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Strontium ferrite filled rubber composites based on EPDM

Strontium ferrite was compounded with ethylene-propylene-diene monomer rubber (EPDM) to prepare rubber magnetic composites. Traditional sulfur as well as peroxide curing system were applied for cross-linking of the rubber matrices. The main objective of the work was to investigate the influence of magnetic filler content and type of curing system on the cross-linking, magnetic and physical-mechanical properties of the prepared composites. The results of the study revealed that EPDM based magnetic composites can be efficiently prepared by applying of both, sulfur and peroxide curing systems. Slightly higher values of tensile strength were found to have composites cured with peroxide curing system, what can be attributed to the suitable combination of dicumyl peroxide and co-agent (i.e. ethylene glycol dimethacrylate). The application of strontium ferrite into both type composites leads to a significant improvement of the remanent magnetic induction.

Keywords: rubber magnetic composite, strontium ferrite, peroxide vulcanization, sulfur vulcanization, cross-link density.

Kompozyty oparte na kauczuku EPDM napełnione ferrytem strontu

Do kauczuku etylenowo-propylenowo-dienowego (EPDM) wprowadzono ferryt strontu w celu uzyskania magnetycznych kompozytów gumowych. Do wulkanizacji mieszanek kauczukowych użyto zarówno tradycyjnych siarkowych, jak i nadtlen-kowych zespołów sieciujących. Głównym celem pracy było zbadanie wpływu zawartości magnetycznego napełniacza oraz rodzaju zespołu wulkanizującego na właściwości magnetyczne i fizyko-mechaniczne otrzymanych materiałów kompozyto-wych. Wyniki uzyskanych badań potwierdziły możliwość efektywnego otrzymywania kompozytów gumowych zarówno poprzez wulkanizację siarkową, jak i nadtlenkową. Nieznacznie większą wytrzymałość na rozciąganie kompozytów usieciowanych za pomocą nadtlenkowego zespołu wulkanizującego można przypisać odpowiedniej kombinacji nadtlenku dikumylu i koagenta (tzn. dimetakrylanu glikolu etylenowego). Zastosowanie ferrytu strontu w obu rodzajach kompozytów przyczyniło się do znacznego zwiększenia szczątkowej indukcji magnetycznej.

Słowa kluczowe: magnetyczne kompozyty gumowe, ferryt strontu, wulkanizacja nadtlenkowa, wulkanizacja siarkowa, gęstość usieciowania.

1. Introduction

Rubber magnetic composites belong to the relatively new class of smart materials that can be prepared by incorporation of magnetic powdery fillers into different rubber matrices. Due to their elasticity and easy processability, there are suitable for applications, in which good elasticity and high flexibility are required. These attributes in combination with unique magnetic characteristics rank them among the progressive developing materials, which have already found the utilization in motor parts, sensors of magnetic and electromagnetic fields, vibration absorbers, microwave and radar technology, variable impedance surfaces, memory devices, inductor cores and in many other technological applications [1–3].

The unique, highly elastic properties of rubber materials are achieved in the process of vulcanization which is among engineers simply termed as curing. It is a chemical process which leads to the forming of three-

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dimensional cross-linked network structure within the rubber matrix, by reactions between functional groups of elastomer chains and suitable curing agents. Chemical cross-links between rubber chains are formed by introduction of different vulcanization systems. Sulfur vulcanization is the oldest and the most widely used method for cross-linking of unsaturated rubbers. It is a complex process which leads to the forming of different types of sulfidic cross-links between macromolecules of rubber, namely monososulfidic C–S–C, disulfidic C–S₂–C and polysulfidic cross-links $C-S_x$ –C (x = 3-6) [4, 5].

Peroxides can be used to vulcanize both unsaturated and saturated rubbers [6]. Cross-linking of elastomers with organic peroxides is radical process which results in forming of carbon–carbon bonds between macromolecular chains. The main benefit of C–C bonds is their high thermal stability, therefore peroxide cured elastomers exhibit hightemperature ageing resistance and low compression set at elevated temperatures. However, there are also some disadvantages compared to sulfur cured systems, as low scorch safety, worse dynamic and elastic properties of vulcanizates [5, 7].

