Devulcanization of Whole Passenger Car Tire Material

Devulcanization · whole tire recycling · SBR · Horickx plot · crosslink density

Ground tire rubber (GTR) from whole passenger car tires is devulcanized using the conditions which are the best compromise for tire rubbers. The elaboration of the optimal devulcanization process for GTR is performed in order to obtain a homogeneous and high degree of devulcanization. Swelling of GTR in a mixture of an oil and a devulcanization aid before devulcanization was studied as a way to achieve a more homogenous breakdown of the crosslink network. Further optimization of the parameters in terms of devulcanization oil loading and process time were elaborated. By using these optimized devulcanization conditions, a significant reduction of crosslink density can be achieved for GTR.

Devulkanisation von PKW-Gesamtreifenmaterial

Devulkanisation · Ganzreifen-Recycling · SBR · Horickx Plot · Vernetzungsdichte

Zerkleinertes Reifengummi aus ganzen Pkw-Reifen wird unter Bedingungen, welche einen Kompromiss für alle im Reifen vorkommenden Elastomere darstellen, devulkanisiert. Die Devulkanisationsparameter sind hinsichtlich einer homogenen Devulkanisation und weitreichenden Reduzierung der Netzwerkdichte optimiert. Untersucht wurde, ob Vorquellen des Gummis in einer Mischung aus Öl und Devulkanisationsmittel einen homogeneren Abbau des Netzwerks ermöglicht. Weitere Optimierungen der Devulkanisationsparameter in Hinblick auf den Anteil des Devulkanisations-Öls und die Reaktionszeit wurden erarbeitet. Durch die Verwendung dieser optimierten Devulkanisationsbedingungen kann eine deutliche Verringerung der Vernetzungsdichte erreicht werden.

Figures and Tables:
By a kind approval of the authors.

Tires consist of several types of vulcanized rubber and various reinforcing materials. When a complete passenger car tire at the end of its life cycle is ground, the resulting rubber powder on average has a composition as given in Table **1** [1]. This blend of several types of rubbers makes devulcanization rather complicated. Many attempts to recycle ground tire rubber (GTR) via mechanical [2,3], mechanochemical [4-6], thermomechanical [7-9] and thermochemical processes [1,10] have been reported. However, the resulting devulcanizates have poor properties. They can be used only in low quality rubber products or in very small quantities in high performance rubber products. Amongst all processes, the thermo-chemical processes is the preferred one in order to produce high quality devulcanizates due to the selective breakdown of the crosslink network.

In thermo-chemical devulcanization, several factors affect the properties of the devulcanized GTR (D-GTR). One of predominant problems is inhomogeneous devulcanization which is caused by poor diffusion of the devulcanization aid into the rubber particles. Swelling of GTR in an oil containing the chemicals before devulcanization was reported as a way to improve the diffusion and consequently the dispersion: Plasticizers such as terpenes and pine oil were added to ground rubber together with devulcanization aids. The softeners swell and soften the surface, and facilitate penetration of the reactive chemicals into the particle [1].

Within this study, GTR was devulcanized using the optimal conditions which are a compromise for all single types of elastomers used in a passenger car tire. A study was performed concerning the devulcanization efficiency, measured as the tendency for crosslink versus main chain scission. Furthermore, the dispersion of DPDS in the rubber matrix and its significance for a homogenous breakdown of the crosslink network was monitored. Finally, the devulcanization conditions for GTR are optimized. Furthermore, the mechanisms of the breakdown processes of GTR are discussed.

Experimental

Materials

The ground rubber from whole passenger car tires used in this investigation was obtained from Rubber Resources BV, the Netherlands. It was mainly based on synthetic rubber and had an average particle size of 0.35 mm. TDAE oil used as processing oil for the devulcanization was supplied by Hansen & Rosenthal, Germany. Diphenyldisulfide (DPDS) used as devulcanization aid was obtained from Sigma-Aldrich, Germany. The solvents, acetone and tetrahydrofuran (THF), which were used for extractions, and toluene, which was used for equilibrium swelling measurements, were obtained from Biosolve.

