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**FORECASTING OF MECHANICAL
PROPERTIES OF ELASTOMERS
ON THE BASIS OF ACCELERATED TESTS**

Summary of Doctoral Thesis

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Riga, 2005

Rēzeknē: RA Izdevniecība, 2005. – 44 lpp.
ISBN 9984 – 779 – 23 – 8
Tirāža 50 eks.

Martinovs A. Forecasting of mechanical properties of elastomers on the basis of accelerated tests. Summary of Doctoral Thesis. - Riga: RTU, 2006. - 44 p.

Iespiests saskaņā ar RTU Mehānikas institūta padomes sēdes 2005.gada 19.decembra lēmumu Nr.4

Šis darbs izstrādāts ar Eiropas Sociālā fonda atbalstu nacionālās programmas “Atbalsts doktorantūras programmu īstenošanai un pēcdoktorantūras pētījumiem” projekta “Atbalsts RTU doktorantūras attīstībai” ietvaros.

This work has been partly supported by the European Social Fund within the National Programme “Support for the carrying out doctoral study programm’s and post-doctoral researches” project “Support for the development of doctoral studies at Riga Technical University”.

Эта работа выполнена при содействии Европейского социального фонда в рамках проекта “Поддержка развития докторантуры РТУ” национальной программы “Содействие осуществлению программ докторантуры и исследований после нее”.

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Atbrīvošanas alejā 90, Rēzeknē, LV 4600
RA Izdevniecība, 2005
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Autors izsaka visdziļāko pateicību saviem promocijas darba vadītājiem - akadēmiķim, dr.habil.sc.ing., prof. **Egonam Lavendelim** un dr.sc.ing., prof. **Vladimiram Goncam**, RTU Mehānikas institūta direktoram, LZA kor.loc., dr.habil.sc.ing., prof. **Jānim Vībam** un visiem **RTU Mehānikas institūta darbiniekiem** par atbalstu un konstruktīvu kritiku, savam darba devējam - **Rēzeknes Augstskolai**, dr.sc.ing., prof. **Irēnai Silinevičai** un dr.habil.geoloģ., prof. **Gotfrīdam Novikam** par sapratni un atbalstu, **Eiropas Sociālajam fondam** un **Rīgas Tehniskajai universitātei** par sniegto materiālo atbalstu promocijas darba izstrādes laikā projekta „Atbalsts RTU doktorantūras attīstībai” ietvaros.

Ich möchte mich bei der **Fachhochschule Oldenburg/ Ostfriesland/ Wilhelmshaven** Standort Wilhelmshaven für die Möglichkeit die experimentellen Teile meiner Promotionsarbeit durchzuführen bedanken. Für die Hilfe und Unterstützung bin ich folgenden Professoren und Mitarbeiter der FH O/O/W besonders dankbar: Prof. Dr.-Ing. **Josef Timmerberg**, der Leiterin des AKA Mg. **Andrea Menn**, Prof. **Hanns Grützner**, Prof. Dr. **Götz Strömsdörfer**, Prof. Dr. **Klaus Schmieder**, Prof. Dr. **Bernd Thoden**, Prof. Dr. **Christoph Thoma**, Prof. **Ingo Poth**, Herrn Dipl.-Ing. **Paul Beckmann**, Herrn Dipl.-Ing. **Albrecht Hirt**, Herrn Labor-Ingenieur **Robert Buse**, Herrn **Helmut Schütte**, Frau Dipl.-Ing. **Petra Galleck**, Herrn Dipl.-Ing. **Volker Gottschewski**, Frau **Judith Legrand**.

SYNOPSIS

This thesis surveys existing mathematical models describing behavior of elastomers and existing methods of forecasting their mechanical properties.

It elaborates new mathematical model to characterise the deterioration of elastomers under different loads. Alongside mechanical and thermal parameters, time, elements that affects ageing of elastomers – temperature and lead-in energy, model also includes dielectric permeability.

Empirical correlations make possible including in the model the specific resistance and the transmission capacity of infrared rays as well.

Technical provision and methods to take dielectric measurements of elastomers are elaborated.

Changes of mechanical, electrical and optical characteristics during ageing are investigated and relations between those changes are established.

Method to forecast mechanical properties and lifespan of elastomers based on existing and experimentally tested procedures is developed.

INTRODUCTION

Rubber products have very wide use in many different economic sectors – transport, machine manufacturing, water supply, construction, electrical engineering etc. During use they are exposed to whole spectrum of mechanical loads, affected by temperature, radiation and aggressive substances. The impact of all these factors causes physical and chemical changes in rubber which results in the deterioration of mechanical and electrical characteristics. This process is called the ageing of rubber. In order to solve specific engineering and technical tasks, it is necessary to determine mechanical and electrical properties of the rubber and forecast how will they change during ageing as well as predict the lifespan of the rubber product.

These issues have been examined by many scientists such as D.Aneli [30], G.Bartenev [33], M.Bolotashvili [30], M.Celina [6], A.Donskoj [44], V.Druzinin [45], K.Gillen [6], I.Golberg [37], A.Goldman [38], V.Gubanov [39], [40], G.Hamed [9], [10], V.Hrichikov [71], A.Iljušin [49], E.Lavendelis [53], [54], [65], V.Moskvitin [58], H.Murashka [40], I.Nikitin [59], [60], J.Nikitin [59], [60], K.Sau [24], M.Shashkina [44], J.Zhao [10], J.Zujev [33], [48]. The analysis of their work in magazines „Каучук и резина” (Caoutchouc and Rubber; in russian) and „Rubber Chemistry and Technology” shows that only little attention is paid to changes in electrical properties (conductivity) of rubber during ageing, but changes in dielectric permeability have not been disclosed at all.

Performing relevant experiments [16], [19], [55] showed that with ageing there are changes in dielectric permeability in some types of rubber. Also correlation between mechanical properties and dielectric permeability was found. This prompted suggestion that mechanical tests can be substituted for relevant electrical measurements. The example of the practical application of this idea would be quality tests for retreaded car tyres which are widely used in Latvia. Tyres are recapped by vulcanizing new rubber on the tread part of tyre, leaving sidewalls unrestored. Endurance of the ageing rubber is lower which makes sidewalls the most vulnerable part of the tyre. In further use possible defects in adhesion between old and new rubber will cause them to separate, resulting in rubber hitting a car body and other traffic on the road, but tyre will still perform its function for long enough time. But if sidewall cracks, tyre can deflate within seconds and can cause serious accident on the road. To avoid situations like this it is necessary to know endurance of the tyre in any particular moment in time. Mechanical tests during usage of the retreaded tyre are problematic and expensive. Instead the suggestion is to perform electrical tests, which

would not have destroying effects on the tyre and could be done in service stations without taking a tyre off a car.

Performed experiments show that prospective expenses of equipment for this kind of electrical tests are not high – in current conditions they would be Ls 100 – 120. Same kind of tests could be carried out not only for car tyres, but also for other elastomers products.

The objective of the Thesis is on the basis of dielectric measurements to develop express-method to define and to forecast mechanical properties and lifespan of elastomers (rubber).

The scientific novelty is the use of dielectric permeability measurements in forecasting techniques of mechanical properties and lifespan of elastomers.

The main results of the Thesis are:

- Ø the disclosure of stochastic correlations between mechanical, electrical and optical characteristics of rubber. This allows in forecast methods of mechanical parameters and life span of rubber to use electrical and optical measurements which do not destroy sample and are easy to automate;
- Ø the developing of experimentally tested express-method to determine and to forecast the mechanical properties of elastomers (rubber) on the basis of dielectric permeability measurements.

The results of Thesis have been issued in publications as follows:

1. Martinovs A., Gonca V. A rubber fall model for yield deformation// In: International Conference on bionics and prosthetics, biomechanics and mechanics, mechatronics and robotics.- Varna, 2004- Vol.4- p. 47- 50.
2. Martinovs A., Gonca V. Research of mechanical properties and dielectrical permeability in aging process of rubber (Исследование механических свойств и диэлектрической проницаемости резины в процессе старения; in russian) // In: International Conference on bionics and prosthetics, biomechanics and mechanics, mechatronics and robotics.- Varna, 2004.- Vol.4- p.41- 46.
3. Martinovs A. Parameter determination for the reological rubber model (Gumijas reoloģiskā modeļa parametru noteikšana; in latvian)// Scientific Proceedings of Riga Technical University. Transport and Engineering. Mechanics.- Riga, 2002.- Vol.7- p. 65- 70.
4. Martinovs A. Selection of physics parameters for prognosing rubber aging processes (Fizikālo lielumu izvēle gumijas novecošanas procesu prognozēšanai;

- in latvian)// Proceedings of Rezekne Higher Education Institution.- Rezekne, 2001, Vol.3. p.71.-80.
5. Martinovs A. On predicting rubber life expectancy (Par gumijas kalpošanas laika prognozēšanu; in latvian)// International conference: Traditions and innovations in sustainable development of society.- Rezekne: Rezeknes Augstskola, 2002.- 67.-74. p.
 6. Martinovs A., Kangro I. Research of correlation between mechanical and electrical parameters of rubber (Korelācijas pētījumi starp gumijas mehāniskiem un elektriskiem raksturlielumiem; in latvian)// 2nd World Latvian Scientists' Congress.- Riga, 2001.- p. 555.