As the cross-linking efficiency of peroxides is sometimes rather low, the so-called co-agents are often introduced to boost peroxide vulcanization [8, 9]. Coagents are mostly polar vinyl monomers with high affinity toward free radicals and readily graft to macromolecular chains to form a complex cross-linked network [10]. They are used to increase the cross-linking efficiency of the vulcanization process but to increase the cross-link density of vulcanizates as well [10–12]. Because of this, also physical properties and process ability of peroxidecured elastomers are improved. The objective of the present work was to investigate the influence of strontium ferrite content on the cross-linking and properties of rubber composites based on ethylene-propylene-diene monomer rubber (EPDM). For cross-linking of rubber magnetic composites, the sulfur and peroxide curing system were applied.

2. Experimental

2.1. Materials

Ethylene-propylene diene monomer rubber (EPDM 70) provided by Zenith Industrial Rubber Products, Mumbai, India was chosen as rubber matrix. Strontium ferrite SrFe₁₂O₁₉, type FD 8/24, supplied as magnetic filler by Magnety, Světlá Hora, Czech Republic was dosed to the rubber formulations in concentration scale ranging from 0 to 150 phr. The SEM images of applied strontium ferrite are presented in Fig. 1 and its characteristics are mentioned in Tab. 1. Dicumyl peroxide (DCP) as peroxide curing agent and ethylene glycol dimethacrylate (EGDMA) as co-agent were provided by Merck Schuchardt OHG, Germany. Besides this, the components of sulfur curing system consisting of zinc oxide (Slovlak, Košeca, Slovakia), stearic acid (Setuza, Ústí nad Labem, Czech Republic), N-cyclohexyl-2-benzothiazole sulfenamide (CBS) (Duslo, Šal'a, Slovakia) and sulfur (Siarkopol, Tarnobrzeg, Poland) were also used. The composition of rubber magnetic composites cured with either peroxide or sulfur curing system is summarized in Tab. 2 and 3, respectively.



Fig. 1. SEM images of strontium ferrite **Rys. 1.** Zdjęcia SEM ferrytu strontu

Table 1. Characteristics of strontium ferrite Tabela 1. Właściwości ferrytu strontu

Characteristics	Value		
Particle size (µm)	1–70		
Specific surface area (m ² /g)	3.3		
Total porosity (%)	54.94		
Specific volume of pores (cm ³ /g)	0.264		
Density (g/cm ³)	4.13		
Coercivity (kA/m)	116		
Remanent magnetic induction (T)	0.127		

 Table 2. Composition of rubber magnetic composites with peroxide curing system

 Tabela 2. Skład magnetycznych kompozytów gumowych z nadtlenkowym zespołem sieciującym

Component	EPDM	DCP	EGDA	Ferrite
Content (phr)	100	1.5	1.5	0–150

Table 3. Composition of rubber magnetic composites with sulfur curing system

Tabela 3. Skład magnetycznych kompozytów gumowych z siarkowym zespołem sieciującym

Component	EPDM	ZnO	Stearic acid	Sulfur	CBS	Ferrite
Content (phr)	100	3	2	1.5	1.5	0-150

2.2. Methods

2.2.1. Preparation and curing of rubber compounds

The rubber compounds were prepared in a laboratory mixer Brabender in the two mixing steps. The temperature of chamber was set up to 90 °C and the mixing process was carried out at the rotor speed of 50 rpm. In the first step, the rubber and the filler were compounded. The mixing of ingredients in the first step was performed for 9 min at a temperature range 90–95 °C. The components of curing systems were introduced in the second step (4 min, 90–95 °C). After that, the mixed rubber compounds were shaped into thin sheets by using the two roll calender.

