CL-12
MICROHARDNESS OF POLYMERS
POLY(BUTYLENE TEREPTHALATE) PBT

DAVID MANAS, MIROSLAV MANAS, MICHAL STANEK, MARTIN OVSÍK, PETR KRATKY, JAKUB JAVORÍK, and MARTIN BEDNARIK

Tomas Bata University in Zlín, Faculty of technology, Department of Production Engineering, nam. T.G. Masaryka 275, 762 72 Zlín, Czech Republic
dmanas@ft.utb.cz

Abstract

Hard surface layers of polymer materials, especially polypropylene, can be formed by chemical or physical process. One of the physical methods modifying the surface layer is radiation cross-linking. Radiation doses used were 0, 30, 45, 60 and 99, 132, 165, 198 kGy for unfilled Poly (butylene terephthalate), PBT with the 5% cross-linking agent (triallyl isocyanurate). Individual radiation doses caused structural and micromechanical changes which have a significant effect on the final properties of the PBT tested. The improvement of micromechanical properties was measured by an instrumented microhardness test.

Introduction

Poly (butylene terephthalate), PBT, is a commercially important engineering polymer with a wide range of applications such as injection molding and extrusion. As a member of the polyester family, it is also often used as the matrix material in glass fiber reinforced composites, having attractive mechanical properties, good moldability and fast crystallization rate. PBT has some processing advantages over its chemical relative, poly (ethylene terephthalate), PET. The melting temperature of PBT is about 230 °C, which is lower than PET, (ca. 270 °C), allowing PBT to be processed at lower temperatures. In addition, PBT has a lower glass transition temperature, a faster crystallization rate.

The irradiation cross-linking of thermoplastic materials via electron beam or cobalt 60 (γ-rays) proceeds is proceeding separately after the processing. The cross-linking level can be adjusted by the irradiation dosage and often by means of a cross-linking booster.

The main difference between β- and γ-rays is in their different abilities of penetrating the irradiated material. γ-rays have a high penetration capacity (Fig. 1). The penetration capacity of electron rays depends on the energy of the accelerated electrons.

The thermoplastics which are used for production of various types of products have very different properties. Standard polymers which are easy obtainable with favourable price conditions belong to the main class. The disadvantage of standard polymers is limited both by mechanical and thermal properties. The group of standard polymers is the most considerable one and its share in the production of all polymers is as high as 90%.

The improvement of micromechanical properties was measured by an instrumented microhardness test.

Experimental part

For this experiment poly (ethylene terephthalate) V-PTS-CREATEC-B3HZC * M800/25 nature; PTS Plastics Technologie Service, Germany (unfilled, PBT+TAIC, MFR – 230 °C/2.16 kg – 7 g/10 min) was used. The material already contained a special cross-linking agent TAIC (triallylisocyanurate) (5 volume %), which should enable subsequent cross-linking by ionizing β-radiation. The prepared specimens were irradiated with doses of 33, 66, 99, 132, 165 and 198 kGy at BGS Beta-Gamma Service GmbH & Co. KG, Germany.

The samples were made using the injection molding technology on the injection molding machine Arburg Allrounder 420C. Processing temperature 240–260 °C, mold temperature 75 °C, injection pressure 80 MPa, injection rate 65 mm s⁻¹.

Instrumented microhardness tests were done using a Micro Combi Tester, CSM Instruments (Switzerland) according to the CSN EN ISO 6507-1. Load and unload speed was 2 N min⁻¹. After a holding time of 90 s at maximum load 1 N the specimens were unloaded. The indentation hardness
HIT was calculated as maximum load to the projected area of the hardness impression according to:

\[ H_{IT} = \frac{F_{\text{max}}}{A_p} \text{ with } \quad h_c = h_{\text{max}} - \frac{F_{\text{max}}}{S} \tag{1} \]

Where \( h_{\text{max}} \) is the indentation depth at \( F_{\text{max}} \), \( h_c \) is contact depth. In this study the Oliver and Pharr method was used calculate the initial stiffness (S), contact depth (\( h_c \)). The specimens were glued on metallic sample holders 1–5.

**Results and discussion**

The values measured during the microhardness test showed that the lowest values of indentation microhardness and microhardness Vickers were found for the irradiated PBT by dose of 33 kGy. On the contrary, the highest values of indentation hardness were obtained for PBT irradiated by a dose of 165 kGy (by 15 % higher in comparison with the irradiated PBT by dose 33 kGy), as can be seen at Fig. 2.

According to the results of measurements of microhardness, it was found that the highest values of indentation modulus of elasticity were achieved at the PBT irradiated with dose of 165 kGy (by 9 % higher than compared with non-irradiated PBT). On the contrary, the lowest values of the indentation modulus of elasticity were found for irradiated PBT by dose 198 kGy as is seen at Fig. 3.

Material deformation in time under constant stress (indentation creep) measured by instrumented test of microhardness showed (Fig. 4) that the highest creep values were measured on non-irradiated PBT (8,2 %), while the lowest creep value was found in PBT irradiated by 198 kGy dose (5,6 %). The creep dropped by 31 % as a result of radiation, which represents a considerable increase of surface layer resistance.

**Conclusion**

Very interesting results were obtained for irradiation modified PBT. When comparing the irradiated and non-irradiated (irradiated by dose 33 kGy) PBT it was apparent that the values of indentation microhardness, Vickers microhardness and the indentation modulus considerably increased, in some cases even by 15 % at the irradiation dose of 165 kGy.

This paper is supported by the internal grant of TBU in Zlin No. IGA/FT/2013/020 funded from the resources of specific university research and by the European Regional Development Fund under the project CEBIA-Tech No. CZ.1.05/2.1.00/03.0089.

**REFERENCES**

A COMPARISON OF MATERIAL PROPERTIES FOR MAGNETORHEOLOGICAL AND CONVENTIONAL ELASTOMERS

JENNIFER MCINTYRE\textsuperscript{a,b} and STEPHEN JERRAMS\textsuperscript{b}

\textsuperscript{a} German Institute for Rubber Technology (DIK), Eupener Stra\ss e 33, D-30519 Hannover, Germany, \textsuperscript{b} Dublin Institute of Technology, Focas Research Institute, Dublin 8, Ireland

Jennifer.mcintyre@dikautschuk.de

Abstract

A magnetorheological elastomer (MRE) compound was tested and compared to a reference material. An anisotropic specimen of the material exhibited an increase in shear storage modulus, $G'$ of approximately 50 \%, which corresponded to 1 MPa for this material. Isotropic specimens of the composite showed a reduced tensile strength and increased elongation at break compared to the reference material. These differences were attributed to variations in the hardness and zero-field modulus of the materials as a result of alterations in their recipes. The MRE material experienced lower values of maximum stress.

Introduction

Anisotropic magnetorheological elastomers (MREs) are adaptive composite materials consisting of chains of magnetic particles in an elastomer matrix. These particles are added to the matrix during the compounding stage prior to curing in the presence of an external magnetic field so that the particles are aligned into chains along the magnetic field lines (see Fig. 1). Components fabricated from these smart materials respond to changes in magnetic flux density. The magnetic interactions between the particles resist external forces applied to the material thereby raising the shear modulus and hence increasing the stiffness of the composite material. These properties can be controlled by increasing the magnetic flux density of an external magnetic field. One potential advantage of such a material is in damping of vibrations in automotive applications where the stiffness and thus the damping ability of the material, could be tuned to suit the vibrations encountered in service.

