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GUMFERENCE

**„Implementation of newest research
Into rubber industry“**

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Conference proceeding



Tomas Bata University in Zlín
Centre of Polymer Systems

2019

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Preamble

Rubber product manufacturers and raw materials suppliers seeking improved materials for next-generation products depend on predictive lab tests to estimate in-product performance. Ideally, predictive lab tests should balance accuracy, relevance and instrument productivity. They should connect to fundamental principles that show how results from a simple sheet of cured rubber are related to realistic geometry and loading conditions that occur in actual rubber product use. Rapidly advancing simulation technology is one way to do this, and it has created new opportunities to link lab testing of materials to real-world rubber product performance. Rubber product durability respective lifetime and lightweighting, in particular, stand to gain as experimental and simulation methods become more capable and better integrated.

The main aim of the conference Gumference 2019 is to introduce to science and industry the newest development applicable in to the real-world rubber product to gain complex information of the material in an early stage of development before rubber products have been produced or even before designs are finalized. The goal is to support the rubber industry to fasten development times and minimize extensive real rubber product test before production.

In Zlín, 13.11.2019
Radek Stoček

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NUMERICAL REPRESENTATION OF THE LIFE CYCLE OF RUBBER COMPONENTS: FROM UNVULCANIZED RUBBER TO VULCANIZED RUBBER AT ITS ULTIMATE SERVICE STATE

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ABSTRACT

Numerical analysis tools become more and more important in our today's world. With the help of numerical methods for multi-field and multi-scale problems, processes and features of rubber materials and components are virtually described to make them accessible for further directed optimization, assessment and monitoring, e.g. in an industrial context. In the past, different model strategies and numerical procedures have been used for each period of the component's service life to virtually best represent its features and objective quantities. However, loss of information and accuracy often occurred during each model change and several stages (e.g. manufacturing) of the whole life cycle of the rubber component remained unassessed. Furthermore, human interaction and additional working hours were required to reformulate and adapt the description for each considered period of the component's service life.

In this contribution, an attempt is made to numerically capture the whole life cycle of a rubber component (tire) from its manufacturing state to its failure state within a consistent virtual representation of the component. Especially the manufacturing state of the rubber component is considered in detail. To capture the transition in the thermo-mechanical properties of unvulcanized rubber during the vulcanization process, a thermo-mechanical two-phase material model in the context of finite strain thermo-mechanics within the finite element method (FEM) has been developed. The isochoric part of the model is formed by a hyperelastic spring and a dashpot device in series, where the properties of the dashpot device depend on the degree of vulcanization and temperature. This dependency allows to capture inelastic isochoric deformations during the forming and vulcanization process. The vulcanization rate is given by a constitutive approach and models the monotonic increase of the degree of vulcanization as a function of current temperature and material history. The volumetric part of the model is formed by a hyperelastic spring and allows to account for volume changes due to mechanical loads and temperature variations (thermal expansion). The material model is implemented into the thermo-mechanically coupled framework of the FEM in 3D for finite strains and represents the starting point of the numerical representation and assessment of the life cycle of the rubber component.

ACKNOWLEDGEMENT

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CHANCES AND CHALLENGES OF CLOSING THE MATERIAL CIRCLE FOR PASSENGER CAR TIRE RUBBER

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ABSTRACT

Sustainability recently became one of the focus areas of material and product development in the elastomer world, including the tire industry. Worn-out-tires pose quantitatively as well as qualitatively the largest problem, but at the same time also the biggest challenge and potential: If the concentration of recycled rubber in tires can be increased significantly, the amount of reused rubber will increase considerably. The challenge is to develop a recycling technology, which produces a recycled rubber of high quality, high enough to fulfill the specifications for use in tires. One of the problems lies in the homogeneity and purity of the feedstock: different types of tires containing different compounds, external contaminations such as stones attached to the tire tread, and internal ones such as reinforcing fibers.

The most promising technology for a high quality recycled rubber is devulcanization. In this process, in the ideal case, only the sulfur crosslinks will be broken, resulting in a processable rubber with properties comparable to the ones of the feedstock.

An extensive study was done to devulcanize different types of feedstock and determine the quality of the resulting products. The feedstock was varied in terms of age of the tires, single tire parts versus whole tires, particle size of the granulated tire rubber, carbon black versus silica filled rubber, and different silica-silane combinations. In terms of the devulcanization process, pre-treatment of the ground tire rubber, type of process as well as processing time are discussed. Besides, the analysis of the recycled rubber will be discussed.