Preparation of devulcanizates

Devulcanization. Thermo-chemical devulcanization was performed batchwise in an internal mixer (Brabender Plasticorder PL-2000), having a mixing chamber volume of 50 ml and a cam-type rotor. A fill factor of 0.5 and a constant rotor speed of 50 rpm were used, and the chamber temperature was 220 °C. Initially, the devulcanization of GTR was carried out using the optimized process conditions as elaborated for the tire rubbers from our previous work [11] and given in Table **2**. After adding ground rubber and TDAE oil (5 phr) into the mixer, the DPDS (30 mmol/100 g compound) was added. The devulcanization was carried out under nitrogen atmosphere at 220 °C, and the devulcanization time was

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6 minutes. After devulcanization, the material was taken out of the internal mixer and directly quenched in liquid nitrogen.

Further studies with the purpose to optimize the devulcanization process conditions for GTR were performed. The optimization of the devulcanization process parameters for GTR was done with respect to three factors, and for the successive steps the earlier optimized conditions were applied.

- Effect of swelling before devulcanization. – This experiment was performed batchwise in an internal mixer. Before putting the rubber into the mixer, GTR was swollen in a mixture of TDAE and DPDS for 30 and 60 minutes at 65 °C, as the temperature at which DPDS is dissolved in TDAE. The devulcanization was carried out under nitrogen atmosphere at 220 °C, and the process time was 6 minutes. After devulcanization, the material was taken out of the internal mixer and directly quenched in liquid nitrogen.
- Effect of amount of devulcanization oil. – In this study, various amounts of TDAE oil were applied: 5 %, 15 %, 30 % and 50 % relative to the GTR content. The GTR was then swollen in the mixture of TDAE and DPDS for 30 minutes. The devulcanization was carried as described above.
- Effect of devulcanization time. – In this series of experiments, the devulcanization time was varied: 6 and 10 minutes. The GTR was pre-swollen in a mixture of TDAE and DPDS for 30 minutes, using 30 % TDAE oil. The devulcanization was carried out under nitrogen atmosphere at 220 °C. After devulcanization, the material was taken out of the internal mixer and directly quenched in liquid nitrogen.

Characterization of the devulcanizates
Rubber soluble fraction. The soluble (Sol) and insoluble (Gel) fractions of the reclaimed materials were determined by extraction in a Soxhlet apparatus. The vulcanized and devulcanized samples were extracted initially for 48 hours in acetone in order to remove polar, low molecular weight substances like remains of accelerators and curatives, followed by an extraction for 72 hrs in THF to remove the apolar components: oil and non-cross-linked polymer residues or soluble polymer released from the network by the devulcanization process. The extraction

Tab. 1: Ground tire rubber composition

Composition	Content, phr
Polymer Base	100
Natural rubber	30
SBR (styrene-butadiene rubber)	40
BR (butadiene rubber)	20
Butyl- and halogenated butyl rubber	10
Carbon black	32-36
Free textile <1,0 mm	0.8
Free textile > 1,0 mm	1.2

Tab. 2: Initial devulcanization conditions

Factors	Conditions
Devulcanization aid	DPDS 30 mmol/100 g compound
Devulcanization oil	TDAE 5 % w/w
Devulcanization temperature	220 °C
Devulcanization conditions	With nitrogen gas purging
Dumping condition	In liquid nitrogen

was followed by drying the samples in a vacuum oven at 40 °C and determining the weight loss until constant weight. The sol fraction was defined as the sum of the soluble fractions in acetone and THF. Correction for the oil contained in the original SBR was made. The gel fraction was calculated by Equation 1:

$$\text{Gel fraction} = 1 - \frac{\text{weight of rubber dissolved in solvents}}{\text{weight of pure rubber in the compound}}$$

