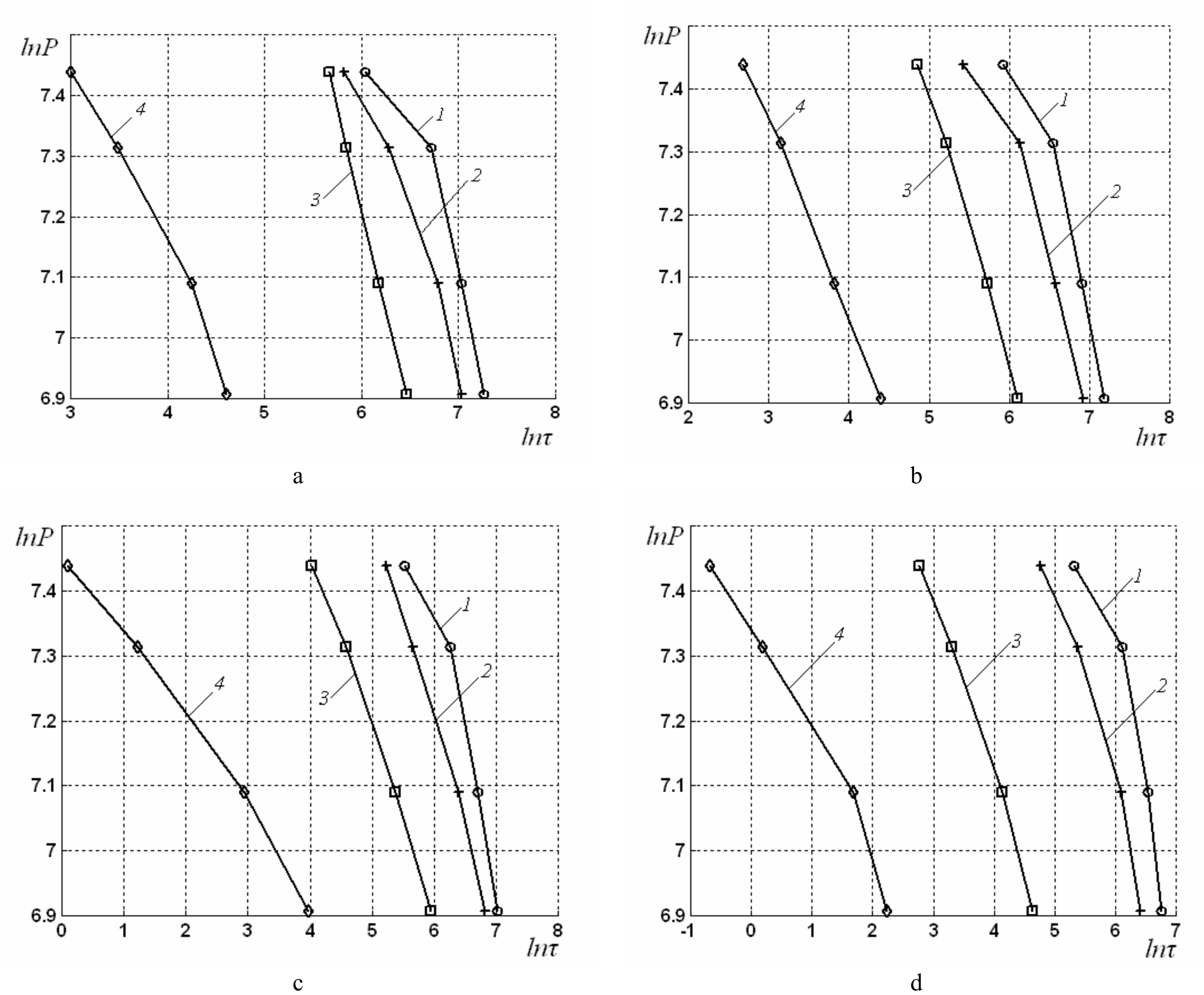


Fig. 2 Relationship between durability of adhesive joints, load and temperature at different surface roughness. Surface was processed with adhesives with the following grain numbers: a - P24; b - P40; c - P60; d - P100. Different point markings match different temperature values (curves: *1*- $T_1 = 303K$ ; *2*- $T_2 = 308K$ ; *3* - $T_3 = 313K$ ; *4* - $T_4 = 318K$ )

It can be seen that the durability of adhesive joints depends on load and temperature as well as on the surface roughness. When temperature is minimal $T_1 = 303K$ , the highest durability is of those adhesive joints where rubber surface was processed with grade number P24 abrasive, and the lowest durability is of those where the abrasive grade number was P100. The same tendency remains in other temperatures as well. This may be explained by the fact that the glue penetrates deeper into bigger irregularities and destruction of the adhesive joint requires larger loads due to mechanical gripping effect.