For Publishing in RTU Scientific Proceedings Accepted Following Works:

1. Martinovs A., Gonca V. Researches of dielectrical properties of tires in aging process (Autoriepu dielektrisko īpašību pētījumi novecošanas procesā; in latvian).- 2005.- 6 p.
2. Martinovs A., Timmerberg J., Gonca V. Research of relevances between mechanical and electrical parameters of rubber (Sakarību starp gumijas mehāniskiem un elektriskiem parametriem pētījumi; in latvian).- 2005.- 8 p.

List of Conferences, where reported about results of the work:

1. The 45th International Scientific Conference of Riga Technical University, 2004.
2. International Conference on bionics and prosthetics, biomechanics and mechanics, mechatronics and robotics.- Varna, 2004.
3. The 44th International Scientific Conference of Riga Technical university, 2003.
4. The 43rd International Scientific Conference of Riga Technical university, 2002.
5. International conference: Traditions and innovations in sustainable development of society.- Rezeknes Augstskola, 2002.
6. 2nd World Latvian Scientists' Congress.- Riga, 2001.

Structure of the Thesis:

Introduction

1. Literature review.
 - 1.1. Structure and properties of rubber in brief.
 - 1.2. Behavior models of rubber in brief.
 - 1.3. Review of literature and standards for determination of physical properties of elastomers.
 - 1.4. Review of existing methods for forecasting mechanical properties of elastomers.
 - 1.5. Conclusions from literature review and scientific objectives.
2. Theoretical researches.
 - 2.1. Hypothesis for use of electrical and optical values in methods for determination and forecasting mechanical properties of elastomers.
 - 2.2. Model of breakdown of rubber in one-way tensile strain.
 - 2.3. Determination of elastic potential in one-way tensile strain.
 - 2.4. Use of dielectric permeability in model of breakdown of rubber.
 - 2.5. Calculation model of rubber in one-way compression strain.
 - 2.6. Calculation model of rubber in two-way tensile strain.
 - 2.7. Calculation model of rubber for cylindrical drum with load of inner pressure.
 - 2.8. Calculation model of rubber in two-way compression strain.
 - 2.9. Calculation model of rubber in shear.
 - 2.10. Calculation model of rubber in torsion.
 - 2.11. Calculation model of rubber in dynamic tensile strain under compression.
 - 2.12. Conclusions of theoretical research.
3. Experimental research.
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 - 3.2. Method of performing tension tests.
 - 3.3. Method of performing creep tests.
 - 3.4. Method of performing hardness tests.
 - 3.5. Method of taking electrical measurements for the rubber.
 - 3.6. Method of taking measurements of infrared transmission capacity for rubber.
 - 3.7. Types of rubber used in experiments.
 - 3.8. Estimation of measurement errors and approximation of experimental data.
 - 3.9. Research on changes in hardness of rubber during ageing.
 - 3.10. Research on changes in maximum tensile strain of rubber during ageing.
 - 3.11. Research on changes in yield point of rubber during ageing.
 - 3.12. Research on changes in elastic properties of rubber during ageing.
 - 3.13. Results of creep tests and their analysis.
 - 3.14. Research on changes in electrical properties of rubber during ageing.
 - 3.15. Research on changes in rubber infrared transmission capacity during ageing.

- 3.16. Research on relations between mechanical, electrical and optical properties of the rubber.
- 3.17. Conclusions of experimental research.
- 4. Method for forecasting mechanical properties and lifespan of rubber and its test in one-way tensile strain.
 - 4.1. Initial data obtaining.
 - 4.2. Determination of structuring energy, de-structuring energy, atomic structure constant and structuring constant.
 - 4.3. Forecasting of lifespan and maximum deformation in creep under constant tensile load.
 - 4.4. Comparison and analysis of numerical and experimental results.

Conclusions

Appendixes

- Appendix No.1 Standards for determination and forecasting physical properties of elastomers.
- Appendix No.2 Changes in rubber hardness during ageing.
- Appendix No. 3 Changes in rubber maximum tensile strain during ageing.
- Appendix No. 4 Changes in rubber breaking tension during ageing.
- Appendix No. 5 Changes in rubber flexible properties during ageing.
- Appendix No. 6 Changes in rubber electrical properties during ageing.
- Appendix No. 7 Changes in rubber infrared transmission capacity during ageing.
- Appendix No. 8 Corelations between mechanical, electrical and optical properties of the rubber.
- Appendix No. 9 Parameters Q_d , Q_s , x and a analysis program.
- Appendix No. 10 Program to forecast durability and maximum creep.
- Appendix No. 11 Rubber lifespan and maximum deformation in creep under constant tensile load by forecasting and experiments at various initial data.

Literature

Doctoral Thesis consists of: Introduction – 2 pages; Section 1 – 20 pages; Section 2 – 46 pages; Section 3 – 57 pages; Section 4 – 14 pages; Conclusions – 1 page; Appendixes – 66 pages; List of Literature – 12 pages; Total – 225 pages.

1. LITERATURE REVIEW

Thesis surveys structure, properties and models of rubber and studies standards and literature on determination of physical properties of elastomers as well as methods for forecasting mechanical properties and lifespan of elastomers.

The review of the literature highlighted the following conclusions:

- 1) existing methods for forecasting physical properties of elastomers and lifespan of rubber products are based only on mechanical parameters;
- 2) there is no mathematical model describing breakdown of rubber which is based on measuring dielectrical permeability;
- 3) existing models do not directly include the impact of destructive environmental elements (radiation, UV rays, ozone etc.) on the rubber;
- 4) developing models to describe rubber, it is expedient to follow the principle of hierarchical adaptive modeling;
- 5) method for determination of dielectric permeability of rubber was not found in the literature;
- 6) changes in dielectric permeability during ageing of rubber and its relation to rubber mechanical characteristics have not been researched;
- 7) changes in infrared transmission capacity of elastomers during ageing and its relation to mechanical characteristics of the elastomers have not been researched.

Based on literature review and conclusions drawn from it, following objectives have been set for the Thesis:

- 1) to test hypothesis about possibilities to use electrical and optical parameters in methods for forecasting mechanical properties and lifespan of elastomers;
- 2) to develop relevant technical provision and techniques to take dielectric measures of elastomer materials;
- 3) to research changes in dielectric permeability, specific resistance and infrared transmission capacity of rubber during ageing;
- 4) to inquire into possible correlations between electrical, optical and mechanical characteristics of rubber during ageing and to verify credibility of those correlations;
- 5) to construct mathematical model at different loads, which allows to find empirical correlation between mechanical properties of rubber and dielectric permeability;

- 6) using model as the base, to establish methods for forecasting mechanical properties and lifespan of elastomers;
- 7) to practically test methods for forecasting mechanical properties and lifespan of elastomers.

2. THEORETICAL RESEARCH

Mathematical model for one-way and two-way tensile strain, one-way and two-way compression strain, shear, torsion strains and dynamic tensile strain under compression is constructed. Model allows calculating lifespan of rubber products under a given load. It links mechanical, thermal, and electrical parameters of rubber, time and factors, that affect the rubber ageing – temperature and lead-in energy. Model does not provide possibility to solve problem analytically, but it can be used in numerical calculations. Model lets to reduce number of mechanical tests by replacing them with dielectric measurements which are easy to automate.

Methods for the determination of elastic potential under constant elongation, and of creep under constant tensile load are elaborated.

Let us have a brief look at mathematical model which describes breakdown of rubber in one-way tensile strain. In this model complicated structure of rubber is substituted with platelike structure (Figure 2.1.). Dimensions of platelike volume element (Figure 2.2.) Δy and Δz are small in comparison with the rubber sample, but big in comparison with the molecule of caoutchouc; dimension $\Delta x=x$ is called atomic structure constant, it is equal to distance between centres of contiguous atoms in unstrained conditions. Platelike volume elements are connected with links. Number of those links are the highest possible if rubber is new but it reduces as rubber ages or strains (Figure 2.1. and 2.3.). During deformation of the sample full mechanical load is taken only by links which are parallel to the direction of the strain (in given example those links are parallel to axle x). Moment of breakdown is at point when all links connecting two platelike elements are broken (Figure 2.4.).

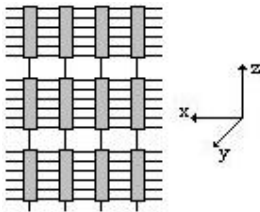


Figure 2.1. Platelike structure of new rubber

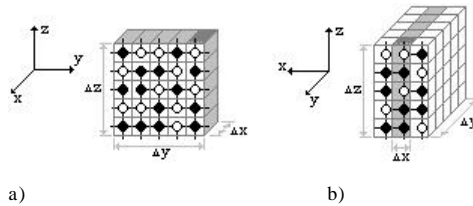


Figure 2.2. Platelike volume element

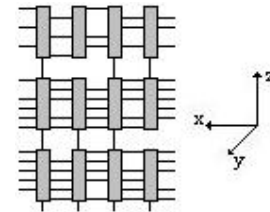


Figure 2.3. Breakdown of links during ageing

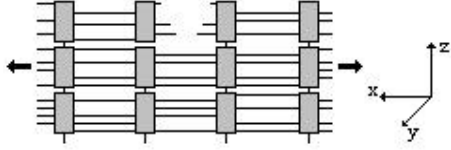


Figure 2.4. Breakdown of the sample in tensile strain

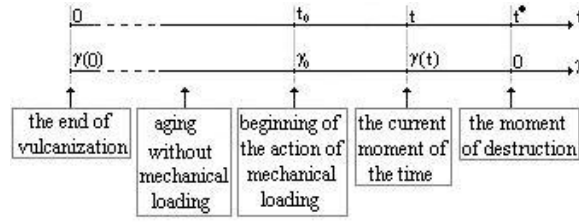


Figure 2.5. Cross-link yield at different moments in time

For quantitative characteristics of rubber ageing for actual platelike volume element we use entity γ which is called cross-link yield:

$$g(t) = \frac{N_1(t)}{N}, \quad (2.1)$$

where N_1 - number of links carrying tension load at moment in time t ; N - maximum number of links carrying tension load in platelike volume element (corresponds with moment in time $t=0$). Cross-link yield shows remaining part of number of links carrying maximum tension load at certain moment in time t . Its numerical values at different moments in time are shown in Figure 2.5.