Finally, the sum of these considerations will result in an overview of the challenges and chances of devulcanization and reuse of whole tire rubber.

THE EFFECT OF TEMPERATURE AND PRESSURE ON TIME-DEPENDENT PROPERTIES OF RUBBER-LIKE MATERIALS

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ABSTRACT

Polymeric materials are used in various applications where they are subjected to different boundary conditions, such as temperature, pressure, humidity, UV radiation, etc.. Pressure affects polymers in the solid state as well as in the molten state (e.g., during polymer processing). The glass transition temperature is shifted when the material is subjected to hydrostatic pressure. A similar effect is observed for melting – the polymer under higher pressure undergoes the phase transition from solid to liquid state at higher temperature than at room conditions. Such effects are very important for the polymer processing industry, where significant pressures are applied to polymer melts and to the solidified polymeric parts during injection molding or extrusion.

Engineering applications, where temperature and pressure play a significant role in material behavior, are, for example, sealing of pipes, insulation of deepwater pipes, deepwater cabling, etc.

In this presentation we discuss the subject and present two advanced experimental setups for studying simultaneous effect of pressure and temperature on fundamental mechanical properties, i.e. shear and bulk properties in time and frequency domain. Furthermore, we will also briefly go through linear and non-linear viscoelastic models, i.e. Fillers-Moonan-Tschoegl (FMT) model and Knauss-Emri (KE) model, for predicting long-term behaviour of polymers under combined effect of temperature and pressure.

2. Experimental setups

2.1. CEM Measuring System - CMS

A unique apparatus, called CMS (CEM Measuring System) was developed to study the combined effects of temperature and pressure on mechanical behaviour of polymers, Figure 1. The measuring system can measure volumetric (bulk) and shear properties of solid polymer specimens through wide range of temperatures (from -20°C to +120°C) and pressures (from atmospheric to 4000 bar) [2].

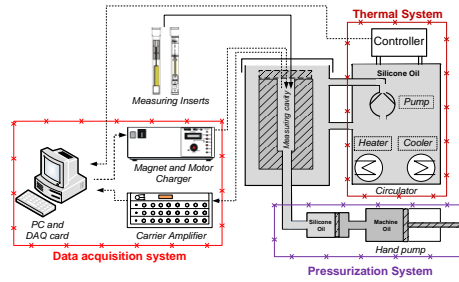


Figure 1: Schematic representation of CMS experimental setup

An example of results, i.e. shear relaxation modulus, $G(t)$ measured on CMS experimental setup are presented on Figure 2. Measurements were conducted on Polyamide 6 (PA6) at different temperature and pressure conditions. From the results it is clearly seen that temperature accelerates shear relaxation process by app. 9 decades in time (10^9 times!), visible through shifting shear relaxation modulus, $G(t)$ in time to the left from room condition (25 °C and 1 bar). Similar but opposite effect has pressure that slows relaxation process and shifts curve to the right for app. 2.5 decades in time from room conditions ($10^{2.5}$ times!).

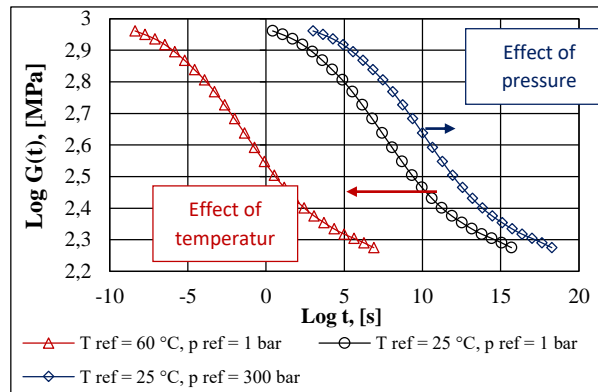


Figure 3: Effect of temperature and pressure on shear relaxation modulus, $G(t)$ for PA6

2.2. Experimental setup for measuring dynamic bulk compliance – DBC experimental setup

Another unique experimental setup for measuring dynamic bulk compliance, also called DBC experimental setup was developed. DBC is specialized to study combined effect of temperature and pressure on volumetric (bulk) properties in frequency domain, i.e. dynamic bulk compliance $B^*(\omega)$ in temperature range from room temperature up to 150 °C and pressures range from atmospheric pressure up to 2000 bar. Experimental setup is schematically shown in Figure 3 [3].