### 3. Forecast of durability of adhesive joints depending on temperature, static load, and surface roughness

When constructing master durability curves the minimal experimental temperature was chosen as the reference temperature $T_s$, and the biggest abrasive grain size that matches the smallest abrasive number was chosen as roughness index $r_s$. Reduction with regard to these values allows to forecast the region of higher durability.

Durability master curves (Fig. 3) that match all values of *r* according to the data given in Fig. 2 were plotted in coordinates $lnP\frac{T_s}{T} - ln\tau a_T$ ; here $a_T$ is shift factor described by the WLF equation (1).

Each master curve from Fig. 3 can be used to forecast the durability of adhesive joints depending on various intermediate values of temperature and load (not necessarily used in experiment) but only under certain values of roughness parameter *r*. It must be noted that after the application of temperature-time analogy, the time interval where the durability can be forecasted is lengthened by 1-2 logarithm units, compared with the values of durability determined experimentally.

Choosing $T_s = 303\text{K}$ , we calculated constants $c_1^s$ and $c_2^s$ of the WLF equation for each roughness *r* by the least-squares method, and we found that they depend on the values of *r*. Further we examined the dependence of constants $c_1^s$ and $c_2^s$ on the parameter $R = \frac{r}{r_s}$ .

After examination we determined that their analytical expressions take the form

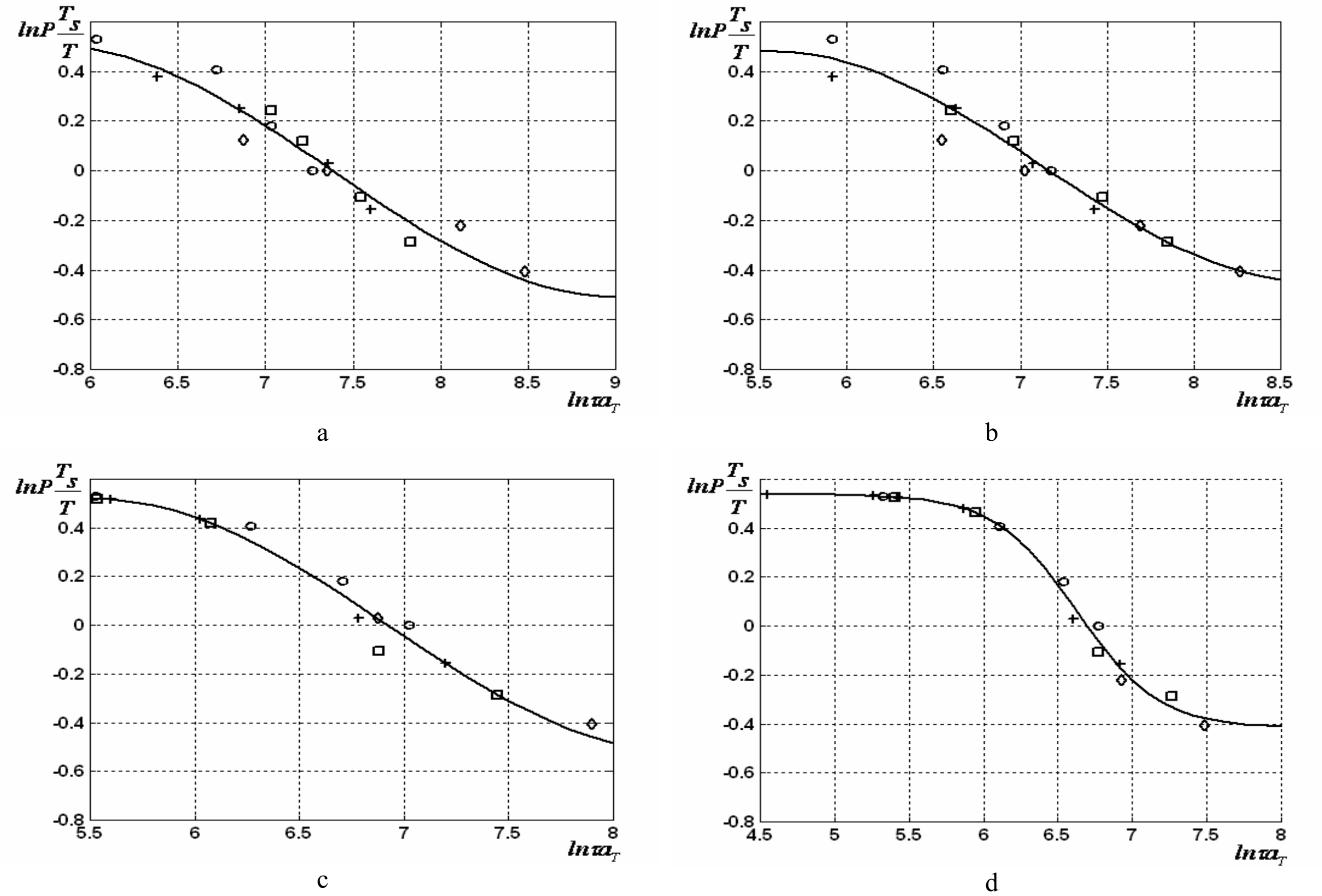