In order to be suitable for industrial applications, product engineers must be confident that the materials will withstand dynamic stresses and strains that can be anticipated in their normal use.

Materials

The MREs investigated consisted of an optimised recipe containing a 25 \% volume fraction of carbonyl iron particles (CIP), Grade SW-S from BASF. Some plasticizer was added to soften the mixture and a small amount of retardant was added to increase the incubation time of the material's vulcanometer curve, allowing more time for the alignment of the magnetic particles. The reference material contained no magnetic powder but had a much higher quantity (50 phr) of carbon black. No plasticizer was added to this reference material. The MRE material had a hardness of approximately Sh A 40 and the reference material had a hardness of Sh A 65.

Two samples of the same MRE mixture were vulcanised, the first placed in a press that had been heated to 45 °C before the magnetic field was switched on. The press was heated to 160 °C and the mixture vulcanised for a period of 7/2 m.

The second sample was placed in a press at 60 °C and a magnetic field of 600 mT was applied. The material was allowed to heat very slowly for approximately 30 m until the temperature within the mould reached 130 °C. At this point the heating was increased to the maximum set-point and the temperature rose rapidly to 160 °C. This resulted in an extended period where there the magnetic particles were free to move and to align before cross-linking occurred. The material was allowed to vulcanise for 7/2 m.

Results and discussion

Uniaxial Testing

A Zwick 1445 universal test machine was employed to conduct a tensile test on three isotropic specimens (containing homogenously dispersed CIP) to determine the ultimate tensile stress (UTS) and elongation at break of the MRE material. The MRE material was found to have a tensile strength of 9 MPa, approximately a third of that of the reference material (30 MPa). The results of the uniaxial testing are summarised in Table I. At almost 650 \% the elongation at break was higher for the MRE material than that of the reference material (480 \%). This makes sense because the hardness of the MRE material is much lower than that of the reference material.

Many researchers have observed a decrease in tensile strength when magnetic particles are added to a rubber compound\textsuperscript{1,2}. However, it should be noted that the higher quantity of carbon black increased the tensile strength in the reference material and that the addition of softener (plasticizer) increased the elongation at break in the MRE material. As with all filled rubbers, dispersion of filler plays a significant role in the properties of the material. Similar MRE recipes with higher tensile strengths are possible and recipes can be adjusted to suit the required application.\textsuperscript{3}
Quasi-static cyclic testing was conducted on two specimens of the MRE material that were cycled at strains of 30%, 70% and 120% for a total of three cycles at each strain. The same tests were carried out with a reference material: five specimens to determine UTS and three specimens for the quasi-static cycling. The maximum stresses experienced by the MRE material were much lower than those experienced by the reference material. Although testing was not continued until failure, it is reasonable to assume that the MRE material would have a longer lifetime than the reference material.

Preliminary crack growth investigations have shown that the addition of micro-sized magnetic particles is not initially harmful to the rubber mixture. However, oxidation of iron particles may reduce the lifetime of MRE materials.

Rheometric testing

Anisotropic specimens of both materials were sheared in an Anton Paar Physica MCR 501 plate-plate rheometer, which contains a magnetic coil. The magnetic field was switched on and off every 20 s and the magnetic flux density increased in steps of 200 mT up to a maximum value of 1 T. The samples were 2 mm thick. Plots of change in shear modulus as the magnetic flux density was raised are shown in Fig. 2 and 3 and the results are summarised in Table II. The two MRE specimens show clear increases in $G'$ in the presence of a magnetic field while the reference material shows a negligible change.

Conclusions

It has been suggested that in order to obtain MREs with a high magnetorheological effect, the matrix viscosity must be quite low. This could limit MREs to soft materials of low zero-field modulus which would in turn limit the applications of such materials. The research described here suggests that this is not necessarily the case. Although the matrix viscosity does hinder the alignment of magnetic particles, it is possible to produce composites with reasonably high mechanical properties which exhibit a significant MR effect.

As the particle diameters of commercial CIPs tend to be quite small (approximately 2–10 µm), they are unlikely to act as stress raisers and so cause problems in filled materials provided they are well-dispersed. Reductions in material properties are more likely to occur when particles agglomerate or as a result of increased chemical activity which would accelerate ageing.

REFERENCES

3. McIntyre J.: ‘‘Development of magnetorheological elastomers for automotive applications.’’ Tire Technology
CL-14
PRE-TREATMENT OF BEECH WOOD BY RADIO-FREQUENCY DISCHARGE PLASMA

IGOR NOVÁK*, ANTON POPELKA*, IVAN CHODÁK*, JAN SEDLIAČIK*, VLADIMÍR VANKO* and ANGELA KLEINOVÁ*

* Polymer Institute, Slovak Academy of Sciences, Dúbravská cesta 9, 845 41 Bratislava 45, ¹ Faculty of Wood Sciences and Technology, Technical University in Zvolen, Department of Furniture and Wood Products, T. G. Masaryka 24, 960 53 Zvolen, v VIPO, a.s., Partizánske, Slovakia

igor.novak@savba.sk

Abstract

The low-temperature discharge plasma has been used to improve the wetting and adhesion properties of wood. The pre-treatment of wood surface using discharge plasma is attractive for various wood applications mainly because of their lower cost. However, we have identified a significant increase of polar component of wood surface energy after modification by low-temperature plasma. Polar component of surface energy is associated with the presence of acid-base forces (electron donor–acceptor bonds). The treatment of wood exhibited a substantial aging effect, but the modified surface never recovers to its initial hydrophobic state. The enhancement of wood wettability is a necessary condition to promote a better adhesion with a water-based adhesives and coatings, which is currently being studied.

Introduction

The bonding of wood after discharge plasma surface modification is of considerable interest with the respect to construction of the strongest wood adhesive joints (1-6). Great efforts have been made in developing various kinds of furniture using wood or plastics veneers in adhesive joints wood-adhesive-veneer. The radio-frequency (RF) discharge plasma at reduced pressure is currently an efficient method for modification of surface and adhesive properties of wood, and is considered as the ‘green’ ecologically friendly method (4, 7). For a common industrial wood application various woods have to possess a large set of various surface characteristics, including polarity (hydrophobicity or hydrophilicity), dyability, scratch resistance, tailored adhesive properties, antibacterial resistance etc. Nanoscale changes to the surface of wood materials enable the changes in materials surface, while maintaining the desirable bulk material properties.

In experimental work we have been focused to study of surface and adhesive properties of beech (Fagus sylvatica) wood modified by RF discharge plasma.

Experimental

Materials

Beech wood plates with dimensions 50 × 15 × 5 mm (TU in Zvolen, Slovakia), ChS Epoxy 510, Telalit T 410 (Spolchemie, Czech Republic), set of 5 testing liquids (water, ethylene glycol, formamide, diiodmethane, 1-bromo-naphthalene), dichloromethane.

Modification method

In this paper the beech wood surface was pre-treated by RF discharge plasma. There are two reasons why in the case of wood to apply discharge plasma modification. Firstly, discharge plasma in air itself significantly increases hydrophilicity of the wood, because of formation various polar groups (e.g. hydroxyl, carbonyl, carboxyl, etc), and, the wood macromolecules are also cross-links (up to a few microns) what leads to the increase in scratch resistance and to the improvement in barrier properties of the wood material. Second reason for the plasma use is an increase of adhesion in adhesive joint between wood substrate, that is important for industrial applications due to growth of wood wettability.