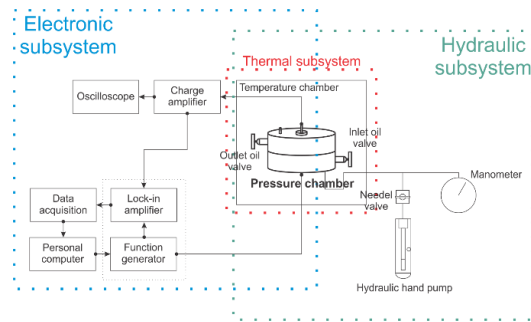


Figure 3: Schematic representation of DBC experimental setup

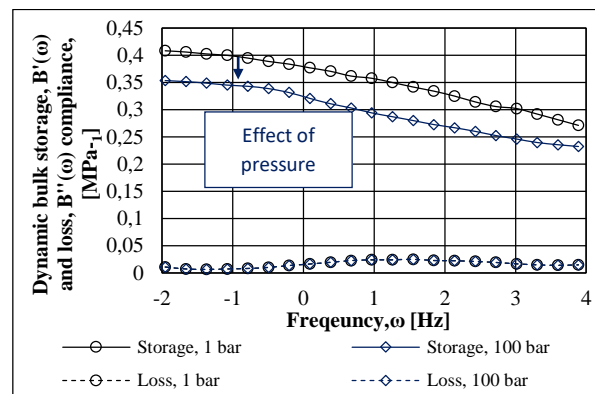


Figure 4: Effect of pressure on master curves obtained through frequency-temperature superposition of dynamic bulk compliance, $B^*(\omega)$ for PVAc [3]

An example of results, i.e. dynamic bulk storage $B'(\omega)$ and loss, $B''(\omega)$ compliance measured on DBC experimental setup are presented in Figure 4. Measurements were conducted on polyvinyl acetate (PVAc) through wide range of temperatures and pressures. Preliminary results on dynamic bulk compliance show that temperature affects bulk properties similarly as in shear, whereas pressure profoundly affects the magnitude of bulk behaviour shifting dynamic bulk storage compliance, $B'(\omega)$ vertically as shown in Figure 4.

3. Linear and non-linear viscoelastic models

Among many models for predicting long term behaviour of polymeric materials, Fillers-Moonan-Tschoegl (FMT) linear model and Knauss-Emri (KE) non-linear model, incorporate combined effect of temperature and pressure on material behaviour [1]. Both models are based on free volume theory and require mechanical properties measured at different temperatures and pressure using the experimental setups described above.

4. Example and Concluding remarks

Each polymer has unique time-dependent mechanical properties and different susceptibility to temperature and pressure effects. Therefore it is of key importance to measure their mechanical properties at different temperature and pressure conditions in order exploit their full potential, and extend their useful range and durability.

As an example of the pressure effect utilisation, we present the new generation vibration isolation based on the patented *Dissipative bulk and granular systems technology* [4]. This technology uses elastomeric materials in granular form to enhance their dynamic properties by exposing them to “self-pressurization”, which shifts material energy absorptions maximum towards lower frequencies, to match the excitation frequency and/or rate of dynamic loading to which a mechanical system is exposed. In the case of TPU materials the stiffness and energy absorption capability of an isolation may be increased between 10 to 100 times.

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CAVITATION DAMAGE IN TIRE RUBBER MATERIALS

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ABSTRACT

The aim of this study was to investigate cavitation in rubber vulcanizates subjected to constrained tensile deformation. In fact, the formation and growth of cavities (= defects, holes, voids) are induced due to multiaxial stress. In practice, such a constrained situation can occur, for instance, by deflecting tire treads. Different experimental techniques were used to study the mechanical properties and cavitation behavior of unfilled and carbon black filled styrene-butadiene-rubber vulcanizates. With the help of in-situ dilatometry, the damaging process in rubber pancake specimens was characterized by a rubber volume increase attributed to cavitation. Additionally, the evolution of cavity populations was investigated by X-ray microtomography. As a result, the effect of microstructural network constraints is comparable to that known from geometrical constraints. Indeed, the higher the degree of constraints is, the higher is the number of microscopic cavities. Although cavitation is often attributed to interfacial failure between rubber matrix and rigid particles, cavitation has also been detected in “carbon black free” and “zinc oxide free” rubber vulcanizates. As a consequence, the cavitation phenomenon is not exclusively triggered by rubber-filler interactions, as widely believed, but also by the polymer properties or the rubber network configurations itself. Moreover, the results suggest that sufficiently well dispersed and distributed reinforcing fillers increase the resistance against cavitation, similar to the effect known from macroscopic fatigue crack propagation tests.