The curing characteristics were investigated from the corresponding curing isotherms measured by Rheometer Monsanto R100 at 160 °C. The rubber compounds were cured at 160 °C regarding their optimum cure time under a pressure of 15 MPa. The hydraulic press FONTIJNE was employed to carry out the curing process.

2.2.2. Determination of cross-link density of composites

The cross-link density v of composites was determined based on swelling of samples in xylene, in which the samples were swelled within the time. The weight of samples was measured every hour until the equilibrium swelling was reached. The experiments were performed in a laboratory conditions and a swelling time was 30 hours. When an equilibrium swelling degree was reached, the Krause modified Flory–Rehner equation (1) for filled vulcanizates was used to evaluate the cross-link density [13].

$$\nu = -\frac{V_{\rm r0}}{V_{\rm S}} \frac{\ln\left(1 - V_{\rm r}\right) + V_{\rm r} + \chi V_{\rm r}^2}{V_{\rm r}^{1/3} V_{\rm r0}^{2/3} - 0,5 V_{\rm r}}$$
(1)

v – cross-link density (mol/cm³)

 $V_{\rm r0}$ – volume fraction of rubber in equilibrium swelling sample of vulcanizate in absence of fillers

 $V_{\rm r}$ – volume fraction of rubber in equilibrium swelling sample of filled vulcanizate

 $V_{\rm s}$ – molar volume of solvent (for xylene = 123.45 cm³/mol) χ – Huggins interaction parameter (for EPDM–xylene χ = 0.5316)

$$V_{\rm r0} = \frac{V_{k0}}{V_{k0} + V_n} \tag{2}$$

 $V_{\rm \scriptscriptstyle n}$ – volume of solvent in sample of vulcanizate in equilibrium swelling

 $V_{\scriptscriptstyle k0}$ – volume of rubber in sample of vulcanizate in absence of fillers

$$V_{k0} = a \cdot \frac{100}{100 + x} \cdot \frac{1}{\rho_k}$$
(3)

$$V_{\rm r} = \frac{V_k}{V_k + V_n} \tag{4}$$

 $V_{\rm b}$ – volume of rubber in sample of filled vulcanizate

$$V_k = a \cdot \frac{100}{100 + (x+p)} \cdot \frac{1}{\rho_k} \tag{5}$$

x – total amount of additives in sample of vulcanizate p – total amount of fillers in sample of vulcanizate a – density of rubber (a – 1.19 g/cm³)

 ρ_k – density of rubber ($\rho_{\text{EPDM}} = 1.19 \text{ g/cm}^3$) *a* – initial weight of sample of vulcanizate before swelling

2.2.3. Determination of physicalmechanical properties of composites

The tensile properties of rubber magnetic composites were determined by employing Zwick Roell/Z 2.5 appliance. The measurements were carried out at in accordance with the valid technical standards at a laboratory temperature and cross-head speed of 500 mm/min. The tested composites were cut into the double side dumbbell-shaped test specimens (width 6.4 mm, length 8 cm, thickness 2 mm). The hardness was measured by using durometer and the unit was expressed in Shore A.

2.2.4. Determination of magnetic properties of composites

Magnetic characteristics of composites were evaluated by using magnetometer TVM-1 at maximum coercivity of $H_{\rm m}$ = 750 kA/m. The basic principle of measurement is induction method of scanning of scattering magnetic flux Φ induced by magnetic vibrating sample. Magnetic field is generated by means of two cores of Weiss electromagnet at a minimum distance of poles adapters 7.5 mm. The induced tension proportional to time dependence of magnetic flux in the sample is scanned with the system of four small cores. The cores eliminate the influence of time instability of electromagnet magnetic fields and the change of magnetic flux is directly proportional to magnetic induction B. The specimens for the magnetic characteristics evaluation were of prism shape $(8 \text{ mm} \times 4 \text{ mm} \times 4 \text{ mm}).$