The modification of wood by the capacitively coupled RF (CCRF) plasma was performed in a laboratory CCRF plasma reactor (Scheme 1) working at reduced pressure 80 Pa consists of two 240-mm brass parallel circular electrodes with symmetrical arrangement, 10 mm thick, between which CCRF plasma was created.

The electrodes of CCRF plasma reactor are placed in a locked-up stainless steel vacuum cylinder. The one is powered and the other one is grounded together with steel cylinder. The voltage of CCRF plasma reactor is 2 kV, frequency 13.56 MHz, current intensity was 0.6 mA, and the

Scheme 1. Scheme of CCRF plasma source
The max. power of the CCRF plasma source is 1200 W. The wood samples were modified by CCRF plasma at the power 300 W. The improvement of hydrophilicity and/or hydrophobicity of the wood, its surface properties, the improvement of strength of adhesive joint of wood/wood composites with epoxy adhesive were studied for the determination of the appropriate structure of the plasma modified wood surfaces.

The surface energy of beech wood was determined using contact angles measurements with selected testing liquids set using SEE (Surface Energy Evaluation) device completed with a web camera (Advex, Czech Republic), and necessary PC software.

The drop of the testing liquid (V = 20 µl) was placed with a micropipette (Biohit, Finland) on the polymer surface, and a contact angle of the testing liquid was measured. The contact angle of testing liquid drop on the wood surface was measured instantly after its placing. The surface energies of wood were evaluated by Owens-Wendt-Rabel-Kaelble (OWRK) equation (7).

**Results and discussion**

The contact angle of water drop on the beech wood surface was measured immediately after drop deposition. The contact angles of water decreased with time of the activation by CCRF plasma. The contact angles of polar testing liquid (water) can be explained by an increase of the hydrophilicity of beech wood surface due to the treatment by CCRF plasma in air. The decrease of the contact angles of polar testing liquid can be explained by an increase of the hydrophilicity of beech wood surface due to the treatment by CCRF plasma in air. The hydrophilicity of the surface depends on the formation of polar oxygenic functional groups on wood surface during the plasma modification in air. After saturation of the polymer surface with polar groups the hydrophilicity was stabilized.

The surface energy and its polar component of beech wood increased with time of plasma activation. The surface energy of beech wood treated 120 s by CCRF plasma in air increased from 66 to 78 mJ/m² (pristine sample) to 27.2 mJ/m² (120 s). If the longer activation time was applied the changes of surface energy and its polar component were very small. This fact relates to saturation of wood surface with oxygen-containing functional groups due to modification by CCRF plasma.

The shear strength of adhesive joint of beech wood modified by CCRF plasma in air—epoxy adhesive increased non-linearly with activation time from 5.2 to 7.8 MPa.

**Conclusions**

The contact angles of water deposited on beech wood surface showed a steep decrease after activation by CCRF plasma in air. The surface energy and its polar component of beech wood increased with time of activation by plasma. The surface energy of beech wood treated by CCRF plasma in air increased from 66 (pristine sample) to 78 mJ/m², and the polar component of the surface energy increased from 17.4 to 27.2 mJ/m². The shear strength of adhesive joint of beech wood using epoxy adhesive increases non-linearly with activation time from 5.2 to 7.8 MPa.

**References**


**CL-15**

**AKROMID LITE AND XTRALITE – THE LIGHTWEIGHT POLYAMID FOR TECHNICAL PARTS**

ONDREJ OSTROLUCKÝ

K. D. Feddersen CEE GmbH Member of the Feddersen Group Fachgasse 35-37, A-1150, Vienna, Austria
Ondrej.Ostrolucky@kdfeddersen.com
EFFECT OF BETA LOW IRRADIATION DOSES ON THE INDENTATION HARDNESS OF GLASS FIBER-FILLED POLYPROPYLENE

MARTIN OVSÍK*, DAVID MANAS, MIROSLAV MANAS, MIČHAL STANEK, MARTIN BEDNARIK, and PETR KRAKTY

Tomas Bata University in Zlin, Faculty of Technology, Department of Production Engineering, TGM 5555, 760 01 Zlin, Czech Republic
Ovsik@ft.utb.cz

Abstract

This study examines micro-mechanical properties of glass fiber-filled polypropylene, that is a modified by low \( \beta \)-radiation doses. The specimens were made using the injection molding technology and subsequently irradiated by \( \beta \)-radiation. Micro-mechanical properties were studied by method DSI – Depth Sensing Indentation on the Micro hardness tester by CSM. This paper examines effect of \( \beta \)-radiation on the micro-mechanical properties (indentation hardness and indentation modulus) of glass fiber-filled PP, which were measured using DSI.

Keywords: polypropylene, glass fiber, microhardness, crosslinking, \( \beta \)-radiation, DSI – Depth Sensing Indentation

1. Introduction

Polypropylene (PP) is a stereospecific polymer prepared by polymerization using an organometallic catalyst system. Commercial PP have up to 95 % iso tactic content, which means that pendant methyl groups are almost all on the same side of the chain. When polypropylene is exposed to ionizing radiation, free radicals are formed and these cause chemical changes. Since PP is highly crystalline, these radicals are relatively immobile, and consequently may not be available for reaction for long periods of time. As with other polyolefins, upon irradiation the free radicals are formed along with evolution of hydrogen gas. If the radical is formed on the pendant methyl, the resulting reaction is cross-linking. However, if the radical is formed in the main chain, the chain end may react with hydrogen, thus causing an irreversible scission. Although the processes of chain scission and cross-linking occur simultaneously, and even though the net effect is crosslinking, the overall effect is the loss of mechanical strength1,2,4.

Crosslinking is a process in which polymer chains are associated through chemical bonds. Crosslinking is carried out by chemical reactions or radiation and in most cases the process is irreversible. Ionizing radiation includes high-energy electrons (electron beam), \( \gamma \)-rays, and \( x \)-rays. These not only are capable of converting monomeric and oligomeric liquids into solids, but also can produce major changes in properties of solid polymers. Also, in comparison to UV and visible radiation, they can penetrate considerably deeper into the material1,2,3.

Electron beams (\( \beta \)-rays) generated by accelerators are monoenergetic and the absorbed dose is greatest just below the surface of the irradiated material and falls rapidly at greater depths in the material (Fig. 1a). The energy range of electron beams used in radiation processing is from 0.15 to 10 MeV. Compared with gamma irradiation, electron accelerators have advantages of higher power and directional beams. The time of irradiation by \( \beta \)-rays is in seconds. The limited penetrating power of electron beams means that they are mainly used for irradiating relatively thin objects like wires and cable insulation1,2,4.

Gamma radiation has a high penetration capability at relatively low dose intensity as shown. The most used source of gamma rays (Fig. 1b) is cobalt-60 (C060). The energy of emitted gamma rays is about 1.3 MeV. Conversely the electron accelerators, source of gamma rays cannot be turned off. Therefore the rays are sheltered, in most cases by water tank. Time of irradiation depends on dose intensity and reaches up to several hours. The gamma radiation is mainly used for radiation sterilization1,2,6.