Crosslink density. The extracted samples were subsequently swollen in toluene for 72 hrs at room temperature. The weight of the swollen vulcanizates was measured after removal of surface liquid with absorption paper. The crosslink density was calculated according to the Flory-Rehner Equations 2 and 3 [12]:

$$\nu_e = \frac{v_r + \chi v_r^2 + \ln(1 - v_r)}{V_s \left(0.5\, v_r \cdot v_r^{1/3}\right)}$$

with

$$v_r = \frac{m_r}{m_r + m_r\,(\rho_r/\rho_s)}$$

where:
ν_e = crosslink density per unit volume;
v_r = polymer volume fraction of the swollen sample;
V_s = solvent molar volume;
m_r = mass of the rubber network;
m_s = weight of solvent in the sample at equilibrium swelling;
ρ_r = density of the rubber;
ρ_s = density of the solvent;

χ = Flory-Huggins polymer-solvent interaction parameter, (taken 0.40 as an average value of SBR, BR and NR in toluene). The Flory-Rehner equation is strictly spoken only valid for non-filled systems. Therefore the Kraus [13] correction for filled compounds was used to give the correct values of the different crosslink densities. In its simplified form, the Kraus correction is given by Equation 4 [14]:

$$\nu_{actual} = \frac{\nu_{apparent}}{1 + K + \Phi}$$

with

$$\Phi = \frac{\text{weight fraction of fillers x density of compound x } W_b}{\text{density of fillers x } W_a}$$

where:
$\nu_{apparent}$ = the measured chemical crosslink density;
ν_{actual} = the actual chemical crosslink density;
K = a constant for a given filler;
Φ = the volume fraction of filler in the specimen which is calculated;
W_b = the weight of the specimen before extraction;
W_a = the weight of the specimen after extraction.
This calculated crosslink density is based on the volume of gel rubber in the rubber network after extraction. However, in order to compare these data with the Horikx theory, it has to be realized that the latter defines the crosslink density ν_{real} after devulcanization with the sol fraction still present. During the swelling test, this sol fraction is also extracted and therefore needs to be included again in the calcula-
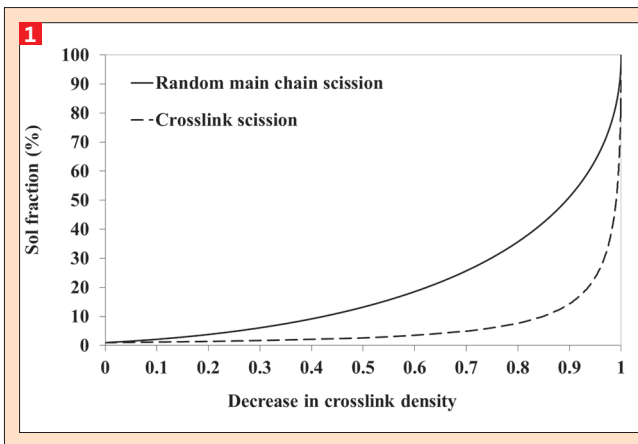
Fig. 1: Random main chain scission and crosslink scission curves in a Horikx plot
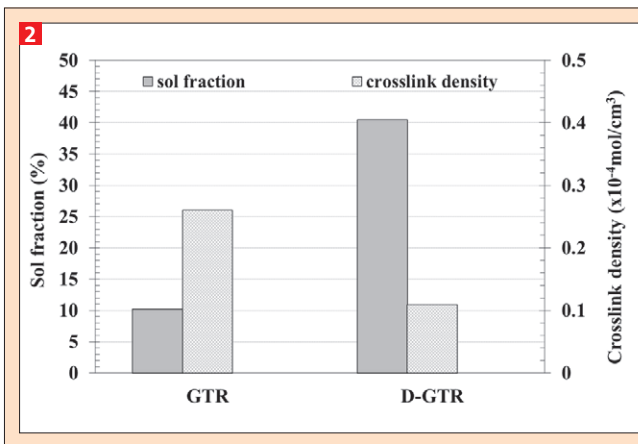