MINERAL FILLERS; TRENDS FOR TIRE AND TECHNICAL RUBBER GOODS

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ABSTRACT

White fillers, mineral fillers and nano fillers have been a part of nearly all technical conferences and meetings in the last years. Particle size and structure, abrasion and surface properties, colour for non black compounds are the main criteria up to now.

WERBA-CHEM defined properties for low cost mineral fillers, suitable for a wide range of rubber applications. The available types of natural fillers were tested and selected. The suitable traditional and new production processes and modifications were defined with the goal to produce fillers with particle size close to 1 μ , non abrasive and agglomerate free. Modern milling and delamellate technology, particle separation and modification for the materials allow also attractive price relation in comparison with carbon black and silica.

Additional properties like plasticity, durability, resistance, gas permeability or CO² parameters like sustainability, transport and packaging play important role. Those parameter and economical evaluation prepare the ground for new applications.

Test results with our close-to-praxis formulations are an optimal starting point for further, customer testing.

Developments in tire applications are possible in compounds for bead area, innerliner and breaker. There are even more applications in technical rubber goods. Especially all kind of insulation compounds for car and cable industry, non black and black compounds for "white goods" and shoe industry, hoses, carpets and flooring with highest technical standard.

Today's technology allows further modifications with processing aids for better dispersion and selected chemical substances for better mechanical properties. Other possibilities are semi and full calcined products. Several products from the product range are tailor made modifications according to customer requirements.

COMBINED SULPHUR – PEROXIDE CURING SYSTEM

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ABSTRACT

Vulcanization with sulphur is the oldest process in cross-linking of unsaturated elastomers. Sulphur curing systems leads to the forming of sulphidic cross-links with various lengths (monosulphidic C-S-C, disulphidic C-S₂-C and polysulphidic C-S_x-C, x = 3-6) between macromolecular chains [1]. Generally, sulphur cured vulcanizates exhibit good elastic and dynamic behavior and possess high values of tensile and tear strength. Sulfur curing of rubber is very complex and intricate process and the mechanism of cross-linking is still not clearly understood.

On the other hand, cross-linking of rubber with peroxides is relatively simple process results in forming carbon-carbon bonds between macromolecular chains which have higher dissociation energy compared to sulphidic cross-links [2]. In order to improve the cross-linking efficiency and properties of peroxide cured elastomers, the multifunctional co-agents are very often used. Co-agents are typically low molecular weight organic molecules with high reactivity towards free radicals. During the curing process, they undergo homopolymerization reaction and easily graft to elastomer chains to form complex cross-linked network [3]. The main advantage of peroxide cured vulcanizates is good heat ageing resistance, good electrical properties and low compression set.

The aim of this work was to investigate the influence of curing system composition on curing process and physical-mechanical properties of prepared rubber blends based on natural rubber NR as well as synthetic acrylonitrile-butadiene rubber NBR. Application of combined sulphur - peroxide curing systems in cross-linking of elastomers leads to the forming not only of sulphidic cross-links between macromolecular chains, while more stable and rigid carbon-carbon bonds. It comes to that all investigated parameters were changed in dependence on the composition of curing system, but on the type of rubber matrix too. While the tensile strength and elongation at break of rubber blends based on NR increased with increasing amount of sulphur in mixed curing systems. In case of vulcanizates based on NBR, the highest values of both characteristics were reached in the case of sample cured with equivalent ratio of sulphur and peroxide. The results revealed that not only the composition of curing system, but also the type of rubber plays an important role of vulcanizates properties.

The influence of the curing system composition on the thermo-oxidative stability of prepared materials was study too. The application of mixed sulphur-peroxide curing systems should combine the pattern of C-C and sulphidic cross-links. The results revealed that physical-

mechanical properties of vulcanizates based on NBR were improved after thermo-oxidative ageing as a consequence of additional cross-linking of the rubber matrix. The improvement of physical-mechanical properties of vulcanizates based on NR after thermo-oxidative ageing was the most pronounced in the case of sample cured only with sulphur system, while almost total degradation of the sample cured only with peroxide system was recorded during the ageing period.