2. Experimental

For this experiment polypropylene PP PTS -Crealen EP-865HS-M0083 filled by 25% glass fiber, that were supplied by PTS Plastics Technologie Service, Germany was used. The material already contained the special cross-linking agent TAIC – triallyl isocyanurate (5 volume %), which should enable subsequent cross-linking by ionizing \( \beta \)-radiation. The prepared PP specimens were modified by radiation with \( \beta \)-radiation doses of 15 and 30 kGy at BGS Beta-Gamma Service GmbH & Co. KG, Germany.

The samples were made using the injection molding technology on an injection moulding machine Arburg Allrounder 420C. Processing temperature 220 °C, mold temperature 55 °C, injection pressure 90 MPa, injection rate 50 mm s\(^{-1}\).

Instrumented microhardness tests were done using a Micro Combi Tester, CSM Instruments (Switzerland) according to the CSN EN ISO 6507-1. Load and unload speed was 2 N min\(^{-1}\). After a holding time of 90 s at maximum load 1 N the specimens were unloaded. The measurements were provided at room temperature.

The indentation hardness \( H \) was calculated as maximum load to the projected area of the hardness impression according to:
where $h_{\text{max}}$ is the indentation depth at $F_{\text{max}}$, $h_c$ is contact depth. In this study the Oliver and Pharr method was used calculate the initial stiffness ($S$), contact depth ($h_c$). The specimens were glued on metallic sample holders.

The indentation modulus is calculated from the Plane Strain modulus using an estimated sample Poisson’s ratio:

$$E_H = E^* (1 - \nu^2)$$

The reduced modulus and Plane Strain Modulus $E^*$ is calculated from the following equation:

$$E_r = \frac{\sqrt{\pi S}}{2\beta \sqrt{A(h_i)}}$$

and

$$E^* = \frac{1}{E_i} - \frac{1 - \nu_i^2}{E_i}$$

Where $E_i$ is the Elastic modulus of the indenter, $E_r$ is the Reduced modulus of the indentation contact, $\nu_i$ is the Poisson’s ratio of the indenter.

### 3. Results and discussion

Indentation characteristics determined by DSI method are depicted in Fig. 3. They characterize course of loading force in dependence on indentor penetration depth, which gives an idea about course of instantaneous values of observed micro-mechanical properties.

With increased dose of $\beta$-radiation caused improvement value of gel content which is apparent in Fig. 4, 5.

When comparing the irradiated and non-irradiated specimens of PP, it was apparent that the values of indentation hardness (Fig. 4) and indentation modulus (Fig. 5) considerably increased. The values measured during the indentation test showed that the lowest values of indentation hardness (58 MPa) and indentation modulus (1.31 GPa) were found for the non-irradiated PP. On the contrary, the highest values of indentation hardness (82 MPa) and indentation modulus (1.89 GPa) were obtained for PP irradiated by a dose of 15 kGy, as can be seen at Fig. 4, 5. The highest radiation doses are used, micro-mechanical properties decreases which can be caused by radiation induced degradation of the material.

### 4. Conclusion

Irradiation of glass fiber-filled polypropylene with a $\beta$-radiation influences the micro-mechanical properties in the following way:

- Radiation of specimens caused improvement values of indentation hardness and indentation modulus.
- The highest values of indentation hardness and indentation modulus were achieved at the PP irradiated with dose of 15 kGy.
- Higher radiation dose does not influence the indentation hardness and indentation modulus significantly.
Values of indentation hardness and indentation modulus correspond to the gel content.

This paper is supported by the internal grant of TBU in Zlín No. IGA/FT/2013/020 funded from the resources of specific university research and by the European Regional Development Fund under the project CEBIA-Tech No. CZ.1.05/2.1.00/03.0089.

REFERENCES


CL-17
A MATERIAL MODEL FOR POROUS ELASTOMERS WITH STRESS SOFTENING PHENOMENON

RATHAN RAGHUNATH and DANIEL JUHRE*

Deutsches Institut für Kautschuktechnologie e.V. (DIK), 30519-Hannover, Germany
daniel.juhre@DIKautschuk.de

Abstract

This paper presents a new prospect of investigating the mechanical behaviour of cellular rubber using a porous hyperelastic material model within the framework of homogenisation method to consider pore volume fraction. There are number of hyperelastic material models to describe the behaviour of homogeneous elastomer, but very few to characterise the complex properties of cellular rubber. The analysis of dependence of material behaviour on pore density using the new material model is supported with experiments to validate the actual material behaviour. The finite element simulations are then followed by compression load tests to validate the material model.

1. Introduction

A cellular rubber is a mixed cell structured porous elastomer with high flexibility, good resilience, high compressibility and very good damping properties. It is widely used in industrial applications as gaskets or sealings due to its leak proof characteristics ranging from acoustics to chemicals. In the event of the heterogeneous cellular rubber, in contrast to conventional materials, there are no suitable tools for general material characterisation.

The porosity in elastomers may arise during the manufacturing process due to the defects or by deliberately introducing the porosity in order to get a high-density foam. When compared to the homogeneous elastomer, even low levels of porosity alters the mechanical properties of the material such as change in the elasticity modulus, decrease in shear and bulk modulus and the common assumption of incompressibility breaks down. A new material model for porous elastomers by Danielsson et al. decouples the influence of porosity from the mechanical properties of the solid material by introducing volume fraction of the pores as an explicit scalar variable and it can be directly implemented into the finite element software packages where it can be used to simulate arbitrary geometries under various loading conditions.

The scalar value that determines the density of pores in the material is characterised by a method called Micro-CT (Computer Tomography) scanning. In addition to pores, the stress softening phenomenon (Mullin’s effect) has been considered in the model with necessary inputs from multi-hysteresis quasi static experiments. These experiments are then accompanied by finite element simulations of a car door sealing and is validated against the test results of compression-load tests to check the accuracy of the new material model.

2. A model for cellular rubber at finite deformations

The material with non-uniformly distributed pores is first homogenised into a pore-containing matrix with a thick walled sphere considered to represent the undeformed porous material subjected to external loading (Fig. 1a). The initial material, whose matrix is taken to be pointwise incompressible, is characterised by an initial void volume fraction \( f_0 = (A/B)^3 \), where A and B are the inner and outer radii of the sphere, respectively. When subjected to the principal macroscopic stretch state \((\lambda_x, \lambda_y, \lambda_z)\), the outer surface of the sphere transforms into an ellipsoid (Fig. 1b,c).

The kinematic relationship of the deformation field subjected to macroscopic stretches together with the boundary conditions is given by

\[
\chi_i = \frac{\chi_i}{F} \left( 1 + B (J - 1) \frac{R^3}{R^3} \right)^{1/3}, \quad J = \det F = \lambda_x \lambda_y \lambda_z = 1
\] (1)

The components of the microscopic/pointwise deformation gradient, \( F \), at every point in the matrix is

Fig. 1. The spherical volume element: (a) undeformed configuration, (b and c) deformed configuration
The pointwise strain energy density function for an incompressible isotropic hyperelastic material determined at every point of sphere can be expressed in terms of the stretch invariants $I_1$ and $I_2$ as

$$W = \frac{1}{2} \left( 1 - \frac{1}{2} \right) B^2 F_{ij} \delta_{ij} + \phi \delta_{ij}$$

$$\phi = (1 + \sqrt{3} B / R) J^{-3}$$

The homogenised strain energy density of the sphere, $\bar{W}$ is

$$\bar{W} = \frac{1}{V_0} \int_{\vartheta = 0}^{\vartheta = \pi} \int_{\phi = 0}^{\phi = \pi} \left( \int_{\rho = 0}^{\rho = \rho_B} \bar{W}(I_1, I_2, f_0) R^2 \sin \vartheta d\rho d\vartheta d\phi \right)$$

where, $B$ is the outer radius of the sphere (Fig.1), the reference volume is given by $V_0 = 4\pi B^3 / 3$. $0 \leq \vartheta \leq \pi$ and $0 \leq \phi \leq 2\pi$ are standard spherical angles, measured in the reference configuration used to define the components of position vector $X$ in spherical coordinate-system.