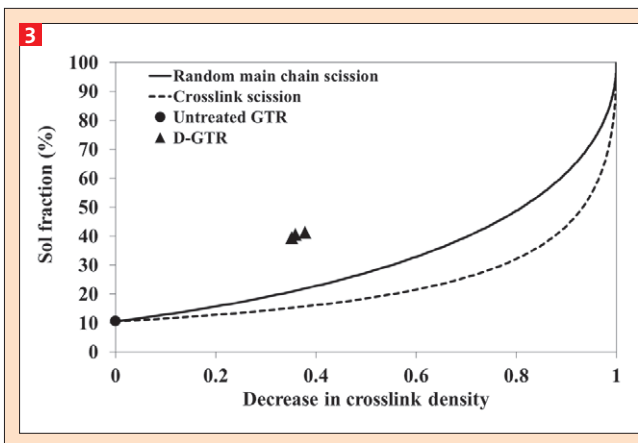Fig. 2: Sol fraction and crosslink density of GTR and D-GTR

Fig. 3: Sol fraction generated during devulcanization versus the relative decrease in crosslink density of devulcanized GTR using initial conditions (devulcanization time: 6 minutes, oil: 5% TDAE, without swelling)

tion of the real crosslink density ν_{real} after the devulcanization but before the extraction. Consequently, in order to obtain the actual remaining crosslink density of the devulcanizate, the volume of total rubber is needed to be taken into account. Correction for this real crosslink density was made according to Equation 6:

$$\nu_{real} = \frac{\text{Number of crosslinks}}{\text{Volume of total rubber}} = \nu_{actual} \times (1\text{-sol fraction})$$

where

ν_{real} = the final corrected crosslink density for the devulcanizate.

Devulcanization efficiency. A useful tool to further understand the devulcanization mechanism is the method developed by Horikx [15]: the rubber sol fraction of the devulcanizates and the crosslink density of the rubber gel fractions are correlated. Horikx derived a theoretical relationship between the soluble fraction generated after degradation of a polymer network and the relative decrease in crosslink density, as a result of either main-chain scission or crosslink breakage. This treatment of polymer degradation can equally well be applied to rubber

reclaiming, where also a mix of main-chain scission and crosslink breakage takes place. When main-chain scission takes place, the relative decrease in crosslink density is given by Equation 7:

$$1 - \frac{\nu_f}{\nu_i} = 1 - \left[\frac{\left(1 - s_f^{1/2}\right)^2}{\left(1 - s_i^{1/2}\right)^2} \right]$$

where s_i is the soluble fraction of the rubber network before degradation or reclaiming, s_f is the soluble fraction of the reclaimed vulcanizate, ν_i is the crosslink density of the network prior to treatment and ν_f is the crosslink density of the reclaimed vulcanizate. For pure crosslink scission, the soluble fraction is related to the relative decrease in crosslink density by Equation 8:

$$1 - \frac{\nu_f}{\nu_i} = 1 - \left[\frac{\gamma_f \left(1 - s_f^{1/2}\right)^2}{\gamma_i \left(1 - s_i^{1/2}\right)^2} \right]$$

where the parameters γ_f and γ_i are the average number of crosslinks per chain in the insoluble network after and before reclamation, respectively. The values for γ_f and γ_i are determined as described by Verbruggen [16]. Figure **1** gives a graphical representation of Equations 7 and 8. The curves in this figure correspond to the situation where only main chains are broken (solid curve) and where only crosslinks are broken (dashed curve). In the case of crosslink scission only, almost no sol is generated until most of the crosslinks are broken; only then the long chains can be removed from the network. In the case of main-chain scission, sol is produced at a much earlier stage, because random scission of the polymers in the network results in small loose chains, which can easily be removed.