Fig. 3 Master curves of the durability of adhesive joints, when the surface is processed with abrasives with the following numbers: a – P24; b – P40; c – P60; d – P100 (different point markings match different temperature values: o – $T_1 = 303K$ ; + - $T_2 = 308K$ ; □ - $T_3 = 313K$ ; ◊ - $T_4 = 318K$ )

$$c_1^s(R) = -1.83R^2 + 0.59R - 1.11 \quad (3)$$

$$c_2^s(R) = -12.22R - 13.14 \quad (4)$$

Using analytical expressions of constants $c_1^s$ and $c_2^s$ in the WLF equation we construct the following dependence of shift factor $a_T$

$$lna_T = -\frac{\left(-1.83R^2 + 0.59R - 1.11\right)\left(T - T_s\right)}{-12.22R - 13.14 + \left(T - T_s\right)} \quad (5)$$

In this paper we construct the complex master curve of durability using two analogies in parallel. For this purpose master durability curves (Fig. 4) were transformed by a parallel shift along the axis $ln\tau a_T$ into a complex master curve (Fig. 5). Complex master curve is the basis for multi-parameter durability forecasting.

We determined that empirical dependence of shift factor $a_r$ on roughness parameter *r* takes the form

$$lna_r = \frac{1}{0.95 + 0.90\left(\frac{r}{r_s} - 1\right)} \quad (6)$$

It is clear from Fig. 5 that complex master curve of adhesive joints durability can be approximated with the help of the following equation

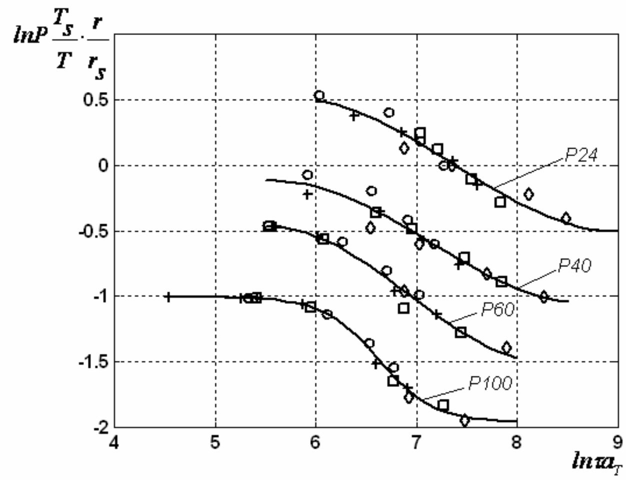


Fig. 4 Master curves of durability of adhesive joints (different point markings match different temperature values: o – $T_1 = 303K$ ; + - $T_2 = 308K$ ; □ - $T_3 = 313K$ ; ◊ - $T_4 = 318K$ )

$$lnP\frac{T_s}{T}\frac{r}{r_s} = kln\tau a_T a_r + m \quad (7)$$

where *k*, *m* are coefficients determined by the least-squares method: $k = -0.46$ ; $m = 3.41$ .