The strain energy density function of the Yeoh model for solid materials is defined by

$$W = C_{\phi}(I_1^{\phi} - 3) + C_{20}(I_1^{\phi} - 3)^2 + C_{30}(I_1^{\phi} - 3)^3 + W^{\text{iso}}$$

yields the resulting energy function for the porous material

$$\bar{W} = \sum_{i=1}^{3} \left( C_{\phi} \left( 2 - \frac{1}{J} \frac{f_0}{J} + \frac{2(J - 1)}{J^2 \eta^2} \right) - 3(1 - f_0^\prime) \right)$$

$$\eta = \left( 1 + \frac{J - 1}{f_0} \right)$$

Considering the stress softening phenomenon in material parameters, $C_\phi$ can be rewritten as

$$C_{\phi} = C_{\phi0} + b \ e^{J(I_{\text{max}} - 3)}$$

where $C_{\phi0}$ is the parameter for virgin loading and $b$, $c$, are for stress softened loading. The second Piola-Kirchhoff stress then can be derived using the general relation

$$S = 2 \frac{\partial W}{\partial I_1} + J \frac{\partial W}{\partial J} C^{-1}$$

3. Experiments and model fitting

For hyperelastic material characterisation and to determine stress softening phenomenon, simple deformation tests (uniaxial tension, equibiaxial tension and pure shear) are conducted. The experiments are not directly conducted on cellular rubber, due to the presence of pores in the material which lead to an inelastic and inhomogeneous deformation. The Fig. 2 shows multi-cyclic experiments conducted on solid filled elastomer (EPDM-50Shr) while the pores are explicitly introduced into the mathematical model as the scalar value $f_0$.

The moderate strain amplitude curves from each of the experiments are prepared for Yeoh hyperelastic material model fitting considering stress softened phenomenon is shown in Fig. 3a, b. The scalar value $f_0$ determines the density of pore in the material and is measured by a method called Micro-CT scanning (a non-destructive method to reproduce three-dimensional images of components including internal inhomogeneities). Here, the Micro-CT Nanotom (GE Phoenix/ X-Ray) has been used to scan the car door sealing (made up of solid elastomer as a base for supporting cellular rubber over it). The material density in the scanned 3D image of car door sealing is then calibrated to determine the range of void volume fraction $f_0$. The range of $f_0$ was between 0.55 to 0.6 and for further simulations $f_0 = 0.6$ has been considered.

4. Validation

To validate the material model, the deformation behaviour of car door sealing has been compared with the results of compression load test. During the experiment, the load is applied on sealing using a metal cell placed over it, which simulates a displacement driven closure process. The tilting base-metal on which the sealing was glued has given an opportunity to investigate the change in deformation behaviour at different angles.

The actual deformed shape during compression load test has been compared with the simulated shape in Fig. 4a. The force-displacement curves in Fig. 4b show a good correlation between the experimental and simulated results. The growth of simulated curve is smoother compared to the test curve,
5. Summary

The results have shown the capability of the new model to predict the deformation behaviour of cellular rubber in a wide deformation range. At present, the energy functions of new model depends on \( I_1 \) and can be extended easily to an additional \( I_2 \) dependence. The advantage of the model's structure is the sufficient use of standard homogeneous tests on the solid rubber for fitting the material parameters. The material parameter \( f_0 \) is physically motivated and is effective enough to describe the influence of the pore volume on the material behaviour. The validation of the cellular rubber model using FE simulations show a good accordance with the experiments thereby predicting the reasonable behaviour of actual material.

The authors would like to acknowledge the financial support by METZELER APS GmbH.

REFERENCES


Fig. 1. SEM micrographs of a) NiZn and b) MnZn ferrite fillers

CL-18

THE INFLUENCE OF SOFT MAGNETIC FILLERS ON THE PROPERTIES OF THE MAGNETOPOLYMER COMPOSITE

JANA REKOŠOVÁ*, MARIANA UŠÁKOVÁ*, ELEMÍR UŠÁK*, RASTISLAV DOSOUDIL*, and IVAN HUDEC*

* Slovak University of Technology in Bratislava, Faculty of Chemical and Food Technology, Department of Plastic and Rubber, Radlinského 9, 812 37, Bratislava.
* Slovak University of Technology in Bratislava, Faculty of Electrical Engineering and Information Technology, Institute of Electrical Engineering, Ilkovičova 3, 812 19 Bratislava, Slovak Republic

In recent years, the area of application of radio-absorbing materials (RAMs) has been substantially extended. In addition to traditional directions (radio masking, electromagnetic compatibility, etc.), the direction related to environmental safety and protection of people from the action of electromagnetic fields has arisen. Polymers integrated into composites with soft magnetic materials are recently attracting the interest in industrial and scientific branch for their beneficial properties. In this paper, the influence of soft magnetic ferrites on physical-mechanical, rheological and magnetic properties of magneto-polymer composites based on natural rubber SMR 20 are studied. The laboratory prepared NiZn and commercially available MnZn ferrites were used as magnetic filler with concentrations 100, 200, 400 and 600 phr. The preparation of ferrite fillers as well as composite samples was described in paper.

The structural properties of the soft magnetic fillers were investigated by scanning electron microscope. The size and shape of NiZn and MnZn ferrite particles are shown in Fig. 1. The SEM micrographs of both ferrite samples illustrate to present of heterogeneous particles of polyhedral shape aggregated to clusters.

The physical-mechanical properties of prepared composites are plotted in Fig. 2. The tensile strength at break rapidly decreased in dependence of filler content, for the NiZn ferrite filled composites from 14.20 to 3.30 MPa (79 %) and
for the MnZn composites it firstly rises up slightly from 15.60 to 16.50 and then decreased to 3.40 MPa (78 %). The hardness of composites for both types of ferrite fillers increased approximately by 103 % for NiZn and 79 % for MnZn ferrite filled samples. The influence of ferrite fillers in the natural rubber matrix expressed by means of Payne effect suggests higher affinity of NiZn ferrite filler to rubber than in the case of MnZn ferrite filler (Fig. 3). The slope of the lines expressing the activity coefficient of both filler materials is given in this figure (see the legend) as well.