Link is broken if it receives energy fluctuation which is bigger than energy needed to tear link (Q_d), let us call it de-structuring energy. Link can receive energy by heat flow from contiguous atoms or it can be received from aggressive environment in forms of chemically active substances or radiation. In addition to that a sample can be deformed mechanically. Together with de-structuring or link breaking process which results in diminishing of cross-link yield, process of structuring occurs – new links are created and it increases cross-link yield. In order to have a new link atom with unpaired electron has to receive energy Q_s , let us call it structuring energy. Model assumes that process of structuring and de-structuring of links complies with Boltzmann distribution. During changes in cross-link yield $d\gamma$, dt are set by differential equation:

$$dg(t) = \left[-g(t) \cdot e^{-\frac{Q_d(t)}{\frac{x^3 \cdot W(t)}{g(t)} + k \cdot T(t) + W_{star}(t) + W_{kin}(t)}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s(t)}{k \cdot T(t) + W_{star}(t)}} \right] \cdot Z \cdot n \cdot dt, \quad (2.2)$$

where W – elastic potential (potential energy of deformation received by one volume unit) of given platelike element, J/m^3 ; $k=1,23 \cdot 10^{-23} J/K$ - Boltzmann constant; T - absolute temperature, K ; a – structuring constant; $Z=6$ – number of the closest contiguous atoms in cubic structure; $\nu=1 \cdot 10^{13} Hz$ – frequency of thermal oscillations of atoms; t – time, s ; W_{star} -

average energy delivered by radiation to one link at certain moment in time t , J; $W_{k_{tm}}$ - average energy delivered by chemical reaction to one link, J; $x^3 \cdot W / \gamma$ - energy mechanically delivered to one link, J; $k \cdot T$ - energy of heat flow relevant to one link, J. The exponent of the first addend describes probability of emergence of energy fluctuation which can trigger a breaking of a link; exponent of the second addend describes probability of emergence of energy fluctuation which can trigger creation of a new link. Given differential equation is characterised by conditions at the beginning, at the moment of mechanical loading and at the end:

$$\begin{aligned} & \text{if } t=0, \text{ then } \gamma=1; \\ & \text{if } t=t_0, \text{ then } \gamma=\gamma_0; \\ & \text{if } t=t^*, \text{ then } \gamma=0. \end{aligned} \quad (2.3)$$

This differential equation applies to one particular platelike volume element. Because rubber product consists of big number of platelike volume elements, this model can be used to describe ageing process in a whole rubber product. It also allows describing anisotropic condition, which develops in rubber as a result of heterogeneous impact of ageing promoting agents. Ageing can be characterized by the field of cross-link yield γ which (in the same way as the fields of mechanical stress and temperature) can differ from the one part of a rubber product to another. At the place where the impact of ageing promoting agents (mechanical stress, temperature, radiation, chemicals), will be stronger, the field of cross-link yield will be weaker. Moment, when γ in same point of the rubber product (platelike volume element) becomes zero, can be considered as the destruction time of the sample.

For approximation of tensile strain under constant elongation with characteristic Thesis offers to use function

$$s_0 = q \cdot e^d, \quad (2.4)$$

where σ_0 - mechanical stress on cross-sectional area at unstrained conditions; ε - deformation; q , d - material constants. Elastic potential for non-compressible material (i.e. rubber) in general case is found as follows:

$$W = \int_0^e s_0 \cdot de. \quad (2.5)$$

Deformation in constant tension:

$$e = \frac{w \cdot t}{l_0}, \quad (2.6)$$

where l_0 - length of the work area of the sample put on tension test in unstrained conditions. Equations (2.4) - (2.6) give expression to calculate elastic potential under constant elongation:

$$W = \frac{q}{d+1} \cdot \left(\frac{w \cdot t}{l_0} \right)^{d+1}. \quad (2.7)$$

This analysis case when sample is subjected to creep under constant tensile load P_0 . Process of creep is divided into two parts: 1) sample is loaded until assigned P_0 value under constant elongation speed w , 2) creep as a result of constant tensile load P_0 . For approximation of deformation ϵ , from moment t_0 , when sample takes full load P_0 , is recommended to use function

$$e = e_0 \cdot \left[1 + b \cdot \langle t - t_0 \rangle^c \right], \quad (2.8)$$

where ϵ_0 - deformation relevant to the moment in time t_0 ; c, d – constants characterizing samples under creep test. The expression to calculate elastic potential in random moment in time t in this case is

$$W = \frac{q}{d+1} \cdot \left(\frac{s_0}{q} \right)^{\frac{d+1}{d}} + s_0 \cdot \left(\frac{s_0}{q} \right)^{\frac{1}{d}} \cdot b \cdot \left\langle t - \left(\frac{s_0}{q} \right)^{\frac{1}{d}} \cdot \frac{l_0}{w} \right\rangle^c, \quad (2.9)$$

where $\sigma_0 = P_0/F_0$ - mechanical stress; F_0 - cross-sectional area of the work area of the sample at unstrained conditions (perpendicular to direction of elongation).

If during ageing anisotropic conditions within rubber are not developed, i.e. rubber ages in uncompressed conditions, it is possible to include in model the differential equation with relative dielectric permeability χ , cross-link yield γ and time t

$$\frac{dC}{C^2} = x \cdot g(t) \cdot dt. \quad (2.10)$$

Coefficient ξ is material constant which characterises electrical properties of rubber and is linked to electrical field frequency ratio, which is used in dielectric permeability measurements. If during ageing relative dielectric permeability χ of rubber increases, then $\xi > 0$, if χ decreases, then $\xi < 0$.

Unfortunately practical use of equation (2.10) is difficult because of complicated function $\gamma = \gamma(t)$ (see equation (2.2)).

This is why it is more expedient right away to look for empirical relations between constants of elastic potential and relative dielectric permeability, which are included in model.

For instance:

$$q = q(c), \quad (2.11)$$

$$d = d(c), \quad (2.12)$$

$$b = b(c), \quad (2.13)$$

$$c = c(c), \quad (2.14)$$

$$c_i = c_i(c), \quad (2.15)$$

where $i= 1,2,3,4$ indexes relevant to material constants in Bidermann's expression of elastic potential [53]. Functions (2.11) - (2.15) allow to replace mechanical gauging with measurements of dialectical permeability which do not destroy sample and are easy to automate. In these function instead χ can be used specific resistance ρ and infrared transmission capacity T.

Mathematical models for other types of deformation are similar. Differences only exist in the formula of calculation of elastic potential and in assumption that cross-link yield varies in different directions of deformation.

3. EXPERIMENTAL RESEARCH

20 different types of rubber are used in the experimental research. Samples are subjected to artificial ageing (standard DIN 53508), tension tests (standard DIN 53504), tests for creep to failure under constant elongation, hardness tests by Shore A or Shore D (standards DIN EN ISO 868, DIN 53505). Measurements of dielectric permeability and specific resistance are taken by method which is developed on the bases of standard DIN ISO 2878. Apart from it easy automated method to take dielectric measurements on tyres is elaborated, changes in dielectric permeability during ageing for different types of tyres are investigated, measurements of infrared transmission capacity are taken (Perkin Elmer FT-IR Spectrometer Spectrum 1000) and existence of functional correlations between mechanical, electrical and optical parameters of rubber are established.

Electrodes (silver lacquer, fluids conducting electricity, graphite lubricant), which are applied on surface of elastomer material in experiments, are used to determine the numerical values of dielectric permeability and specific resistance, however it is difficult to apply them in the automatic measuring of dielectric permeability. Thesis offers solution to this problem by using capacitance sensors. The system of aluminum electrodes is applied to single-side of 3mm thick plate of organic glass (round or square) with dimensions 2...2,5cm. Then it is covered by approximately 0.2mm thick dielectric. This sensor is pressed with constant force to the surface of researched elastomers material, i.e. a sidewall of a car tyre, and at the moment in time 2 ± 1 s after pressing capacity of sensor is measured. In this case numerical value of dielectical permeability is not determined, but its changes in material can be judged by the capacity of the sensor which presents the evidence of increase or decrease in dielectric permeability of the material. Capacity is measured with multimeter Fluke 189. In order to exclude interference of capacity of delivery wire on results of measurements, capacity of sensor is measured twice. First, capacity of sensor C is measured when it is pressed on the surface of the tyre. After that sensor is taken off the tyre and capacity C_0 is taken again. Capacity increase, which is created by rubber in comparison with air, is calculated

$$\Delta C = C - C_0. \quad (3.1)$$

Measuring is repeated 20 times on the same tyre in different places along perimeter and average ΔC is calculated.

The following is the examples of the results of experimental tests. Diagrams indicate the interval of measurement error as well. Full information about results of experiments is found in Doctoral Thesis and its appendixes.