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DEVELOPMENT AND ANALYSIS OF MATERIALS FOR RUBBER SEALS IN AUTOMOTIVE

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ABSTRACT

Rubber compounding allows a very precise tailoring of material properties according to requirements of a specific application. Because of that, rubber products such as seals can be used in very harsh environments where other materials of polymeric nature fail. These seals can survive temperatures as low as -40 °C while under dynamic loading, can be elongated to as much as 1000 % of their original dimension, survive hundreds of hours in aggressive solutions such as brake fluids, diesel or urea or in long-term under temperatures as high as 125 °C without a change in properties. Thus the development of rubber compounds is a very complex science requiring deep knowledge in rubber formulations chemistry and mixing. Testing and understanding the behaviour of these materials at different conditions is then undisputedly part of the development process. The development process and testing of rubber seals based on EPDM is discussed in this paper.

EFFECT OF COUPLING AND DISPERSANT AGENTS ON BEHAVIOR OF SILICA FILLED RUBBER BASED ON NATURAL RUBBER

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ABSTRACT

Present work is focused on investigation of effect of coupling and dispersant agents on dispersibility of polar silica particles in non-polar Natural Rubber (NR). As coupling agent, widely used Bis[3-(triethoxysilyl)propyl] tetrasulfide (TESPT) was added into rubber compounds. For comparison of effect, Polyethylene Glycol (PEG) and Polypropylene Glycol (PPG) as dispersant agents from group of polyethers were used. Mechanical properties influenced by degree of silica filler dispersion were determined for uncured and cured rubber compounds. The Rubber Process Analyzer (RPA) was used to quantifying the changes in microstructure interaction of uncured rubber compounds. Mechanical properties of cured rubber were analyzed using tensile test machine. Finally, level of silica particles dispersion in rubber matrix was observed by a reflected light microscope DisperGRADER. It has been found, that addition of used additives leads to improve of dispersibility of silica particles in rubber matrix associated with improving mechanical properties. Rubber with addition of silane coupling agent exhibited significantly improved mechanical properties due to formation of durable chemical bonds between silica particles and elastomer chains whereas polyglycols creating hydrogen bonds with physical nature exhibited lower level of mechanical properties improvement compare to rubber without any coupling and dispersant additives.

ACKNOWLEDGEMENTS

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INVESTIGATIONS ABOUT THE PLASTICIZER INFLUENCE IN TECHNICAL ELASTOMERS

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ABSTRACT

Plasticizers are used to match the properties of the raw mixture and the vulcanizate. They affect the viscosity and miscibility of the raw mixture and e.g. hardness, deformability, strength and cold flexibility of vulcanizates. Plasticizers are subject to various requirements in terms of volatility and durability. Furthermore, the plasticizer should not affect the cross-linking, in contact with other media it should come to no dissolution and the vulcanizate should not be changed color. To meet these requirements, a careful selection of the plasticizer to be used is appropriate. This takes place according to the required material properties, with regard to the compatibility with the polymer, the application temperature and the costs.

Plasticizers interact with the macromolecules and result in greater mobility of the chain segments. They form homogeneous systems with the rubber until a critical segregation concentration is reached. The segregation limit is system specific and temperature dependent, which, if exceeded, results in the sweating of the plasticizer. For example, during the use of plasticized elastomers, migration of the plasticizer from the bulk may occur due to mechanical stress and / or higher temperatures. This burdens the environment and leads to changes in the properties of the material. In recent years, vegetable oils have become increasingly important in the chemical industry. For example, oils based on soya or sunflower and rape are used.

In the work, the influence of the plasticizer on various elastomers was investigated. By determining the rubber layer L , it could be shown that the rubber-filler-interaction is influenced by the plasticizer. This is also dependent on when the plasticizer is add into the mixing process. In addition to the rubber-filler-interaction, the mechanical and fracture mechanical characteristics of the vulcanizates are also changed.

Aging studies showed that the plasticizer migrated from the vulcanizates, which led, among other things, to a change in the optical properties.

The FTIR method was used to evaluate the migration. For example, it has been demonstrated that the modification of bio-based plasticizers leads to a reduction in migration behavior.