The measured frequency dependencies of the complex permeability \( \mu = \mu' - j \mu'' \) for synthesised composite materials was measured in the frequency range from 1 to 3000 MHz by means of two vector network analyzers and a coaxial holder can be found in Figs. 4 and 5. In these figures, the parameter of curves is the ferrite particle concentration in phr. It can be seen that the values of \( \mu' \) and the peak values of \( \mu''(f) \) dependencies (at the resonant frequency \( f_r \)) get higher with increasing ferrite particle concentration. On the other hand, the resonant frequency shifts towards lower values (see also Fig. 6). One can state that the complex permeability of composite materials is strongly dependent on ferrite particle concentration and frequency. As can be seen from the presented figures, also the choice of ferrite filler material significantly affects resulting dynamic magnetic properties represented by the complex permeability (see the differences between Figs. 4 and 5 – both vertical scales are kept the same). Since pure MnZn ferrite material exhibits in general larger value of initial permeability than NiZn one, the composites with MnZn ferrite filler in general reach higher values of the complex permeability components for the same filler concentration than those with NiZn filler. Note that the differences in quasi-static initial permeability (measured at \( f = 50 \text{ Hz} \)) of both materials are relatively small (Fig. 6). As the ferrite concentration grows up, the influence of ferrite filler becomes more important and the differences boost as well. Observed facts are in a good agreement with our previous investigations. An important role in \( \mu(f) \) dependencies plays the demagnetising field \( H_d \) which is created in filler particles embedded in the non-magnetic polymer matrix due to the presence of magnetic poles on the opposite sides of particles. \( H_d \) is antiparallel to applied high-frequency magnetic field and increases with the decrease of induced magnetic moment in particles. Therefore, \( \mu' \) at low frequencies apparently decreases and \( f_r \) increase with the drop of filler concentration.

The results achieved in this study confirmed considerable influence of the soft magnetic filler on the physical-mechanical, rheological and magnetic properties of magneto-polymer composites with matrix based on natural rubber. The use of various magnetic filler materials with various concentrations enables to prepare magneto-polymer composite materials of required properties for a wide range of applications, such as EM-wave absorbers, wireless radio communication systems, EMF shielding, sensors, etc.

Fig. 2. Influence of NiZn and MnZn ferrite contents on tensile strength at break and hardness of composite samples

Fig. 3. Influence of NiZn and MnZn ferrite fillers on the normalised parameter \( P_{norm} \) of natural rubber composites

Fig. 4. Frequency dependencies of the real (solid symbols) and imaginary (open symbols) component of complex permeability for NiZn-NR composites with various filler contents
This work was supported by the Slovak Research and Development Agency under the contract No. APVV-0062-11 and by the Scientific Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic and the Slovak Academy of Sciences (VEGA), projects No. VG-1/1163/12 and VG-1/1325/12.

REFERENCES


CL-19
INFLUENCE OF CARBON BLACK TYPE ON ELECTRICAL CONDUCTIVITY OF FRC IN AUTOMOTIVE

LADISLAV FOJTLa, SOŇA RUSNÁKOVÁa,
VLADIMÍR LABAŠb, ONDREJ BOŠÁKb, EMIL SELIGAb, MARIAN KUBLIHAb, and MILAN ŽALUDEKa

a Department of Production Engineering, Faculty of Technology, Tomas Bata University in Zlín, Nad Stráňemi 4511, 760 05 Zlín, Czech Republic, b Department of Physics, Institute of Materials Science, Faculty of Material Science and Technology, Slovak University of Technology, Paulinska 16, 917 24 Trnava, Slovak Republic
fojtl@ft.utb.cz

1. Abstract

Polymer composites allow creation of specific materials depending on trends in research and development due to large-scale combinations of individual components. Polymeric materials in consideration of their electrical conductivity belong to the insulators and their specific electrical conductivity is less than $10^{-12}$ S cm$^{-1}$.

The conductivity of those materials can be increased by the addition of the electro-conductive fillers into the polymer matrix. This research paper presents some results about electrical conductivity of polyester composites with carbon reinforcement consisting carbon black fillers.

2. Introduction

The electrical conductivity belongs to the physical quantities describing the conduction of the electric charge in specific material. Polymer materials are normally electrically non-conductive. To transform those materials to electrically conductive, the electrical conductive substances have to be added.

The electrical conductivity of dielectrics is divided by electric charge carriers to ionic or electron conductivity. The ionic conductivity ensures the transfer of electric charge through the free electrons. Most of dielectrics have an absence of such electrons, therefore the conductivity manifests only at high intensities of electric field.

The electrical conductivity is also divided by the movement of charges on the inner and surface electrical conductivity. The inner electrical conductivity depends on the composition of the substances and on the intensity of the acting electric field. Furthermore, conductivity can be divided into ion and electron according to the character of the charge.
carriers. The ion conductivity occurs at the insulator in the solid phase at low or medium intensities of the electric field. On the other hand, the inner conductivity of insulators depends on the temperature, due to the temperature changes of the concentration of charge carriers. The electrical conductivity of insulators and semiconductors grows with increasing temperature. The surface conductivity depends on the moisture content of the material and their ability to repel water.

Possible charge carriers in polymers are ion, electrons and holes. The electrical conductivity in polymers has hopping character and the move of electrons is driven by tunneling mechanism.

3. Materials and methods

The samples were prepared from carbon fabric reinforcement (bidirectional 420 g m\(^{-2}\), 45/45\(^{\circ}\), 5 plies) impregnated by polyester resin with two types of electroconductive carbon black with mass percentage 0 %, 2,5 % and 5 % (Tab. I).

Electric and dielectric parameters were measured on LCR meter HIOKI 3522-50. Totally, eighty frequencies in the range from 0,1 Hz to 100 kHz were used for measuring in alternating electric field (AC). The measuring apparatus for experiments was adapted for monitoring of the changes in the polymer systems, possibly to other materials with low electrical conductivity.

The measurements on the samples (Tab. I) were conducted in a single measuring cycle sequentially at temperatures from 30 °C to 150 °C gradually by 10 °C. The samples were conditioned for measuring temperature of 1200 s before each measurement. The measurements were carried out in a continuous cycle without interruption (removing from the electrode system). Samples were prepared in the quadrat shape with the flat base of 15 × 15 mm without application of the graphite layer.

4. Results

The frequency dependence of the AC conductivity and loss factor of prepared composite samples measured at 100 °C are shown in Fig. 1 and Fig. 2.

![Fig. 1. The frequency dependence of the AC conductivity (samples Tab. I) with various concentration and kind of carbon blacks measured at 100 °C](image1)

![Fig. 2. The frequency dependence of the loss factor (samples Tab. I) with various concentration and kind of carbon blacks measured at 100 °C](image2)

The values of AC conductivity are relatively high. Those high values of conductivity are apparently influenced by the dominant carbon fabric at the sample. The effect of carbon black on the loss factor is shown in Fig. 2, where 2,5% mass percent of carbon black represents a full saturation of the material.

The courses of the temperature-dependent electrical conductivity for individual samples are shown in Fig. 3. The different course of conductivity in Polyester 5 can be described by two possible changes in the material. Firstly, the release of the conductive filler probably water. This effect, however, should occur at least in part on other samples. Second possible change could be the release of the gas in volume of the sample.

5. Conclusion

The presented results show that contrary to the expectation, the addition of carbon black increased the electrical conductivity only above the temperature of 100 °C. We suggest that this result is caused by the inappropriate
dispersion of carbon black into the matrix of the composite, which was suitable and verified for dispersion of other electro conductive fillers. Carbon black VXC 605 XC did not increase the conductivity as much as VXC 605. Furthermore, this type of carbon black showed a decreased of conductivity with higher mass percentage.

This work was supported by the Ministry of Education and Youth of the Czech Republic under project APVV CZ–0168-11,7AMB12SK109 and by Slovak National Science Foundation under grant VEGA No. 1/0356/13 and by internal grant of TBU in Zlín No.IGA/FT/2013/022 funded from the resources of specific university research.