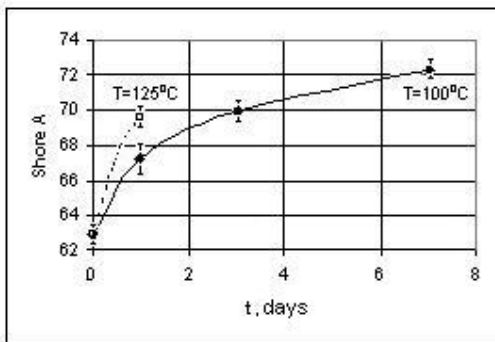


Figure 3.1. Change in hardness during ageing at 100⁰C and 125⁰C. Rubber No.2.

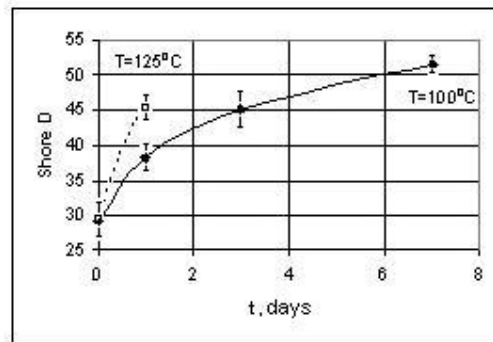


Figure 3.2. Change in hardness during ageing at 100⁰C and 125⁰C. Rubber No.9.

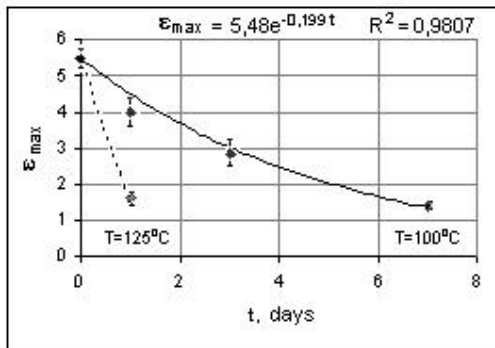


Figure 3.3 Change in maximum deformation during ageing. Rubber No.1. Temperature of ageing - 100⁰C and 125⁰C, Rate of elongation - 200mm/min

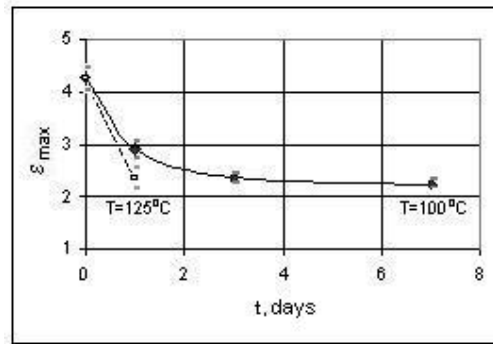


Figure 3.4. Change in maximum deformation during ageing. Rubber No.2. Temperature of ageing - 100⁰C and 125⁰C, Rate of elongation - 200mm/min

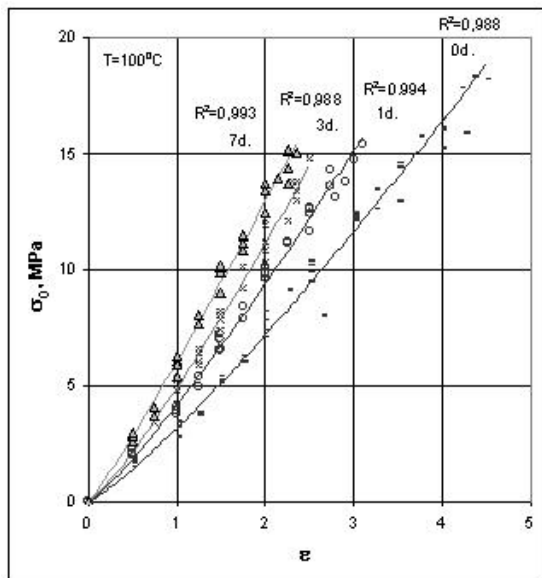


Figure 3.5. Elongation characteristics in different times at 100⁰C for aged rubber Nr.2. Rate of elongation - 200mm/min; 0d- new rubber; 1d, 3d, 7d- 1, 3 and 7 days at 100⁰C temperature aged rubber; function used for approximation (2.4); R²- coefficient of determination

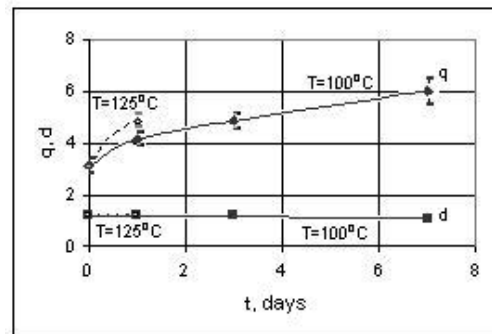


Figure 3.6. Change in elastic properties parameters q and d during ageing at 100⁰C and 125⁰C temperatures. Rubber No.2.

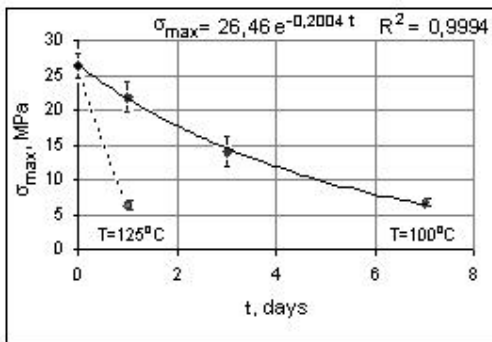


Figure 3.7. Change in breaking tension during ageing. Rubber No. 1.
Temperature of ageing - 100°C and 125°C,
Rate of elongation - 200mm/min

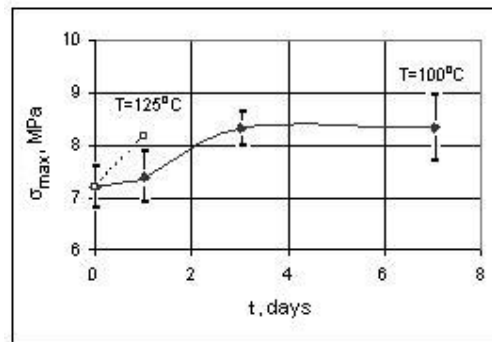


Figure 3.8. Change in breaking tension during ageing. Rubber No. 4.
Temperature of ageing - 100°C and 125°C,
Rate of elongation - 200mm/min

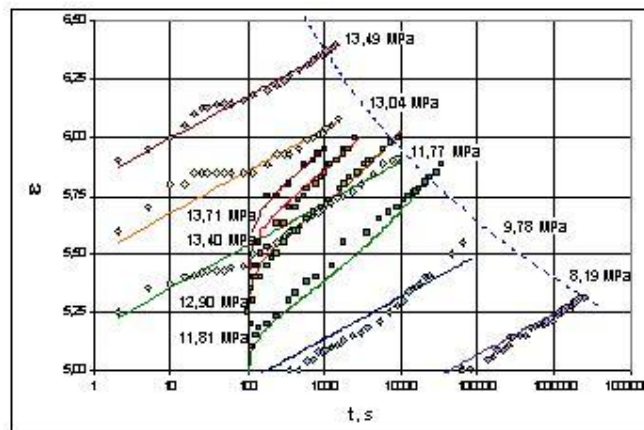


Figure 3.9. Deformation in correlation with time in creep under different tensile loads at two different loading rates. Rubber No.14.
Loading rates: $w=0,10\text{m/s}$ (rhombs, straight parallels) and $w=0,96\text{mm/s}$ (squares, curves in middle part of diagram); dots show experimental data, lines – graphs of approximation functions; relative measurement error: deformation - 1,2%; tension - 6,8%; interrupted line on the right side of experimental curves shows lifespan t^* at given tensile load and loading rate $w=0,10\text{m/s}$; tension given against unstrained area; creep test temperature 15°C

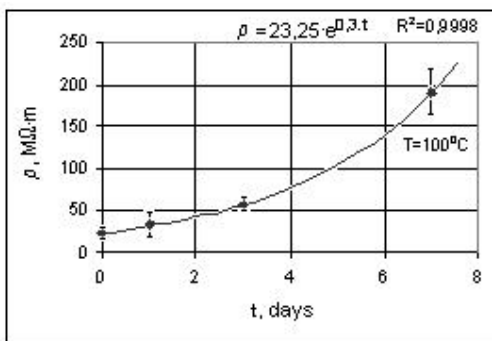


Figure 3.10. Change in specific resistance during ageing at 100°C temperature.
Rubber No. 1
Measured with multimeter Fluke 189,
voltage- 6V

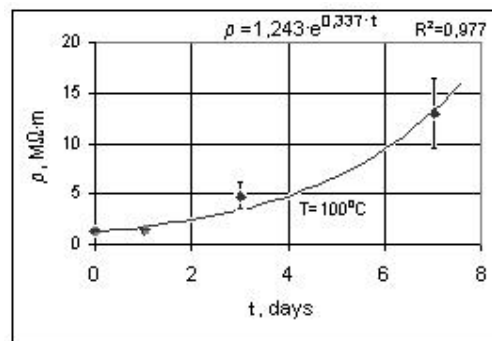


Figure 3.11. Change in specific resistance during ageing at 100°C temperature.
Rubber No. 1
Measured with Sefelec megohmmeter
Megohmmetre M1500P, voltage - 1000V