3. Results and discussion

3.1. Influence of ferrite and type of curing system on the curing characteristics and cross-link density of composites

The influence of ferrite content and type of curing system on the curing process of prepared rubber compounds was considered based on their curing characteristics, the scorch time t_{s1} and the optimum cure time t_{c90} . As seen in Fig. 2, the optimum cure time of rubber compounds cured with sulfur system was almost twofold longer when compared to rubber compounds cured with peroxide and co-agent. With increasing of ferrite loading, the next extension of t_{c90} was observed. The optimum cure time increased from 22 to almost 33 min, when the amount of ferrite increased from 0 up to its maximum content. The optimum cure time of rubber compounds cured with peroxide curing systems moved around 12 min, almost independently on the amount of magnetic filler. The scorch time of both type rubber compounds was relatively low (Fig. 3). The low scorch time of rubber compounds cured with peroxide system was expected. The regulation of scorch safety of peroxide cured elastomers is rather complicated. It is determined mainly by type of peroxide and its ability to dissociate into free radicals at vulcanization temperature. Peroxide free radicals then rapidly react with macromolecules of rubber to form cross-links [14]. The regulation of t_{s1} can only hardly be influenced by concentration of peroxides, or by using inhibitors of radical reactions, because their presence in rubber compounds usually leads to the lowering of cross-linking efficiency.

The scorch time of peroxide cured rubber compounds was about 1 min, without significant influence of the amount of magnetic filler. Although, the scorch time of rubber compounds with applied sulfur system was a bit higher, its values seem to be low, too. Sulfur vulcanization of rubber compounds is much more complex and intricate process. A sequence of parallel and consecutive reactions runs during this process in several steps. By the suitable selection of activators, accelerators, vulcanization retarders, or prevulcanization inhibitors, it is possible to control the scorch time, but also the entire process of sulfur curing. Sulfenamides (CBS), the so-called accelerators with delayed effect, generally exhibits long scorch safety when used in sulfur curing of rubber compounds. Therefore, much longer scorch time of rubber compounds cured with sulfur system was expected. The next decrease of the scorch time was recorded with increasing content of strontium ferrite.

The cross-link density and the structure of crosslinks in vulcanizates are very important characteristics of all cured rubber systems. Not only the original properties, but also the changes of original properties of vulcanizates during their using are dependent on these characteristics. From Fig. 4 it is possible to observe almost no influence of magnetic filler content on the cross-link density of composites. Higher values of cross-link density were found to have composites cured with sulfur curing system.



Fig. 2. Influence of strontium ferrite content and type of curing system on optimum cure time t_{c90} of rubber compounds

Rys. 2. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na optymalny czas wulkanizacji t_{c90} mieszanek kauczukowych



Fig. 3. Influence of strontium ferrite content and type of curing system on scorch time t_{s1} of rubber compounds

Rys. 3. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na czas podwulkanizacji t_{s1} mieszanek kauczukowych



30

Fig. 4. Influence of strontium ferrite content and type of curing system on cross-link density v of composites

60

90

Strontium ferrite content (phr)

120

150

Rys. 4. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na gęstość usieciowania *v* otrzymanych kompozytów

3.2. Influence of ferrite and type of curing system on the physicalmechanical properties of composites

0.0

0

The values of physical-mechanical properties of rubber magnetic composites are graphically illustrated in Figs. 5–8. The hardness of composites was improved by incorporation of magnetic filler, as hardness of ferrite is much higher that of the rubber matrix (Fig. 5). Higher hardness of composites cured with sulfur curing system can be attributed to the higher cross-link density of the corresponding composites. Higher cross-link density of composites cured with sulfur system was also reflected in higher modulus M100 of those composites (Fig. 6). The modulus M100 of the composite with maximum ferrite content was not possible to measure, because this sample was ruptured at deformation less than 100%. The increasing amount of magnetic filler in



Fig. 5. Influence of strontium ferrite content and type of curing system on hardness of composites