Results and Discussion

Devulcanization using conditions as elaborated for tire rubbers

The sol fractions and crosslink densities of untreated GTR and GTR devulcanizates (D-GTR) are shown in Figure **2**. When using the initial conditions as elaborated for the tire rubbers from previous work [11], the devulcanization process results in a significant increase in sol fraction and decrease in crosslink density.

The sol fraction of GTR devulcanizates as a function of the relative decrease in crosslink density is shown in Figure **3**. It can clearly be noticed that the D-GTR

experimental data are clustered on the right hand side of the graph, which indicates that the crosslink density of the treated rubber is reduced. However, they are located above the line of main-chain scission. This may be attributed to an inhomogeneous process: the devulcanization is not uniform throughout the rubber particle, but outer layers of the particles are devulcanized and peeled off, while the inner cores of the particles stay more or less unchanged at the initial crosslink density. This inhomogeneity in devulcanization causes in actual practice a smaller decrease in crosslink density at a particular sol fraction than would have been obtained for homogeneous breakdown.

The optimal devulcanization conditions used in this study are a good compromise of the conditions for the single elastomers used in a passenger car tire, and it is expected to work also for real ground tire rubber. However, in actual practice not only the various types of rubber have to be taken into account, but also the ratio of the different rubber types in conjunction with the filler and oil contained in the material. Therefore, the devulcanization conditions need to be optimized again taking these factors into consideration. The peeling-off mechanism as concluded from the first experiment gives an indication how to improve the process: the devulcanization aid has to be more homogenously distributed within the rubber particles. The method of choice to achieve this is swelling of the rubber powder in a blend of oil and devulcanization aid prior to the devulcanization process.

Effect of the swelling period before devulcanization
Homogeneous devulcanization is one of the main factors affecting the devulcanization efficiency. Basically, a devulcanization oil, TDAE, is used with the aim of improving the dispersion of the devulcanization aid DPDS into the elastomer. Once DPDS is homogeneously dispersed within the rubber particles, a more homogeneous devulcanization will be achieved. The sol fractions of the GTR devulcanizates after various swelling times as a function of the relative decrease in crosslink density are shown in Figure **4**.

A significant improvement of the devulcanization efficiency is observed after the swelling process. For D-GTR swollen for 30 minutes, the experimental data
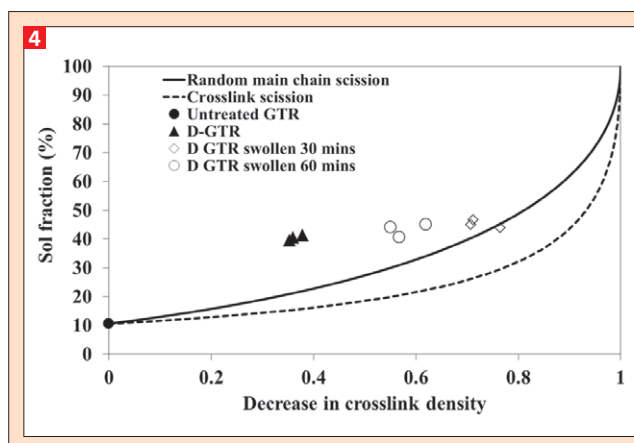

Fig. 4: Sol fraction versus relative decrease in crosslink density of devulcanized GTR at various swelling periods (devulcanization time: 6 minutes, oil: 5% TDAE)
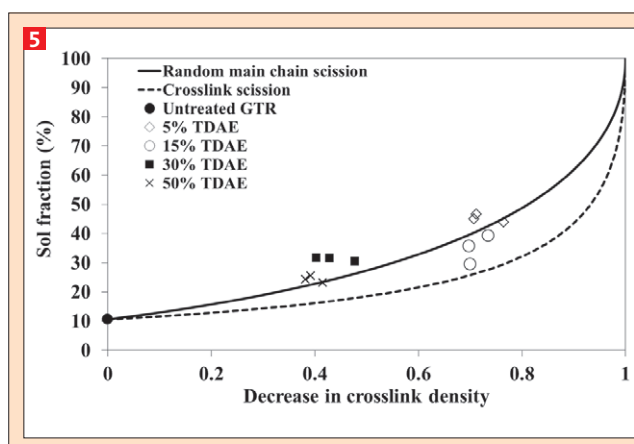