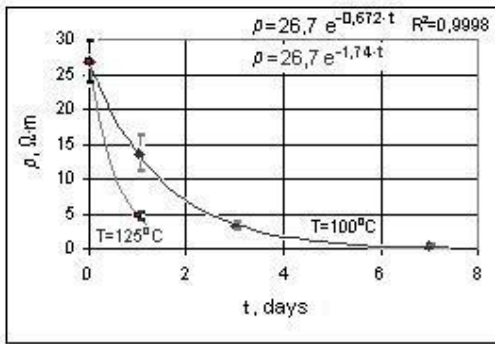


Figure 3.12. Change in specific resistance during ageing at 100⁰C and 125⁰C temperature. Rubber No. 9. Measured with multimeter Fluke 189

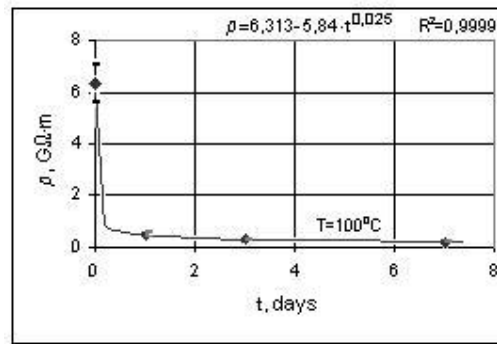


Figure 3.13. Change in specific resistance during ageing at 100⁰C temperature. Rubber No. 14. Measured with Sefelec megohmmeter Megohmmetre M1500P

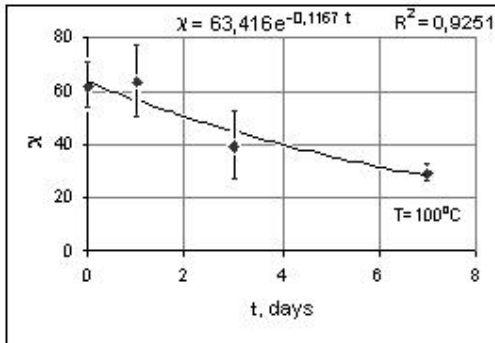


Figure 3.14. Change in relative dielectric permeability during ageing at 100⁰C temperature. Rubber No. 1.

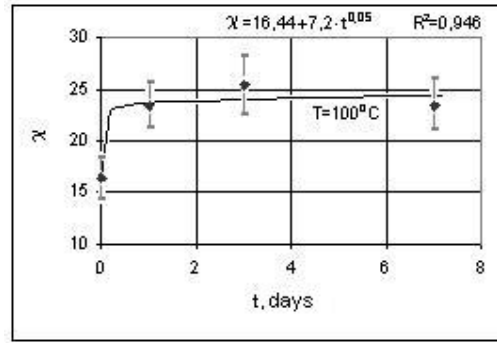


Figure 3.15. Change in relative dielectric permeability in during ageing at 100⁰C temperature. Rubber No. 14.

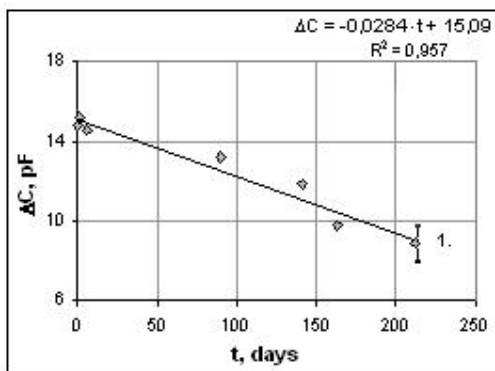


Figure 3.16. Change in sensor capacity increase ΔC in winter tyres Marshal, stored in warehouse

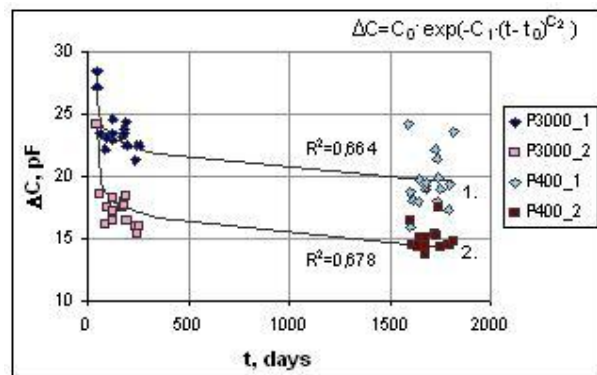


Figure 3.17. Change in sensor capacity increase ΔC in winter tyres Pirelli during sustained ageing at regular operational conditions. 2 different sensors are used; 1st sensor: $C_0=28,4\text{pF}$, $C_1=0,089$, $C_2=0,19$, $t_0=47$ days; 2nd sensor: $C_0=21,4\text{pF}$, $C_1=0,126$, $C_2=0,19$, $t_0=48$ days

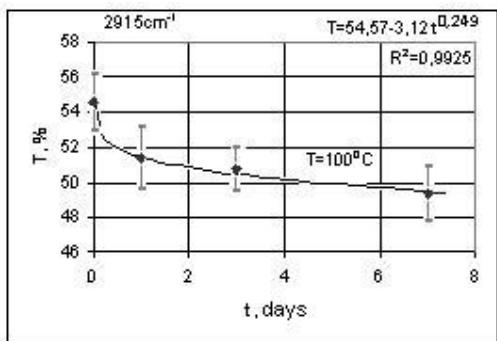


Figure 3.18. Change in infrared transmission capacity at 2915cm^{-1} during ageing at 100°C temperature. Rubber No.1.

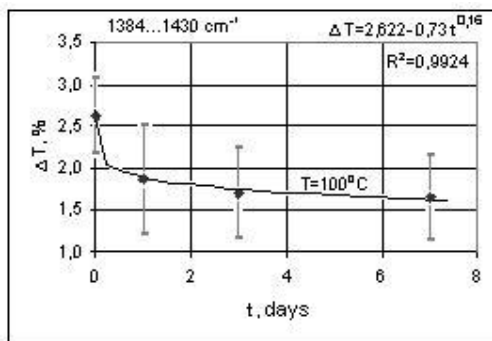


Figure 3.19. Changes in infrared transmission capacity at 2867cm^{-1} and 2915cm^{-1} during ageing at 100°C temperature. Rubber No.1

Correlations between hardness, electrical and optical characteristics of rubber No. 1 are shown in diagrams 3.20.- 3.23.

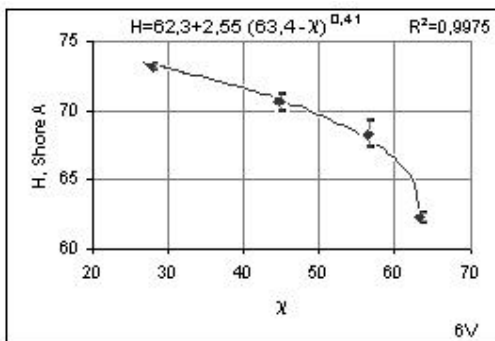


Figure 3.20. Correlation between hardness and relative dielectric permeability. Rubber No. 1

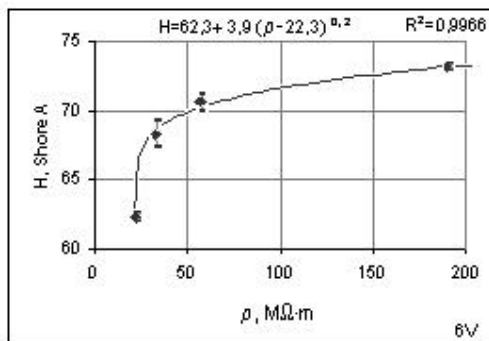


Figure 3.21. Correlation between hardness and specific resistance. Rubber No. 1

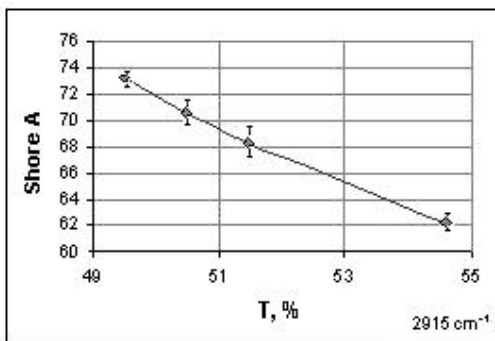


Figure 3.22. Correlation between hardness and infrared transmission capacity at 2915cm^{-1} . Rubber No. 1.

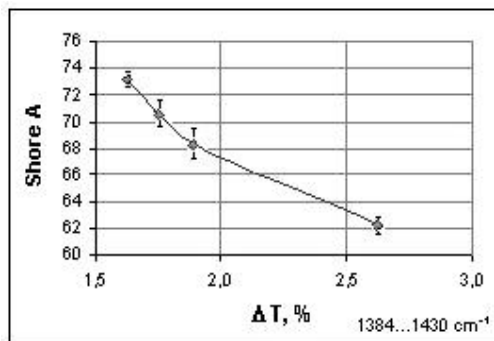


Figure 3.23. Correlation between hardness and infrared transmission capacity in the range of $1384\text{...}1430\text{cm}^{-1}$. Rubber No. 1.

Correlations between maximum deformation and electrical as well as optical characteristics of rubber No. 1 are shown in diagrams 3.24. – 3.27.