Rys. 5. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na twardość otrzymanych kompozytów composite materials leads to the decrease of modulus. Although, the values M100 of composites cured with peroxide system were lower, the increase in ferrite loading resulted in a slight increase of M100. On the other hand, higher cross-link density of composites with applied sulfur curing system was responsible for a lower elongation at break of the equivalent composites (Fig. 7). In the case of both type composites, the elongation at break showed decreasing trend with increasing amount of strontium ferrite. The possible explanation of this could be the fact, that particles of fillers might act as steric hindrance against rubber chains mobility and thus they could restrict the mobility and orientation of macromolecular chain segments. This could be the reason for the deterioration of elastic properties and decrease of elongation at break. From Fig. 8 it becomes apparent that the presence of strontium ferrite has negative influence on tensile strength of composites which exhibited a decreasing tendency with increasing amount of strontium ferrite. As also possible to see, composites cured with peroxide system showed slightly higher tensile strength when compared to equivalent composites cured with sulfur system in all ferrite concentration scale. The results are not in correlation



Fig. 6. Influence of strontium ferrite content and type of curing system on modulus M100 of composites

Rys. 6. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na moduł M100 otrzymanych kompozytów



Fig. 7. Influence of strontium ferrite content and type of curing system on elongation at break of composites

Rys. 7. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na wartość wydłużenia przy zerwaniu otrzymanych kompozytów

with the general theoretical predictions, according to which sulfur cured vulcanizates are characterized by higher tensile stress characteristics in comparison with peroxide cured vulcanizates. This is mainly due to the structure of the formed cross-links. The cross-link redistribution in sulfur cured vulcanizates results in a less stressed, stronger network, in which the stress is uniformly distributed onto higher amount of elastomer chains. Such network stress relaxation results in higher tensile characteristics of cross-linked elastomers. By contrast, rigid and shorter carbon–carbon cross-links restrict the mobility and orientation of macromolecular chains when stretched. Moreover, the formed bonds cause increased deformation stiffness, because of less mobility of macromolecular chains, thus lower mechanical properties of vulcanizates [15].



Fig. 8. Influence of strontium ferrite content and type of curing system on tensile strength of composites

Rys. 8. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na wytrzymałość na rozciąganie otrzymanych kompozytów







It is well known that sulfur vulcanization of rubber compounds is intricate process and the chemistry of accelerated sulfur vulcanization is very complex and still not clearly understood. In general, it is suggested that sulfur vulcanization of elastomers runs in three stages. In the first stage, the interaction of components of curing system leads to the forming of transition complexes, which together with sulfur form active sulfurating agent. The second stage is characterized by forming of primary vulcanizate network with dominance of polysulfidic crosslinks. During the third stage, this network is restructured in consequence of modification of cross-links (polysulfidic cross-links are transferred into di- and monosulfidic cross-links) and macromolecules of rubber (isomerization, dehydrogenation, cyclization) and the final spatial network of vulcanizate is formed (Scheme 1) [16-19].

On the other hand, it is supposed that cross-linking of rubber macromolecules with organic peroxides has radical character in substance. The first step is a homolytic dissociation of peroxides into radicals at high temperatures, their possible fragmentation and subsequent reactions of peroxide radicals with macromolecules of rubber. Peroxide radicals could potentially react by addition to the double bonds or by abstraction of allylic hydrogens from rubber chains. It was also proposed, that the formed elastomer radicals, besides mutual recombination reactions, can also undergo addition reactions to the double bonds in rubber chains. Peroxide cross-linking of EPDM proceeds via both, hydrogen abstraction and addition mechanism (Scheme 2) [20-22]. It has been reported, that in the case of EPDM, the forming of chemical intermolecular bonds by addition of EPDM macroradicals to the double bonds in its unsaturated structural units is practically equivalent with recombination of macroradicals [16].