Fig. 5: Sol fraction generated during devulcanization versus the relative decrease in crosslink density at various amounts of devulcanization oil (devulcanization time: 6 minutes, swelling period: 30 minutes)

move to right hand side with about a 30% further decrease in crosslink density compared to the un-swollen D-GTR. This means that swelling is a necessary process step for GTR devulcanization, as it increases the percentage of soluble polymer only slightly, but it significantly reduces the crosslink density. The improvement of the devulcanization efficiency may be attributed to the good dispersion of DPDS throughout the polymer particles which occurred during the swelling step. Moreover, some other chemicals involved in devulcanization might be activated during the swelling period. The differences observed between 30 and 60 minutes of swelling time are small. It is not clear from this study why the data points for 60 minutes swelling time are positioned left of the data points for 30 minutes swelling time, this needs further investigation. However, it is obvious that 30 minutes swelling is sufficient to reach an acceptable dispersion of the DPDS in the 0.35 mm size ground rubber particles.

Effect of the amount of devulcanization oil
Further experiments with the purpose of optimizing the devulcanization process in terms of concentration of the devulca-

nization oil were performed. Various amounts of TDAE oil were applied in the swelling step with the aim of improving the DPDS dispersion in the elastomer. The sol fractions of the GTR devulcanizates as a function of the relative decrease in crosslink density are shown in Figure **5**. An increasing TDAE oil amount from 5% to 15% by weight causes the experimental data points in the Horikx plot to move to the right hand side, to a further reduction of the crosslink density. However, when using a very high amount of TDAE oil, 30% and 50% by weight, the data points turn back to the left hand side, to higher final crosslink densities: the exceeding oil results in an inefficient devulcanization. An explanation of this effect might be that shearing forces are less effective when the material has a lower viscosity and is lubricated by the excess oil.

Effect of devulcanization time
Figure **6** shows the sol fractions of GTR devulcanizates at 2 different devulcanization times as a function of the relative decrease in crosslink density: An increasing devulcanization time results in an increase in crosslink density.
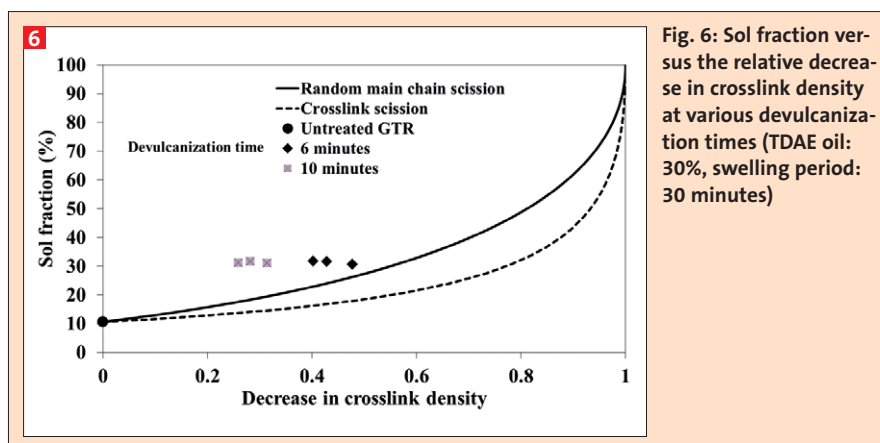
Fig. 6: Sol fraction versus the relative decrease in crosslink density at various devulcanization times (TDAE oil: 30%, swelling period: 30 minutes)
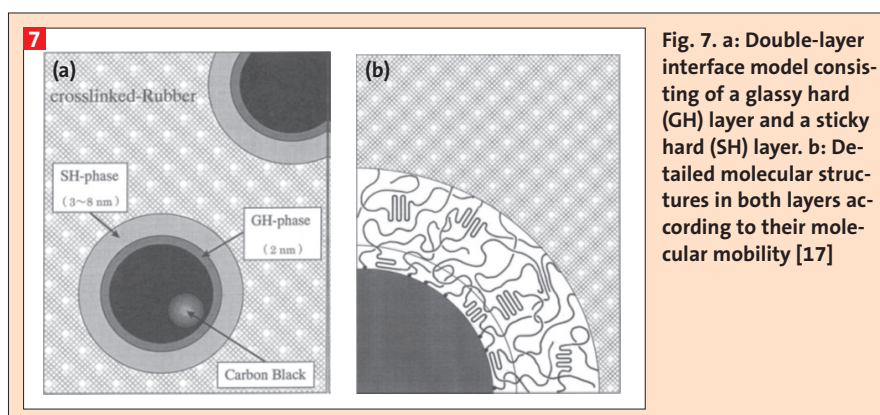