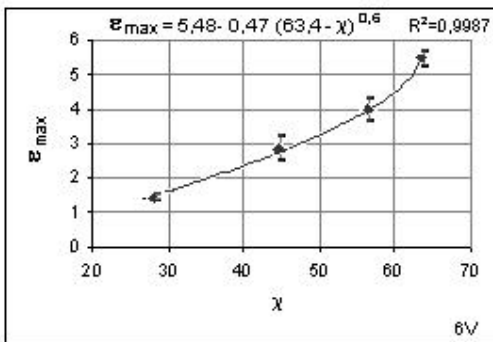


Figure 3.24. Correlation between maximum deformation and relative dielectric permeability. Rubber No. 1.

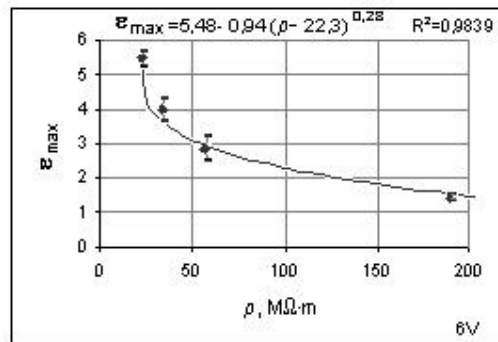


Figure 3.25. Correlation between maximum deformation and specific resistance. Rubber No. 1.

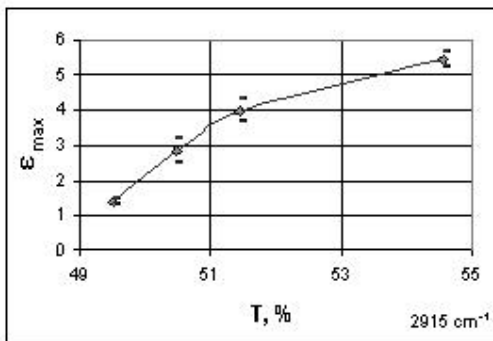


Figure 3.26. Correlation between maximum deformation and infrared transmission capacity at 2915cm⁻¹. Rubber No. 1

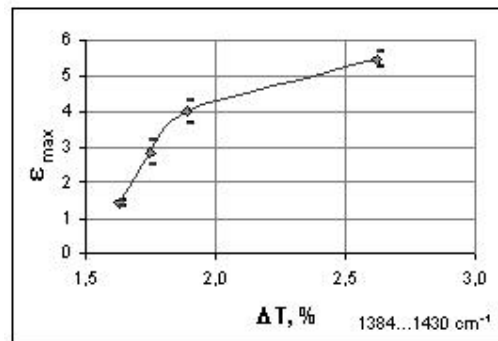


Figure 3.27. Correlation between maximum deformation and infrared rays transmission capacity in the range of 1384... 1430cm⁻¹. Rubber No. 1

Correlations between breaking tension and electrical, optical characteristics of rubber No. 1 are shown in diagrams 3.28. – 3.31.

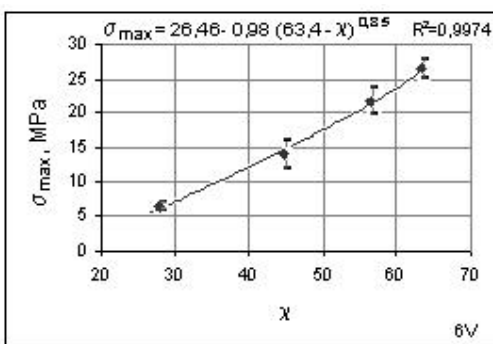


Figure 3.28. Correlation between breaking tension and relative dielectric permeability. Rubber No. 1

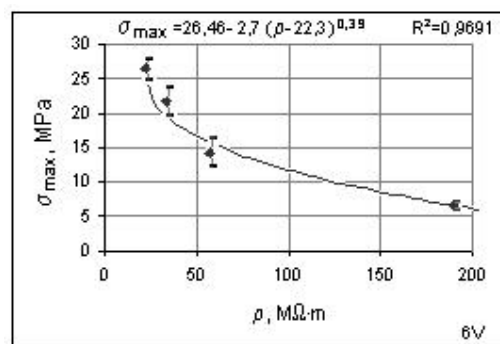


Figure 3.29. Correlation between breaking tension and specific resistance. Rubber No. 1

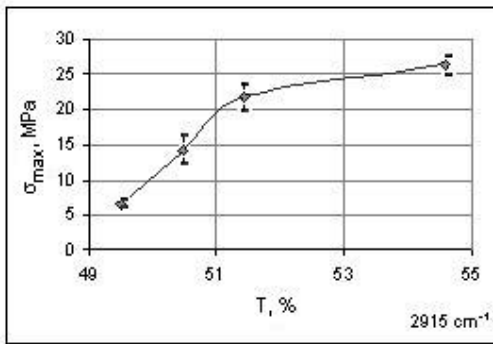


Figure 3.30. Correlation between breaking tension and infrared transmission capacity at 2915cm^{-1} . Rubber No. 1

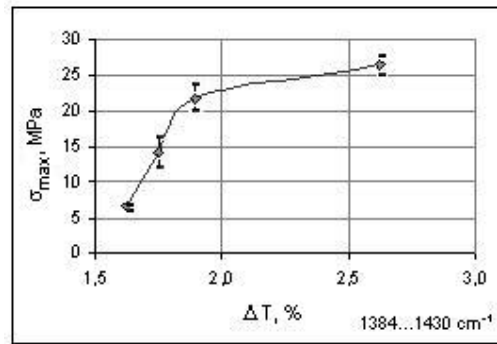


Figure 3.31. Correlation between breaking tension and infrared rays' transmission capacity in the range of $1384...1430\text{cm}^{-1}$. Rubber No. 1

Correlations between parameters of elastic properties q , d and electrical, optical characteristics of rubber No. 1 are shown in diagrams 3.32.- 3.39.

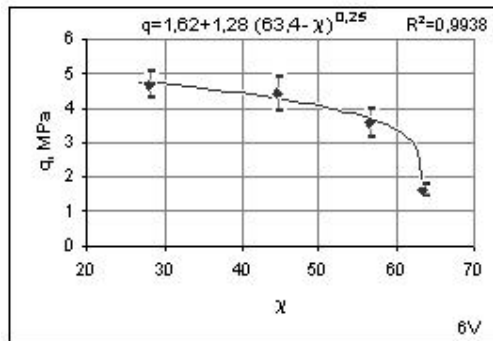


Figure 3.32. Correlation between parameter q of elastic properties and relative dielectric permeability. Rubber No.1.

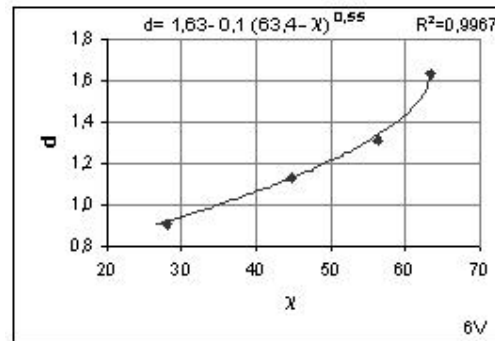


Figure 3.33. Correlation between parameter d of elastic properties and relative dielectric permeability. Rubber No.1.

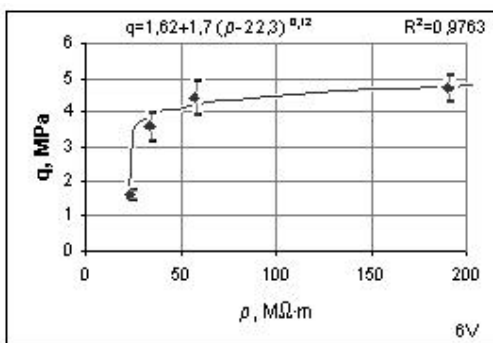


Figure 3.34. Correlation between parameter q of elastic properties and specific resistance. Rubber No.1.

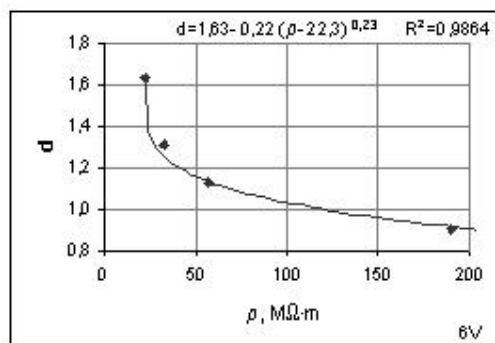


Figure 3.35. Correlation between parameter d of elastic properties and specific resistance. Rubber No.1.