EVALUATION OF PLASTICIZERS IN CURING BLADDER COMPOUND

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ABSTRACT

The main application of curing bladders is curing of the tires within rubber industry. Development of curing bladders has always been complex and incorporates innovation not only in area of bladder contour, surface and wall construction, but also applying the newest knowledge from compound development. Bladders are manufactured using a well proven long-lasting heat conductive butyl rubber compound.

As stated the main compound component is butyl rubber which ensures exclusive properties such as excellent impermeability and good flex properties. The high degree of elasticity of the bladder guarantees trouble-free molding and stripping. The recipe composition is containing several no less important ingredients.

As filler are used different carbon blacks, worth mentioning are so called thermal carbon blacks, which provides higher thermal conductivity with the aim to reduce overall curing time of the tires.

Castor oil play a roll of typical plasticizer and because its tendency to migrate to the bladder surface has as well a role of lubricant, which provides additional slip effect for bladder - tire interaction during molding and demolding of tire vulcanization processes.

Nevertheless from the inside of the bladder castrol oil might be extracted by curing medium during bladder exploitation, which is not in favour for stable properties over life time. This is main motivation for investigating several different plasticizers available on the market.

MOULD SURFACES FOR RUBBER MIXTURES PROCESSING

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ABSTRACT

The paper deals with the study of contamination of mould cavities. The solution to this problem is highly required in practice because especially the processors of rubber mixtures face the contamination of mould cavities very often. [1] The subject of the study were aluminium alloy plates imitating a mould cavity. The rubber composition is based on a combination of NR and SBR rubbers. Experimental plates simulating the mould cavity surface were previously used to mould 150 moulding cycles of tread or sidewall compound. Processing conditions: vulcanization temperature 170°C, time 7 minutes or vulcanization temperature 170°C, time 4.5 minutes. The plates were cleaned after the previous experiment with technical gasoline and acetone only. In the following experiment, the plates were subjected to 200 pressing cycles of the rubber mixture and then their surfaces were examined for the presence of adhering contaminants. A visual comparison of the surface of the mould cavities was performed by optical microscopy before and after the 200 pressing cycles. The images were supplemented with surface roughness measurement before and after 200 pressing cycles. The surface roughness of the plates was measured non-contact to avoid the surface being affected by the measuring tip. The same surface roughness was found for all boards. It was assumed that the surface roughness would decrease due to clogging of the surface texture depressions. [2, 3] The experiment did not prove this assumption, it cannot be unambiguously specified that the surface roughness was reduced or increased. Fourier transform infrared spectroscopy (FTIR) was used to determine the composition of the contaminants, indicating the presence of contaminants from plasticizers. [4] X-ray fluorescence spectrometry (XRF) was used to determine the elemental composition. Sulphur (probably from the vulcanization system - vulcanizing agent) and zinc (ZnO - vulcanization activator) were detected on the surface of the plates. FTIR analysis did not identify these elements by any band of spectra. To complete the experiment, a thermogravimetric analysis (TGA) of the rubber compound used was performed. Its results show that decomposition processes occur during the vulcanization process which releases volatile substances that are likely to adhere to the mould cavity surface.

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FUTURE TRENDS FOR DETERMINATION OF RUBBER BEHAVIOR IN LAB APPLIED FOR TIRES AT SIMULATION OF REAL LOADING CONDITIONS IN SERVICE

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ABSTRACT

The aim of this work was to describe the future trends in experimental laboratory characterization of fracture processes of rubber material used for tire application from the energy point of view, and to contribute to the establishment of efficient, however highly exact complex methodology for fracture investigation. Two very new experimental laboratory equipment and methodological processes have been introduced. These are able to characterize the fracture processes in rubber material over very broad range of tearing energies. It includes the estimation of the minimal tearing energy, necessary for the crack initiation as well as critical tearing energy, defining the total ultimate rupture. Introduction and capability of these methods was demonstrated on experimental investigation and estimation of fracture behaviour of fundamental rubber types, which were NR and BR. The determined data for the experimental analysis of rubber materials, used in this work, exactly corresponds with the previously evaluated data from the work done by Gent et al., which was done still in the 1964. Thus, receiving identical data over the broad range of tearing energy by using the new methods and equipment demonstrates the huge potential and efficiency improvement. The complex investigation, including the equipment, clearly can be labelled as the future trends in experimental laboratory tools, characterizing the fracture behavior of rubber material with respect to real loading conditions, applied on rubber products in the field.

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