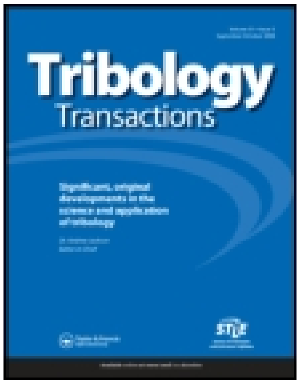

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Effects of Magnesium Borate Whiskers on the Antiwear and Mechanical Performance of Natural Rubber

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Effects of Magnesium Borate Whiskers on the Antiwear and Mechanical Performance of Natural Rubber

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Magnesium borate whiskers (MgBWs) are utilized to improve the antiwear and mechanical performance of natural rubber (NR). The results suggest that the wear resistance of the vulcanizates was increased significantly and the mechanical performance of the vulcanizates was increased to some extent with the inclusion of MgBW. Bis-(3-[triethoxysilyl]-propyl)-tetrasulfide (Si-69) and tea polyphenols (TPs) were used to improve the inferior interface between MgBW and the NR matrix and their effectiveness and the relationship of structures and the properties were explored. The micrographs illustrate that the interfacial interactions between the whiskers and matrix were obviously enhanced after surface modification by Si-69. With the incorporation of TP, the wear resistance of the vulcanizates was further increased. The present investigations attribute the improvement of antiwear properties of NR to the high antiwear ability of whiskers and the improved mechanical performance of the vulcanizates.

KEY WORDS

Rubber; Antiwear; Surface Modification; Magnesium Borate Whiskers; Mechanical Properties

INTRODUCTION

It is well known that the properties related to wear resistance and friction-reducing ability are crucial for rubber materials (Park, et al. *(1)*; Rattanasom, et al. *(2)*; Pongdhorn, et al. *(3)*; Van Der Heide, et al. *(4)*; Findik, et al. *(5)*; Jin and Park *(6)*). For instance, the wear resistance and brake performance of rubber tires are the key factors that influence the safety, driving efficiency, and economy of the car (Moore *(7)*). Recently, many methods and technologies have been developed to decrease the abrasion of rubber (Karnath, et al. *(8)*; Kuriakose and De *(9)*; Yang, et al. *(10)*; Feng and Yang *(11)*), including surface chemical modification such as fluoridation, sulfonation, bromination, the use of coating films with high antiwear properties (such as polytetrafluoroethylene, etc.), and incorporation of antiwear additives such as molybdenum disulfide and graphite. Unfortunately, some difficulties, including deterioration of the mechanical properties of the matrix, process complexity, and increased costs, may occur during the improvement of abrasive resistance. The wear resistance of rubber is related to the comprehensive effect of a variety of properties, including abrasive resistance, lubricity, and mechanical properties.

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Inorganic whiskers, a kind of needle-like fiber material with a single-crystal structure growing in artificial control conditions, is a type of reinforcing material for composites with high performance (Xu, et al. *(12)*; Liang *(13)*). Boron and boron compounds, which possess excellent antiwear ability and lubricity, have been widely used to improve the antiwear ability and lubricity of materials (especially for metal materials; Liu, et al. *(14)*; Radev and Zakhariev *(15)*). Organic borates used as lubricant additives have received much attention in recent years for the good combination of properties such as wear resistance, friction-reducing ability, oxidation inhibition, etc. (Hu and Dong *(16)*; Zhang, et al. *(17)*). Magnesium borate whiskers (MgBWs) are a new type of inorganic borate whisker with the combined characteristics of inorganic whiskers and boron compounds, such as high mechanical performance, high wear resistance, and relatively low cost. At present, MgBWs are mainly used in the reinforcement of metal composite materials (Wu, et al. *(18)*) and recently the use of inorganic borate whiskers for reinforcement of plastics has been reported (Ljungqvist, et al. *(19)*; Liang and Hu *(20)*; Zhu, et al. *(21)*). To our knowledge, the application of inorganic borate whiskers to enhance the antiwear and mechanical performance of rubber has not been reported.

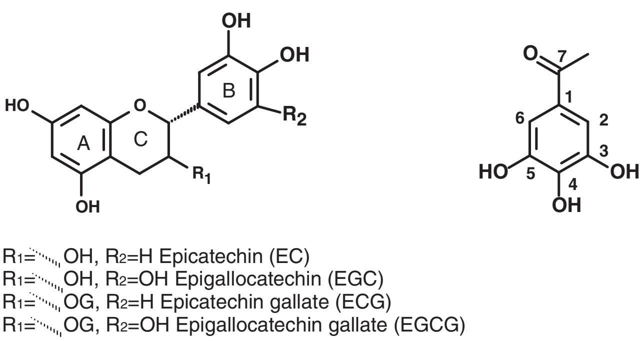
In the present work, MgBWs were introduced to natural rubber (NR) to improve the antiwear properties. It was expected that the high antiwear ability and high mechanical performance of MgBW would be beneficial for the improvement of antiwear property and mechanical performance of the rubber matrix.

The effectiveness of MgBWs and surface modification and tea polyphenols (TPs) on the mechanical and antiwear performance of NR and the relationship of structure and the properties were explored.