Based upon the above outlined facts relating to the substance of the cross-links formed during sulfur and peroxide vulcanization, the opposite tendency of tensile strength, in dependence of curing system applied, should be observed. The reason of a higher tensile strength of peroxide cured rubber magnetic composites can be attributed to the composition of peroxide curing system and the presence of co-agent ethylene glycol dimethacrylate. EGDMA as co-agent contributes to the forming of more complex cross-link structure within the rubber matrix on the one hand (Scheme 3) [16, 23].

On the other hand, having polar character, it is expected that it improves the adhesion to polar substrates, like ferrite filler is [24, 25]. Thus, also the improvement of compatibility and adhesion between ferrite and the rubber on the phase interface is achieved, by means of co-agent chemically bound to rubber macromolecules and physically bound to the surface of ferrite particles. As a result, higher tensile strength of peroxide cured composites was achieved.

3.3. Influence of ferrite and type of curing system on the magnetic characteristics of composites

The dependences of magnetic induction B on intensity of magnetic field H are called magnetizing



Fig. 9. Magnetizing curve of magnetic substances Rys. 9. Krzywa magnesowania substancji magnetycznych

curves, which provide complete information about technical properties of magnetic substances. Fig. 9 illustrates the dependence of magnetic induction *B* in the material on intensity of magnetic field *H*, when the sample is cyclically magnetized in the presence of external magnetic field. The remanent magnetic induction B_{p} , which represents the value of residual magnetism of material, when an external magnetic field is removed and the coercive intensity of magnetic field (coercivity, H_{o}), which represents energy needed to abolish the remanent magnetic induction, are the most important parameters of all permanents magnets. Magnetic hard materials, as also ferrites, usually exhibit high values of both magnetic characteristics [25, 26]. The incorporation of strontium ferrite into non-magnetic rubber matrix should impart magnetic characteristics to the rubber composites as well. Therefore, the investigation of magnetic filler content and a possible influence of type of curing system on magnetic properties of rubber composites were under consideration. The values of remanent magnetic induction B_r and coercivity H_c for all prepared composites are graphically illustrated in Figs. 10, 11. From Fig. 10 it becomes clearly obvious that the incorporation of



Fig. 10. Influence of strontium ferrite content and type of curing system on remanent magnetic induction B_r of composites

Rys. 10. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na wartość szczątkowej indukcji magnetycznej *B*_r otrzymanych kompozytów



Fig. 11. Influence of strontium ferrite content and type of curing system on coercivity H_c of composites

Rys. 11. Wpływ zawartości ferrytu strontu oraz rodzaju zespołu wulkanizującego na wartość koercji *H*_c otrzymanych kompozytów

ferrite into rubber matrix leads to a significant increase of the remanent magnetic induction. By contrast, the coercivity of composites seems not to be influenced by the amount of magnetic filler as its values moved in close experimental range (Fig. 11). The differences in B_r and H_c of both types of composites are rather low and based upon the obtained results it might be stated that the type of curing system has almost no influence on the values of observed magnetic properties.

4. Conclusion

In this work, rubber magnetic composites were prepared by incorporation of strontium ferrite in various loading level into EPDM based rubber matrix. Sulfur and peroxide curing system were applied for crosslinking of rubber magnetic composites.

The achieved results revealed that higher crosslink density exhibited composites cured with sulfur system, while the influence of strontium ferrite content on the cross-link density of both type composites was nonsignificant. Higher cross-link density of the sulfur cured composite materials was subsequently reflected in higher hardness and modulus as well as lower elongation at break of the corresponding composites when compared to peroxide cured composites. Although, the tensile strength showed a decreasing trend with increasing amount of magnetic filler, higher values of tensile strength were found to have composites cured with peroxide system. This can be attributed to the composition of peroxide curing system. EGDMA as co-agent contributes to the forming of more complex cross-link structure within the rubber matrix on the one hand and on the other hand, it is expected that it improves the adhesion to ferrite particles. The presence of strontium ferrite in rubber composites leads to a remarkable increase of the remanent magnetic induction which increased with an increasing content of strontium ferrite.

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