REFERENCES


**CL-20**

**ARTIFICIAL NEURAL NETWORKS PREDICTION OF CHOSEN RUBBER BLENDS PHYSICAL AND MECHANICAL PROPERTIES**

**IVAN RUŽIÁK**, **JANKA JURČIOVÁ**, **MILADA GAJTANSKÁ**, **IÚBOŠ KRIŠTÁK**, **PAVEL HUSA**, **PAVEL KOŠTIAL**, **ZORA JANČIKOVÁ**, **and VLADIMÍR RUSNÁK**

"Technical University of Zvolen, Faculty of Wood Sciences and Technology, T. G. Masaryka 24, 960 53 Zvolen, Slovak Republic, b VŠB – Technical university of Ostrava, Faculty of metallurgy and material engineering, 17. listopadu 15/2172, 708 33 Ostrava, c Saar Gummi Slovakia s.r.o, Gumárenská 397/21, 97223 Dolné Vestenice, Slovak Republic

**Abstract**

In our work we have studied influence of different types of plasticizers on mechanical properties of rubber blend mixtures. We have used Oleic acid as a plasticizer. Reference sample was mixed with plasticizers containing 1 DSK and 3 DSK of ETOXON 2, 4, 6, 8, 10, 20, 30 wt.%. Mechanical properties such as tensile strength, ductility, modulus modules M100, M200, M300 have been measured. All measured mechanical properties change with chemical composition.

Artificial neural networks (ANN) have been used for prediction of mechanical properties with respect to rheological properties and chemical composition. ANN application was controlled by statistical functions RMSE_rel and R² which value for all predicted values was higher than 0.93.

Key Words: rubber blend, artificial neural networks, physical – mechanical properties, prediction

**1. Introduction**

Rubber blends are mixtures of rubber with fillers such as carbon black, activators, plasticizers, accelerators. To their unique properties belong super elasticity, small thermal conductivity, relatively low thermal expansion coefficient, low electrical conductivity and relatively low density. Due to their properties variation with composition they are frequently used in various applications.

Carbon black is active filler which raise electrical and thermal properties because of his strong interactions with natural rubber. This interaction causes Payne’s effect responsible for the elastic modulus decrease under static and dynamic load of rubber blends.

This article deals with the usage of artificial neural networks (ANN) for characterization of rubber blend mixtures with different plasticizers.

Rubber blends belong to elastomers which have high ductility \(\varepsilon\) between 100% and 1000%. Basic properties of rubber mixtures – Young’s modulus of elasticity \(E\), tensile strength \(R_m\) and density \(\rho\) are shown in Table I.
Essential ingredient for the manufacture of rubber compounds is plasticizer, which affects the workability of mixtures (mixing, rolling, extrusion ...), reduction of stiffness as well as the final properties of vulcanizates. The positive effect of plasticizer is in a favourable influence of processing characteristics, physical and mechanical properties as well as dynamic – mechanical properties of mixtures.

Artificial neural networks (ANN) are used for materials properties prediction when analytical physical approximation cannot be found. From this very robust mathematical tool, material properties can be predicted. Some of the examples are:

- mechanical properties of PS rubber blends composites
- mechanical properties of LLDPE rubber blends composites prediction
- location of acoustic emission source prediction in CFR plastic composites

ANN usage is tested by statistical parameters REL_RMSE and $R^2$ which are defined by following equations

$$REL_{RMSE} = \sqrt{\frac{1}{n(n-1)} \sum_{i=1}^{n} (y_i - o_i)^2}$$

$$R^2 = \left(\frac{E(y)e(o)}{E(y,o)}\right)^2$$

Where:
- $y_i$ – measured values
- $o_i$ – predicted values
- $n$ – number of input values
- $E(y)$ – statistical mean value of inputs
- $E(o)$ – statistical mean value of predicted values
- $E(y,o)$ – mean value of multiplication of predicted and input values

From publications is good to see, that ANN prediction is very useful tool for technology especially in cases of preparation of large amount of samples.

### 2. Experimental procedures

#### 2.1. Samples

In our experiments we used as reference rubber mixture of composition in DSK shown in Table II.

#### 2.2. Experimental procedure

We have measured physical and mechanical properties of oleic acid mixtures with oleic acid content 1 DSK and 3 DSK. Measured values of tensile strength $R_m$, ductility $A$, moduluses $M_{100}$, $M_{200}$, $M_{300}$ of samples with 1 DSK oleic acid are shown in Table III. Measured values of tensile strength $R_m$, ductility $A$, moduluses $M_{100}$, $M_{200}$, $M_{300}$ of samples with 3 DSK oleic acid are shown in Tab. IV.

### 3. ANN prediction

In Fig. 1 is shown function of predicted tensile strength $R_m$ versus measured tensile strength $R_m$. In Fig. 2 is shown

### Tables

#### Table I
Basic properties of chosen polymeric materials

<table>
<thead>
<tr>
<th>Material</th>
<th>$E$ [MPa]</th>
<th>$R_m$ [MPa]</th>
<th>$\rho$ [MPa]</th>
</tr>
</thead>
</table>

#### Table II
Chemical composition of reference rubber mixture

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>DSK content</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMR</td>
<td>100</td>
</tr>
<tr>
<td>Sulphur</td>
<td>2</td>
</tr>
<tr>
<td>ZnO</td>
<td>5</td>
</tr>
<tr>
<td>Stearine</td>
<td>2</td>
</tr>
<tr>
<td>Sulfenax</td>
<td>2</td>
</tr>
<tr>
<td>N339 CB</td>
<td>50</td>
</tr>
<tr>
<td>Gumodex</td>
<td>10</td>
</tr>
</tbody>
</table>

#### Table III
Measured values of 1 DSK oleic acid mixtures

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>17</td>
<td>474</td>
<td>3,60</td>
<td>7,20</td>
<td>10,80</td>
</tr>
<tr>
<td>2%</td>
<td>18,01</td>
<td>473</td>
<td>3,80</td>
<td>7,60</td>
<td>11,40</td>
</tr>
<tr>
<td>4%</td>
<td>18,71</td>
<td>458</td>
<td>4,10</td>
<td>8,20</td>
<td>12,30</td>
</tr>
<tr>
<td>6%</td>
<td>19</td>
<td>455</td>
<td>4,20</td>
<td>8,40</td>
<td>12,50</td>
</tr>
<tr>
<td>8%</td>
<td>19,28</td>
<td>447</td>
<td>4,30</td>
<td>8,60</td>
<td>12,90</td>
</tr>
<tr>
<td>10%</td>
<td>19,83</td>
<td>428</td>
<td>4,60</td>
<td>9,30</td>
<td>13,90</td>
</tr>
<tr>
<td>20%</td>
<td>20,50</td>
<td>404</td>
<td>5,10</td>
<td>10,10</td>
<td>15,20</td>
</tr>
<tr>
<td>30%</td>
<td>21,90</td>
<td>399</td>
<td>5,50</td>
<td>10,90</td>
<td>16,50</td>
</tr>
</tbody>
</table>