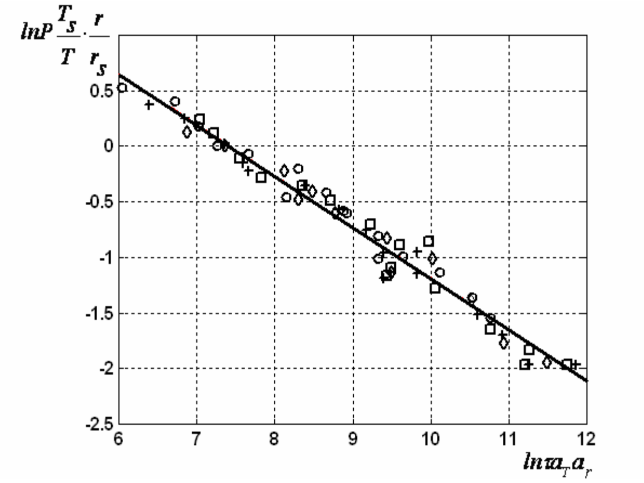


Fig. 5 Complex master curve of durability of adhesive joints (different point markings match different temperature values: o $-T_1 = 303K$ ; + - $T_2 = 308K$ ; □ - $T_3 = 313K$ ; ◊ - $T_4 = 318K$ )

After using the values of $a_T$ and $a_r$ (that are defined by Eqs. (5) and (6) respectively) in Eq. (7), we find that the dependence of durability on load $P$, temperature $T$, and surface roughness parameter $r$ can be forecasted from the following equation

$$\tau = \frac{A}{a_T a_r}\left(\frac{T}{PT_s R}\right)^2 \qquad (8)$$

where $a_T = exp\left(\frac{(-1.83R^2 + 0.59R - 1.11)(T - T_s)}{-12.22R - 13.14 + (T - T_s)}\right)$,

$a_r = exp\left(\frac{1}{0.95 + 0.90(R-1)}\right)$; $A = e^m$ ; $R = \frac{r}{r_s}$ .

We see that the complex master curve extends the time interval where the durability can be forecasted by 5 time logarithm units, compared with the durability values determined experimentally.

## 4. Research of fractal characteristics of rubber surface profilogramms

In this paper we make a premise that roughened rubber surface profile is a fractal curve. We calculated box dimensions of each profilogramm – fractal from data of discretized profilogramms.

We calculated fractal dimensions of surface profilogramms of different roughnesses for 5 profilogramms that represent each roughness. The calculated fractal dimensions were averaged after. The received results are presented in Table 2.

Table 2

Fractal dimensions of profilogramms

| Abrasive grade number | Mean fractal dimension of profilogramms | 95% confidence intervals fractal dimension |
|---|---|---|
| P24 | 1.288 | (1.249; 1.328) |
| P40 | 1.432 | (1.417; 1.446) |
| P60 | 1.531 | (1.511; 1.551) |
| P100 | 1.552 | (1.538; 1.565) |

In this paper roughness is considered to be the entirety of micro-irregularities that are positioned in a relatively small distance from one another. So from this point of view the surface processed with grade number P100 abrasive is rougher that the surface processed with grade number P24 abrasive. Since the fractal dimension characterizes how "densely" fractal fills the space, it is natural to assume that profilogramms of the surfaces processed with different abrasives will have different fractal dimensions.

Later we analyzed the dependence of mean fractal dimension $D$ on abrasive grain size $r$. We determined that the relationship between the mean fractal dimension $D$ and the abrasive grain size $r$ is linear (Fig. 6), as indicated in the relation

$$D = 1.638 - 0.351R \qquad (9)$$

where $R = \frac{r}{r_s}$ . The model explains 97.98 % of all data.