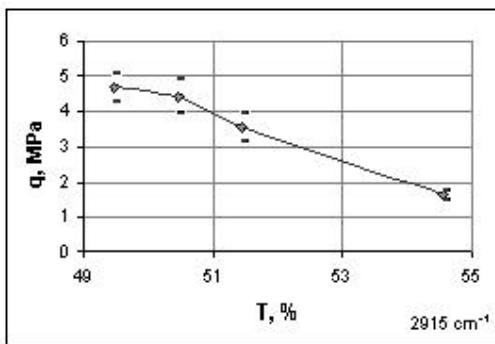


Figure 3.36. Correlation between parameter q of elastic properties and infrared transmission capacity at 2915cm^{-1} . Rubber No. 1

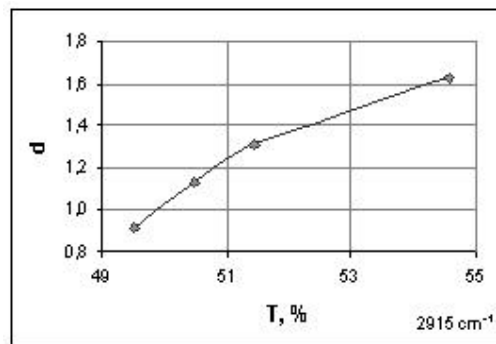


Figure 3.37. Correlation between parameter d of elastic properties and infrared transmission capacity at 2915cm^{-1} . Rubber No. 1

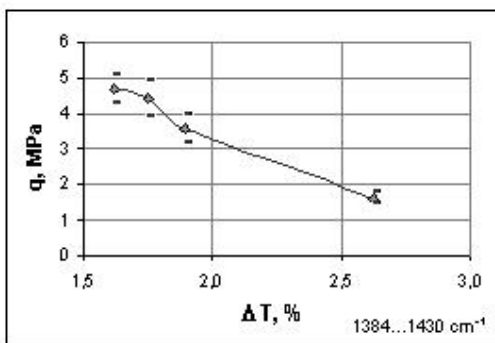


Figure 3.38. Correlation between parameter q of elastic properties and infrared transmission capacity in the range of $1384\dots 1430\text{cm}^{-1}$. Rubber No. 1.

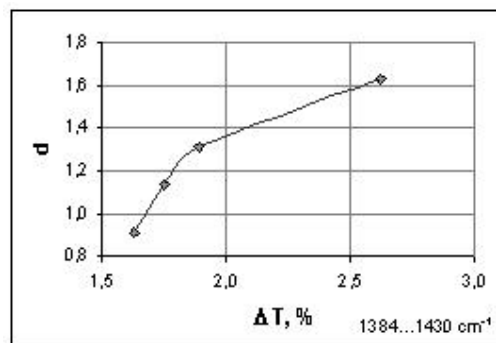


Figure 3.39. Correlation between parameter d of elastic properties and infrared rays' transmission capacity in the range of $1384\dots 1430\text{cm}^{-1}$. Rubber No. 1.

Correlations between maximum deformation, breaking tension and hardness of rubber No.1 are shown in diagrams 3.40.- 3.41.

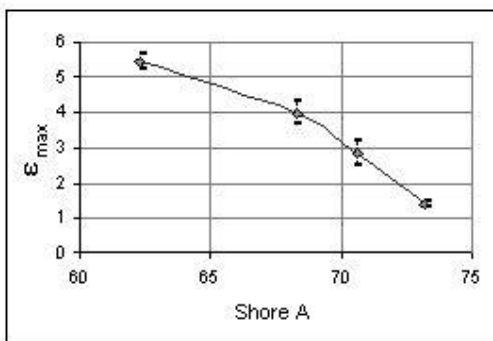


Figure 3.40. Correlation between maximum deformation and hardness. Rubber No.1.

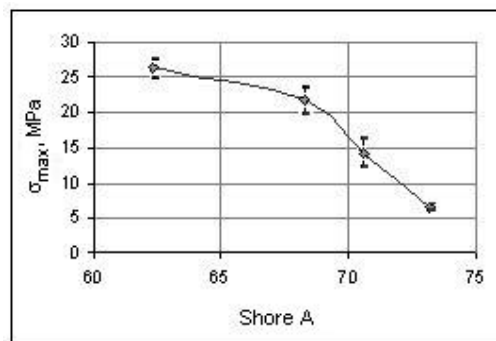


Figure 3.41. Correlation between breaking tension and hardness. Rubber No.1.

Experimental research allows drawing following conclusions.

As rubber ages, its mechanical (hardness, breaking tension, maximum deformation, stiffness), electrical (dielectric permeability, specific resistance) and optical (infrared transmission capacity, the differences of infrared transmission capacity in different spectral ranges) properties change. There are stochastic correlations between mechanical and electrical or mechanical and optical parameters. This allows replacement of mechanical tests with electrical or optical measures.

The approximation of elongation characteristics is recommended to perform with formula (2.4); in case of the approximation of deformation in creep under constant elongation adequate results are obtained with expression (2.8).

The diagram of creep under constant tensile load and given rate of loading (Figure 3.9) can be used to forecast deformation at any taken moment in time, maximum deformation and the lifespan of a sample.

Technical provision and methods to measure dielectric permeability on elastomers are developed. Dielectric permeability must be measured in the standard interval of time after voltage is connected to electrodes, because value of measuring capacity with time increases almost without limits. Standard voltage values must be used (Figure 3.10. and 3.11.). For taking dielectric measurements optimum is low ($<100\text{Hz}$) frequencies of electrical field. For rubber with high electrical resistance ($\text{G}\Omega\cdot\text{m}$, $\text{M}\Omega\cdot\text{m}$) dielectric permeability must be measured, but for rubber with low electrical resistance ($\Omega\cdot\text{m}$), measuring of electrical resistance must be done. In that way it is possible to provide electrical measurements with low (up to 10V) voltage.

During ageing dielectric permeability of Pirelli and Marshal tyres reduces. In methods to determine and to forecast mechanical parameters of tyres electrical measurements with capacitance sensors can be taken. Tyres and storage must not be exposed to high temperatures during exploitation or electrical measurements will not give correct values of mechanical characteristics. Electrical measuring must no be performed on wet tyres.

In methods for determination and forecasting mechanical properties of elastomers which use non-mechanical measurements for higher accuracy of the results it is advisable to combine different measurements - electrical (dielectric permeability, specific resistance) and optical (infrared transmission capacity at different wavelengths, difference of transmission capacity in different spectral ranges).

4. METHOD TO FORECAST MECHANICAL PROPERTIES AND LIFESPAN OF RUBBER AND ITS EXPERIMENTAL TEST IN ONE WAY TENSILE STRAIN

In order to forecast mechanical properties and lifespan of rubber in the beginning it is necessary to obtain initial data. To do this it is proposed to take samples of new rubber and rubber aged 24, 72 and 168 hours at 100⁰C in air. For these samples with different levels of ageing elongation characteristics are produced and doing approximation with formula (2.4.) using method of the least square parameters q and d of elastic properties are determined. Breaking time in elongation t* is measured for each sample. Then average rate of elongation w, with which work area border marks separate, are measured. Temperature T, at which tension tests are performed, ageing temperature T_n and ageing time t_n are taken. It is experimentally established that the biggest relative measurement errors are related to determination of breaking time t* and parameter q of elastic properties. This is why measurement error of these characteristics Δq and Δt* further are used in forecasting method. It is regarded that parameter d is absolutely accurate because error of this parameter is included in measurement error of parameter q. Relative measurement error for other characteristics - l₀, w, T, T_n, t_n, is much smaller compared to measurement errors of t* un q, therefore average values of these characteristics are used in forecasting method, leaving errors of measurement out.

If functional correlations between mechanical parameters q, d, t* and electrical (χ, ρ, ΔC) or optical (T, ΔT) are known, then electrical or optical measures can be used for determination of mechanical parameters of given type of rubber.

Forecasting method requires knowing values of de-structuring energy Q_d, structuring energy Q_s, atomic structure constant x and structuring constant a. Assuming, that during ageing the values of these parameters are constant. To determine their values will use correlation (2.2), where 4 cases of elongation under constant rate and 3 cases of artificial ageing without mechanical load can be written down as equation system:

$$0 = g_0 + \int_0^{t_0^*} \left[-g(t) \cdot e^{-\frac{Q_d}{\frac{x^3 \cdot W(t)}{g(t)} + k \cdot T}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T}} \right] \cdot Z \cdot n \cdot dt, \quad (4.1)$$

$$g_1 = g_0 + \int_{t_{n0}}^{t_{n1}} \left[-g(t) \cdot e^{-\frac{Q_d}{k \cdot T_n}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T_n}} \right] \cdot Z \cdot n \cdot dt, \quad (4.2)$$

$$0 = g_1 + \int_0^{t_1^*} \left[-g(t) \cdot e^{-\frac{Q_d}{\frac{x^3 \cdot W(t)}{g(t)} + k \cdot T}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T}} \right] \cdot Z \cdot n \cdot dt, \quad (4.3)$$

$$g_3 = g_1 + \int_{t_{n1}}^{t_{n3}} \left[-g(t) \cdot e^{-\frac{Q_d}{k \cdot T_n}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T_n}} \right] \cdot Z \cdot n \cdot dt, \quad (4.4)$$

$$0 = g_3 + \int_0^{t_3^*} \left[-g(t) \cdot e^{-\frac{Q_d}{\frac{x^3 \cdot W(t)}{g(t)} + k \cdot T}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T}} \right] \cdot Z \cdot n \cdot dt. \quad (4.5)$$

$$g_7 = g_3 + \int_{t_{n3}}^{t_{n7}} \left[-g(t) \cdot e^{-\frac{Q_d}{k \cdot T_n}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T_n}} \right] \cdot Z \cdot n \cdot dt, \quad (4.6)$$

$$0 = g_7 + \int_0^{t_7^*} \left[-g(t) \cdot e^{-\frac{Q_d}{\frac{x^3 \cdot W(t)}{g(t)} + k \cdot T}} + a \cdot (1-g(t)) \cdot e^{-\frac{Q_s}{k \cdot T}} \right] \cdot Z \cdot n \cdot dt. \quad (4.7)$$

Equation (4.1) describes process of elongation at constant rate to failure for new rubber. In this process cross-link yield changes from $\gamma_0=1$ (new rubber) to $\gamma=0$ (time of failure). t_0^* designates time in which sample is destroyed.