#### Table IV
Measured values of 3 DSK oleic acid mixtures

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>18,50</td>
<td>465</td>
<td>3,90</td>
<td>7,90</td>
<td>11,90</td>
</tr>
<tr>
<td>2%</td>
<td>18,72</td>
<td>456</td>
<td>4,10</td>
<td>8,20</td>
<td>12,30</td>
</tr>
<tr>
<td>4%</td>
<td>18,90</td>
<td>455</td>
<td>4,20</td>
<td>8,30</td>
<td>12,50</td>
</tr>
<tr>
<td>6%</td>
<td>19,16</td>
<td>450</td>
<td>4,30</td>
<td>8,50</td>
<td>12,80</td>
</tr>
<tr>
<td>8%</td>
<td>19,85</td>
<td>423</td>
<td>4,70</td>
<td>9,40</td>
<td>14,10</td>
</tr>
<tr>
<td>10%</td>
<td>20,50</td>
<td>403</td>
<td>5,10</td>
<td>10,20</td>
<td>15,30</td>
</tr>
<tr>
<td>20%</td>
<td>21,42</td>
<td>399</td>
<td>5,40</td>
<td>10,70</td>
<td>16,10</td>
</tr>
<tr>
<td>30%</td>
<td>22,49</td>
<td>393</td>
<td>5,70</td>
<td>11,40</td>
<td>17,20</td>
</tr>
</tbody>
</table>
3. Conclusions

From presented results of ANN prediction is good to see, that ANN can predict mechanical properties of rubber mixtures as a function of different plasticizers and ETOXON tenside.

REFERENCES


CL-21
A UNIQUE APPROACH TO MODELLING OF COMPOSITE MATERIALS AND STRUCTURES

ZDENĚK RŮŽIČKA*, JAN SEYFARTH*, and ROGER ASSAKER*

*SimulPlast s.r.o. (CZ), Bratrská 1114, 751 31 Lipník nad Bečvou, Česká republika, e-Xstream engineering (L), Z. I. Bommelscheuer, L-4940 Bascharage, Luxembourg
simulplast@simulplast.cz

Abstract

Even though a notable rise in the practical use of composite materials can be observed in the Automotive industry, the simulation of composite structures still poses a challenge. The combination of the softwares Moldex3D and DIGIMAT offers a unique approach to stand up to this challenge.

Composites in Automotive Industry

The complexity of structural design in the automotive industry rises. One of the challenges is the need to develop greener vehicles with a minimal CO₂ footprint while improving safety, quality and performance. The reduction in CO₂ emissions can be achieved via different technologies, but all combined with light-weight structures. For the design of light-weight vehicles, the optimal use of high performance composite materials is the key enabler.

Composites cover a large variety of materials where a matrix phase is reinforced by one or more inclusion phases like fibers and/or other fillers. Most commonly composites based on thermoplastic or epoxy matrices are combined with short, long or continuous fibers. Depending on the type of material automotive parts are processed via injection molding, draping or other technologies.

From this situation real complexity arises. The material microstructure is directly influenced by the processing conditions. This change in microstructure influences the physical properties of the material and thus the final performance of the composite part.

The Simulation Challenge

For the simulation engineer this complexity poses a special challenge. Only a good solution to the question of how to best describe these parts in computations the assured design of automotive parts can be guaranteed. Computer Aided Engineering (CAE) has been used for many years to reduce the time and cost of vehicle design and manufacturing. However, the majority of the CAE processes, tools and even
engineering mindset have been optimized and mainly targeted toward anisotropic materials which are homogeneous like steel.

There is a lack of sufficient description of heterogeneous composite materials on the simulation market. For them, all different kinds of performance must be tackled, among them mechanical stiffness, thermal as well as strain rate dependency and failure.

**The Composite Solution**

A predictive solution that shows excellent correlation with the experimental response is based on multi-scale modeling techniques taking into account the influence of material microstructure on the material and part response. This technology has been implemented in the DIGIMAT software. On the base of micromechanics a new type of material model is offered to the simulation engineers. These models are able to read in the local microstructure information coming from some processing simulation – for instance simulation software for injection molding Moldex3D. This software is unique among the available softwares for injection molding by its precision of the 3D fiber orientation changing through the wall thickness thanks to hybrid 3D volumetric mesh composed from different types of volumetric elements with more detailed description in the surface layers (see Fig. 1).

After reading the information about the material structure DIGIMAT calculates the correct nonlinear, strain rate and/or temperature dependent material response and communicate this to the FEA solver (see Fig. 2).

Today, these models cover all of the above described material performances and are used to describe static as well dynamic testing of automotive parts.

**Applications**

The usage of micromechanical material models proves highly advantageous. Compared to the classical isotropic approach vast differences can be seen in the prediction of the performance of the part.

A typical injection molded plastic part like an air intake manifold must be able to withstand a high pressure peak resulting from piston backfire (see Fig. 3). In simulation two different time points during this dynamic event have been investigated (see Fig. 4). A complete and qualitative difference in the performance could be observed with the classical isotropic and the state-of-the-art micromechanical approach. The classical method fails completely to predict correctly the stress distribution not only over the part, but also

---

Fig. 1. Layers with different orientation of short fibers in injection molded parts (cut through the wall thickness) vs. unique hybrid 3D volumetric mesh in the simulation software for injection molding Moldex3D, which is enabling precise fiber orientation.

Fig. 2. Micromechanical based material models are sensitive to the fiber orientation. On the foundation of injection molding simulation this can be used to predict the local stiffness on the part. (Courtesy of Renault)

Fig. 3. An intake manifold is prone to dynamic pressure loads caused by engine piston backfire. (Courtesy of JSOL Corporation)

Fig. 4. Two different time points in this dynamic event have been investigated for isotropic homogeneous and anisotropic inhomogeneous material. (Courtesy of JSOL Corporation)
in time domain whereas the DIGIMAT modeling gives a realistic picture.

To improve on this well-known limitation of isotropic models usually some scaling factor is introduced to describe an average of the material properties as varying over the part. However, this procedure is not predictive as no unique scaling factor can be derived from any rule of thumb. As a consequence, even though experimental curves might be fitted via this approach, the simulation itself is not predictive.

This fact shows well on a plastic part in a sunroof system where two cases, one global and one local loading, have been investigated (see Fig. 5). To fit the experimental curves for both cases vastly different scaling factors had to be used (see Fig. 6). In contrary, re-running the analyses with one unique DIGIMAT material directly gave excellent correlation with the experiment.

**Future in material modelling**

In a similar manner all kind of performances of composites have been investigated so far using the DIGIMAT approach. For precise description of the material microstructure (especially the change of the fiber orientation) for injection molded parts Moldex3D simulation software with unique 3D hybrid volumetric mesh can be used.

Thanks to the excellent correlation of the simulation with reality this technique today is developing a new standard on the market. Engineers correctly predict static or dynamic scenarios including the failure behavior of the part. Most recently temperature dependency has been included to be able to tackle all kind of under-the-hood applications. The same as today with injection molding and short fibers, draping as a processing technology for continuous fiber composites will enter the stage soon. But also new material models are in preparation which will be able to describe the dependency of local fatigue properties on the material microstructure. The final goal is to go for a full life time prediction of composite parts.


---

**Fig. 5.** Injection molded part with varying fiber orientation in a sunroof system. (Courtesy of Ticona and ArvinMeritor)

**Fig. 6.** Comparison between isotropic and anisotropic approach for two load cases. The DIGIMAT approach correlates excellently with the experiment. (Courtesy of Ticona and ArvinMeritor)