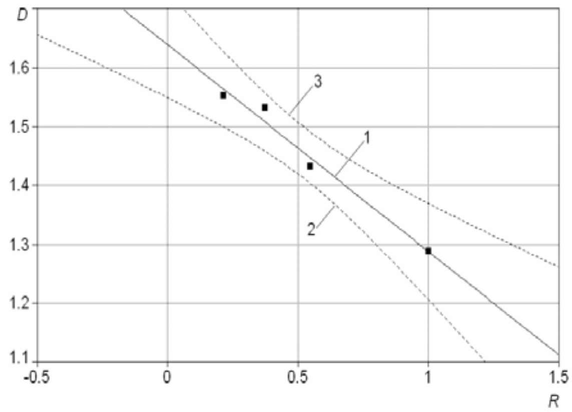


Fig. 6 Relationship between mean fractal dimension $D$ and abrasive grain size $r$ (curve *1*) and 95 % confidence intervals of this relation (curves *2* and *3*)

Since the change of roughness influences fractal dimension size, it is important to analyze how roughness parameters depend upon the fractal dimension. We calculated two parameters of surface roughness: arithmetical height mean $R_a$ and average square profile deviation from the middle line $R_q$ . They are presented in Table 3.

We determined that it is possible to describe the dependencies of parameters $R_a$ and $R_q$ on mean fractal dimension by the following equation

$$y = \frac{a + cx}{1 + bx} l \qquad (10)$$

where $y = R_a$ or $y = R_q$ , $x = D$ ; $a$, $b$, and $c$ are coefficients determined by the least-squares method; $l$ is the profile length.

It can be seen from Table 4 that the difference between coefficients $a$, $b$, $c$ that match $R_a$ and $R_q$ is small. This model explains 99.60 % of the values of parameter $R_a$ and 90.01 % of the values of parameter $R_q$.

Eqs. (9) and (10) dependencies are very important because they allow to forecast fractal dimension of surface profile processed with any abrasion paper and to calculate roughness parameters without performing the experiment.

Table 3

Surface roughness parameters

| Abrasive grade number | Average size of abrasive grain $r$, mm | $R_a$, µm | 95% confidence intervals of parameter $R_a$ | $R_q$, µm | 95% confidence intervals of parameter $R_a$ |
|---|---|---|---|---|---|
| P24 | 0.698 | 18.932 | (17.502;20.362) | 19.756 | (18.091; 21.421) |
| P40 | 0.382 | 16.514 | (13.675;19.354) | 18.101 | (15.124; 21.078) |
| P60 | 0.260 | 11.030 | (9.899; 12.161) | 16.115 | (14.739; 17.491) |
| P100 | 0.149 | 10.018 | (9.724; 10.312) | 10.825 | (10.458; 11.192) |

Table 4

Comparison of model coefficients for parameters $R_a$ and $R_q$

| Coefficients | For $R_a$ | For $R_q$ |
|---|---|---|
| $a$ | 0.00177 | 0.00166 |
| $b$ | -0.59491 | -0.62846 |
| $c$ | -0.00110 | -0.00106 |

In this work we wanted to check the suitability of relationships of (9) and (10), and we performed the forecast of parameter $R_q$ and compared the received results with experimental results. Firstly, we forecasted fractal dimension of the surface profile processed with abrasion paper of grade number P36. The average size of abrasive grain of this abrasion paper is 0.476 mm. Then according to Eq. (9) fractal dimension $D$ equals 1.399. After putting this value into Eq. 10 we received that $R_q = 18.324$ µm. Then we performed the control experiment: we roughened the rubber surface with abrasion paper (grade number P36) and, having discretized the received profilogramms of the processed surface, we calculated the value of parameter $R_q$ . It equals 18.84 µm. Relative error is 2.74%.

The Eq. (8) that was received earlier describes the dependence of adhesive joints durability on abrasive grain size $r$. Since there is a relation also between abrasive grain size $r$ of abrasive paper and fractal dimension $D$ (which is described by relationship (9)) the durability of adhesive joints also depends on fractal dimension. Then the dependence of durability of adhesive joints on fractal dimension can be expressed in the following way

$$\tau = \frac{A}{a_T a_r}\left(\frac{T}{PT_s(4.67-2.85D)}\right)^2 \qquad (11)$$

where

$$a_T = exp\left(\frac{\left(-14.86D^2+47.03D-37.15\right)\left(T-T_s\right)}{34.83D-70.21+\left(T-T_s\right)}\right)$$

$$a_r = exp\left(\frac{1}{4.25-2.57D}\right);\ A=e^m;\ R=\frac{r}{r_s}$$

This dependence is important when we do not know the grain size of abrasion paper used to process a rubber surface. Then after determining the fractal dimension experimentally it is possible to forecast the adhesive joints durability.

## 5. Conclusions

It was shown that it is possible to forecast the durability of adhesive joints depending on temperature, load, and abrasive grain size from the complex master curve received after the application of two analogies in parallel. After approximation of the complex master curve the dependence of durability on the three mentioned factors is determined.