Fig. 7. a: Double-layer interface model consisting of a glassy hard (GH) layer and a sticky hard (SH) layer. b: Detailed molecular structures in both layers according to their molecular mobility [17]

3 Tab. 3: Various experimental devulcanization conditions

Factors	Conditions
Swelling time	0, 30, 60 minutes
Devulcanization oil	5, 15, 30 and 50 % w/w
Devulcanization time	6 and 10 minutes

4 Tab. 4: Final optimal devulcanization conditions for the GTR

Factors	Conditions
Devulcanization aids	DPDS 30 mmol/100 g compound
Devulcanization oil	TDAE 5 % w/w
Swelling time	30 minutes
Swelling temperature	65 °C
Devulcanization time	6 minutes
Devulcanization temperature	220 °C
Devulcanization atmosphere	With nitrogen gas purging
Dumping condition	Exclusion from air/oxygen

Basically, two reactions can occur during devulcanization: chain scission and recombination of active chain segments. It is crucial to achieve a breakdown of the polymer network as far as possible before the re-formation of polymer-polymer bonds from active chain fragments becomes the prevailing reaction. To avoid this, the devulcanization time should be as short as possible.

Mechanistic considerations

The devulcanization of GTR is rather complicated since there are various aspects to be considered. The main factors involved are the presence of several types of elastomers in the different tire parts, various types of fillers and, as a consequence, different interactions between elastomers and fillers. When applying the devulcanization parameters as elaborated for the model materials, that is the best compromise of the devulcanization parameters for the single types of elastomers used in a passenger car tire, inhomogeneous devulcanization occurs: outer layers of the particles are devulcanized and peeled off, while the inner cores of the particles stay more or less unchanged at the initial crosslink density. Basically, in thermo-chemical devulcanization, there are three main factors contributing to the efficiency of the process: temperature, the chemical reaction and shear forces. However, the present experimental work is carried out using a lab scale internal mixer that allows variation of only the temperature and devulcanization aid, with little shear. Therefore, the dispersion of the devulcanization aid plays a major role for the efficiency of the process and the prevention of inhomogeneous devulcanization.