Equation (4.2) describes process of rubber ageing at elevated temperature T_n without mechanical load in period of time $t_{n0}=0$ to $t_{n1}=24$ hours. During this process cross-link yield changes from $\gamma_0=1$ to γ_1 .

Equation (4.3) describes process of elongation at constant rate to failure of rubber which has been aged for 24 hours at elevated temperature T_n (temperature during elongation tests is T). Cross-link yield of rubber before elongation test is γ_1 , at the moment of failure - $\gamma=0$. t_1^* designates time in which sample is destroyed.

Equation (4.4) describes process of ageing of rubber at elevated temperature T_n without mechanical load during period of time from $t_{n1}=24$ hours to $t_{n3}=72$ hours. Cross-link yield during this process changes from γ_1 to γ_3 .

Equation (4.5) describes process of elongation at constant rate to failure (temperature T) of rubber which has been aged for 72 hours at elevated temperature T_n . Cross-link yield of rubber before elongation test is γ_3 , at the moment of failure - $\gamma=0$. t_3^* designates time of destruction of the sample.

Equation (4.6) describes process of ageing of rubber at elevated temperature T_n without mechanical load during period of time from $t_{n3}=72$ hours to $t_{n7}=168$ hours. Cross-link yield during this process changes from γ_3 to γ_7 .

Equation (4.7) describes process of elongation at constant rate to failure (temperature T) of rubber which has been aged for 168 hours at elevated temperature T_n . Cross-link yield of rubber before elongation test is γ_7 , at the moment of failure - $\gamma=0$. t_7^* designates time in which sample is destroyed.

Function $W(t)$ of elastic potential contained in equations (4.1), (4.3), (4.5), (4.7) for each level of rubber ageing with elongation at the constant rate can be found by putting relevant constants q and d , rate of elongation w and the length of sample work area in unstrained conditions l_0 in equation (2.7). Consequently we can assume that function $W(t)$ in equations (4.1), (4.3), (4.5), (4.7) is known.

System of equations (4.1) – (4.7) has 7 independent equations with 7 unknown quantities Q_d , Q_s , x , a , γ_1 , γ_3 , γ_7 . It means that system has unequivocal solution. Unfortunately the analytical solving of such equation system is practically impossible. Therefore it is solved using numerical methods. Thesis elaborates numerical calculation algorithm for parameters Q_s , Q_d , x and a as well as relevant software in programming language C++ (Appendix 9).

Let us have a look at case when rubber sample is subjected to creep under constant tensile load P . Process can be described by equation (2.2) which in this case look like following:

$$0 = g_0 + \int_0^{t_m} \left[-g(t) \cdot e^{-\frac{Q_d}{g(t) + k \cdot T}} + a \cdot (1 - g(t)) \cdot e^{-\frac{Q_s}{k \cdot T}} \right] \cdot Z \cdot n \cdot dt, \quad (4.8)$$

where γ_0 - cross-link yield before loading, t_m - maximum lifespan of sample in creep, W – elastic potential in creep which is found with formula (2.9.). Parameters c and d , which characterise sample, are found with creep test under constant tensile load; deformation ε is approximated by function (2.8) using the method of the least square. The longer sample is subjected to creep test under constant tensile load the more accurate are parameters c and d . To determine parameters q and d it is necessary to perform elongation tests or to take electrical/optical measurements depending which ones from correlations $q=q(\chi)$, $d=d(\chi)$, $q=q(\rho)$, $d=d(\rho)$, $q=q(T)$, $d=d(T)$, $q=q(\Delta T)$, $d=d(\Delta T)$ are known. Other measurements taken from sample subjected to creep are: cross sectional area of work area F_0 and length l_0 in unstrained conditions as well as temperature T during creep, tensile load P , speed w with which in period of time - from the moment when mechanical loading starts until full load - work area border marks separate from each other. With this all initial data necessary for forecasting are collected. Equation (4.8) gives opportunity to determine the lifespan t_m of

the sample in creep under constant tensile load, but equation (2.8) – maximum deformation. In Thesis this calculations are done numerically, but software in programming language C++ is added in Appendix 10. The comparison of numerical and experimental results of lifespan and maximum deformation are shown in diagrams 4.1 and 4.2. Rhombs show the values of highest probability of lifespan and maximum deformation, small lines – range of forecasted error interval; uninterrupted line characterises experimental values, ideal congruence between forecasted and experimental results. As shown, all values of lifespan and maximum deformation are within forecasted error interval.

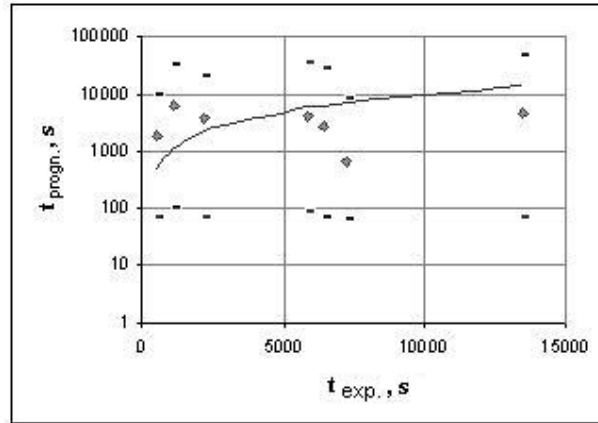


Figure 4.1. Comparison of forecasted and experimentally determined lifespans of rubber sample No.4 in creep under constant tensile load

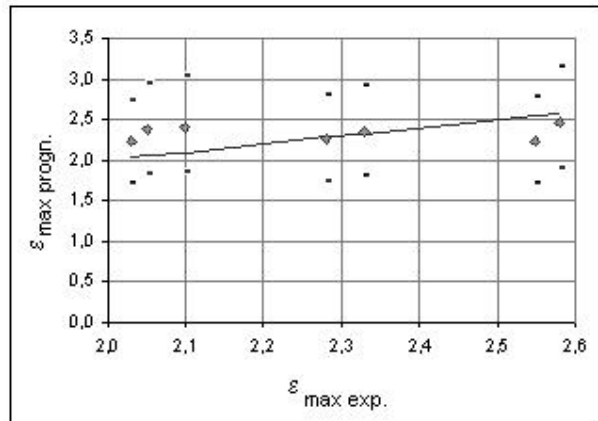


Figure 4.2. Comparison of forecasted and experimentally determined values of maximum deformation of rubber sample No.4 in creep under constant tensile load

CONCLUSIONS

1. Thesis experimentally proves hypothesis that there are possibilities to use measurements of dielectric permeability, specific resistance and infrared transmission capacity in forecasting of mechanical properties and durability of elastomers.
2. Mathematical model for different types of rubber deformation - one-way and two-way tensile strain, one-way and two-way compression strain, shear, torsion, dynamic tensile strain under compression - is constructed. Model allows calculating lifespan of rubber product at given load. It links mechanical, thermal, and electrical parameters of rubber, time and elements which affect ageing of rubber – temperature and lead-in energy.
3. Technical provision and method for taking dielectric measurements of elastomers are developed.
4. Changes in dielectric permeability, specific resistance and infrared transmission capacity in rubber during ageing at elevated temperatures are investigated. At the same time changes in mechanical properties – hardness, breaking tension, maximum deformation and elastic properties are researched.
5. The existence of stochastic correlations between mechanical properties of elastomers – hardness, maximum deformation, breaking tension, modulus of elastic properties and dielectric permeability, specific resistance and infrared transmission capacity (or differences of infrared transmission capacity in different spectral ranges). It gives opportunity to determine mechanical properties of elastomers with electrical and optical measurements which do not have damaging impact on rubber and are easy to automate.
6. Method for forecasting mechanical properties and lifespan of elastomers in creep under constant one-way tensile load is developed and experimentally tested. Electrical and optical measurements can be used to obtain initial mechanical parameters necessary for forecasting method. At the same time use of electrical/optical parameters helps increase accuracy of determination of mechanical parameters and consequently improve the quality of forecast.

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280. ГОСТ 8.544-86. Государственная система обеспечения единства измерений. Относительная диэлектрическая проницаемость и тангенс угла потерь твердых диэлектриков. Методика выполнения измерений в диапазоне частот от 10 в ст. 9 до 10 в ст. 10 Гц.
281. ГОСТ Р МЭК 811-5-1-95. Специальные методы испытаний герметизирующих составов электрических кабелей. Температура колебания. Масловыделение. Хрупкость при низкой температуре. Общее кислотное число. Отсутствие коррозионно-активных компонентов. Диэлектрическая проницаемость при 23 град. С. Условное электрическое сопротивление при 23 и 100 град. С.

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**DOCTORAL THESIS SUBMITTED TO OBTAIN
DOCTOR'S DEGREE IN ENGINEERING
RIGA TECHNICAL UNIVERSITY**

Doctoral Thesis is presented publicly in 25 April 2006 in auditorium No 310, at No. 6 Ezermalas street, Faculty of Transport and Mechanical Engineering of Riga Technical university

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CONFIRMATION

I confirm that I have written Doctoral Thesis which is submitted to evaluate in Riga Technical University for obtaining doctor's degree. Doctoral Thesis is not submitted in any other university in order to obtain scientific degree.

Andris Martinovs.....

Date.....

Doctoral Thesis is written in Latvian, it consists of introduction, 4 parts, conclusions, list of literature, 11 appendixes, 112 diagrams and pictures, in total – 225 pages. List of literature has 285 titles.