Fractal dimensions of roughened surfaces that were used for adhesive joints were examined, and their relation with abrasive grain size was determined. The received dependence relates the durability of adhesive joints with fractal dimensions of roughened surface profiles.

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MINKŠTŲ POLIMERINIŲ MEDŽIAGŲ SANKLIJŲ ILGAAMŽIŠKUMO IR SUKLIJUOJAMŲ PAVIRŠIŲ ŠIURKŠTUMO CHARAKTERISTIKŲ RYŠIO TYRIMAS

R e z i u m ė

Darbe ištirtas monolitinės juodos gumos, suklijuotos poliuretaniniais klijais su tekstiliniu audiniu, sanklijų ilgaamžiškumas, priklausomai nuo temperatūros ir apkrovos. Panaudojant temperatūros ir laiko analogiją, nubraižytos apibendrintos kreivės. Nustatyta VLF lygties koeficientų priklausomybė nuo abrazyvinės medžiagos, kuria buvo pašiurkštintas paviršius, grūdelių dydžio. Lygiagrečiai panaudojant dvi analogijas, nubraižyta kompleksinė apibendrinta kreivė, iš kurios galima prognozuoti sanklijų ilgaamžiškumą priklausomai nuo trijų faktorių – temperatūros, apkrovos ir abrazyvinio grūdelio dydžio. Šios priklausomybės analizinė išraiška gauta, aproksimavus kompleksinę apibendrintąją kreivę trijų kintamųjų funkcija. Surastos gumos paviršių profilių fraktalinės dimensijos ir nustatytas jų ryšys su abrazyvinio grūdelio dydžiu. Gauta sanklijų ilgaamžiškumo priklausomybė nuo temperatūros, apkrovos ir fraktalinės dimensijos.

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INVESTIGATION OF RELATION BETWEEN DURABILITY OF ADHESIVE JOINTS OF SOFT POLYMERIC MATERIALS AND ROUGHNESS CHARACTERISTICS OF GLUED SURFACES

S u m m a r y

Dependence of durability of adhesive joints of monolithic black rubber glued applying polyurethane glue with a textile material on temperature, load and surface roughness was investigated in this paper. Master curves were constructed using the temperature-time analogy. Dependence of the coefficients of the WLF equation on grain size of the abrasive material that was used to roughen the surface was determined. A complex master curve was plotted using two analogies in parallel that can be used to forecast the durability of adhesive joints depending on three factors – temperature, load, and abrasive grain size. Analytical expression of this dependence was derived after approximation of the complex master curve with the three-variable function. Fractal dimensions of rubber surface profiles were found, and their relation with the abrasive grain size was determined. A dependence of adhesive joints durability on temperature, load, and fractal dimension was found.

Л. Маченайте, В. Пекарскас

ИССЛЕДОВАНИЕ ВЗАИМОСВЯЗИ МЕЖДУ ДОЛГОВЕЧНОСТЬЮ КЛЕЕВЫХ СОЕДИНЕНИЙ МЯГКИХ ПОЛИМЕРНЫХ МАТЕРИАЛОВ И ХАРАКТЕРИСТИКАМИ ШЕРОХОВАТОСТИ СКЛЕИВАЕМЫХ ПОВЕРХНОСТЕЙ

Р е з ю м е

В работе исследована долговечность клеевых соединений черной монолитной резины и текстильного материала на полиуретановом клее в зависимости от нагрузки, температуры и характеристик шероховатости поверхности. Для построения обобщенных кривых долговечности использована температурно – временная аналогия. Найдена зависимость коэффициентов уравнения ВЛФ от величины зерн абразивного материала, которым обработана поверхность субстрата. Комплексная обобщенная кривая долговечности клеевых соединений, из которой можно прогнозировать долговечность в зависимости от трех факторов - нагрузки, температуры и величины абразивного зерна, построена с помощью параллельного применения двух аналогий. Аналитический вид этой зависимости найден путем аппроксимации комплексной обобщенной кривой функцией трех переменных. Найдена фрактальная размерность профилей поверхности резины и установлена связь между фрактальной размерностью и величиной абразивного зерна. Найден вид зависимости долговечности клеевых соединений от нагрузки, температуры и фрактальной размерности.

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