Based on the results, the optimized devulcanization conditions for GTR are given in Table **4**. By using the optimum devulcanization conditions, D-GTR reaches a 70 % decrease in crosslink density compared to the virgin GTR. This seems to be a limit for GTR devulcanization. The remaining 30 % of crosslink density are partly due to remaining network and filler-polymer interactions commonly known as "bound rubber". Bound rubber being rubber physically bound to carbon black before vulcanization, which cannot be released or dissolved in a solvent. 30 % Bound rubber is quite normal for carbon black filled rubber. This puts a limit to the analysis of the devulcanization with swelling tests. Figure **7** shows the rubber-filler interface model of a carbon black filled rubber: a double-layer model consisting of an inner polymer layer in a glassy state, a glassy hard or GH layer, and the outer polymer layer which is characterised as sticky hard or SH layer [17]. The molecular motion in both layers is considerably constrained [17,18] due to a strong molecular packing. The molecular structure of the GH layer is rather fixed and more immobile than that of the SH layer as shown in Figure 7 (b). The devulcanization process is focused on breaking of sulfur crosslinks, therefore strong filler-polymer bonds will not be affected. This polymer-filler network will not be broken during the devulcanization process, and will therefore remain as gel fraction within the rubber. Therefore, 100 % sol will be never reached even if all crosslinks are broken because of the bound rubber remaining bound to the carbon black.

Conclusions

The devulcanization of GTR is more complex than just finding the best compromise of the devulcanization parameters for the single polymers; a further optimization for this blend of polymers and compounds was required. The most efficient method for increasing the devulcanization efficiency is an improvement of the dispersion of the devulcanization aid (DPDS) in the rubber matrix by pre-swelling of the rubber powder in a blend of the devulcanization aid and oil. This results in a more homogeneous breakdown of the crosslink network throughout the rubber particles. After further optimizing the devulcanization parameters in terms of devulcanization oil loading and process time, D-GTR reaches a 70% decrease in crosslink density compared to the untreated GTR. However, this limit might be caused by bound rubber, which in no case is soluble.

Acknowledgements

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VERANSTALTUNG

Science meets Tires – Perspectives for Tire Technology

IKV, IKA Das Institut für Kunststoffverarbeitung (IKV) in Industrie und Handwerk an der RWTH Aachen und das Institut für Kraftfahrzeuge (ika) der RWTH Aachen laden zur internationalen Fachtagung „Science meets Tires – Perspectives for Tire Technology" am 11. und 12. September nach Aachen ein. Beide Institute organisieren die englischsprachige Veranstaltung zum zweiten Mal nach 2011; als Moderator konnten sie Dr. Gerard Nijman von Apollo Vredestein B.V., Enschede, Niederlande, gewinnen.

Der interdisziplinäre Austausch zwischen Experten aus Industrie und Forschung widmet sich der Bedeutung des Reifens für die Automobilindustrie: Der Reifen ist das Bindeglied zwischen Fahrzeug und Straße. Er beeinflusst die Kraftübertragung und die Fahrdynamik. Mit wachsenden Ansprüchen aus der Automobilindustrie wachsen deshalb die Anforderungen an die Reifenbranche. Maximale Effizienz, Sicherheit und Komfort sind dabei Kernfragen. Antworten darauf finden sich entlang der gesamten Wertschöpfungskette – über Forschung und Produktentwicklung hin zum Rohstofflieferanten und Reifenhersteller bis zur Qualitätssicherung.

Die Vorträge und Diskussionen dieser Fachtagung decken diese gesamte Wertschöpfungskette ab und geben einen Ausblick auf zukünftige Trends und Entwicklungen. Im Fokus der diesjährigen internationalen Fachtagung in englischer Sprache stehen

Foto: @Stefan Redel-Fotolia

Design, Dimensionierung und Tests der Reifen. Anforderungen der Automobilindustrie und innovative Prozesstechnologie werden ebenso vorgestellt.

Die Referenten kommen von Apollo Tyres, Niederlande, Ford Motor Company, USA, Goodyear, Luxemburg, Tyre Industry Research, Großbritannien und aus Unternehmen und Institutionen aus Deutschland. Hierzu gehören cosin scientific software, Deutsches Institut für Kautschuktechnologie, Ford Research, Fraunhofer ITWM, Harburg-Freudenberger Maschinenbau, Porsche, Volkswagen, und das ika und IKV an der RWTH Aachen.

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