EXPERIMENTAL SECTION

Materials

Natural rubber (STR 5L) was manufactured in Thailand and provided by Xin Tai He Trade Co., Ltd., Shenzhen, China. MgBWs were supplied by Qinghai Haixing Technology Co., Ltd, XiNing, China. TPs were provided by Xuancheng Baicao Plant Industry and Trade Co., Ltd, XuanCheng, China and the chemical structure is as follows (main chemical composition: EGCG, 45.3%; EGC, 12.3%; ECG, 9.1%; EC, 4.3%):



Sch. 1—Chemical structure of TP.

Bis-(3-[triethoxysilyl]-propyl)-tetrasulfide (Si-69), precipitated silica, sulfur, zinc oxide, stearic acid, accelerators, and other additives were all of industrial grade and used as received.

Surface Modification of MgBWs

An ethanol–water solution (95 vol%) was adjusted to pH 5 with acetic acid in a necked flask. Si-69 (5 g) was added dropwise while stirring. Hydrolysis at 80°C took place during 15 min. Then MgBWs were added to the flask and gently agitated. The mixture was refluxed at 80°C for 5 h and then precipitated and rinsed with the ethanol–water solution three times. The modified magnesium borate whiskers (OMgBW) were vacuum dried at 80°C for 5 h to remove the residual solvent.

Preparation of Vulcanizates

The formulation of NR compounds expressed as parts per hundred rubber (phr) is as follows: sulfur (2.0), zinc oxide (ZnO; 5.0), stearic acid (1.0), N-isopropyl-N′-phenyl-p-phenylenediamine (4010NA; 1.5), tetramethyl thiuram disulfide (TT; 0.5), dibenzothiazole disulfide (DM; 2.0), and precipitated silica (30). The vulcanizates were prepared in an open two-roll laboratory mixing mill at room temperature. Vulcanization was carried out in an electrically heated hydraulic press at 143°C using the optimum cure time (t_{90}) previously determined with a rubber process analyzer.

Characterizations

Mechanical Properties Determinations

An Instron 3367 universal testing machine (Instron, USA) was used to determine the tensile strength of the vulcanizates according to ISO 37; Shore A hardness was measured by an XY-1 type rubber hardness tester according to ISO 7619. The antiwear properties of the specimens (12.7 in length and 3.2 in width) were tested in a GT-7012-A Akron wear loss machine (Gotech Testing Machines Inc., Taiwan) according to ISO 4649to characterize the abrasion performance of rubber with the grinding wheel at a speed of 24 m/min for 90 min under a 26.7 N load and 15° angle.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was carried out under an N_2 atmosphere with a TGA 2050 (TA Instrument, USA) at a heating rate of 10°C/min from 30 to 700°C.

Scanning Electron Microscopy

The fracture surfaces of tensile samples and worn surfaces of Akron wear samples were plated with a thin layer of gold before observations. Scanning electron microscopy (SEM) observations were done using a Hitachi S-3000N SEM (Hitachi, Japan).

X-ray Photoelectron Spectra

X-ray photoelectron spectra (XPS) of MgBWs, TPs, and vulcanizates were recorded using an X-ray photoelectron spectrometer (Kratos Axis Ultra DLD, Kratos, UK) with an aluminum (mono) Kα source (1,486.6 eV). The aluminum Kα source was operated at 15 kV and 10 mA. For all samples, a low-resolution

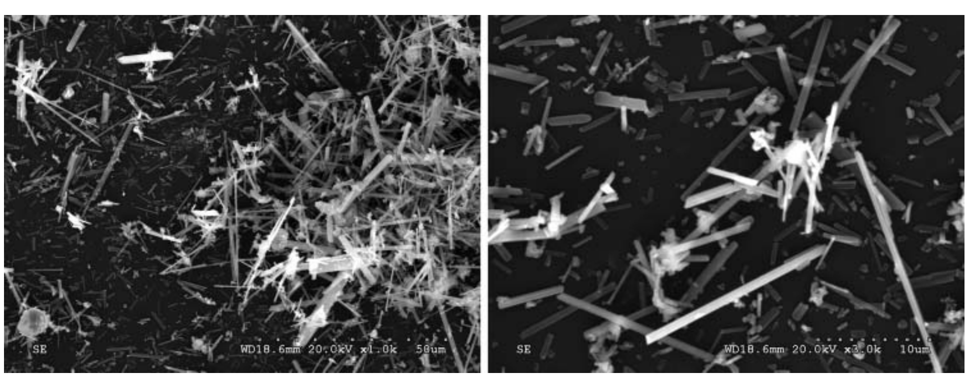


Fig. 1—SEM micrographs of MgBW.

survey run (0–1,100 eV, pass energy = 160 eV) was performed. In order to obtain more information about the formation of hydrogen bonding, a high-resolution survey (pass energy = 48 eV) was performed at spectral regions relating to O and Mg atoms.

RESULTS AND DISCUSSION

Effects of MgBW on the Antiwear and Mechanical Performance of NR

As is well known, MgBWs are a typical type of inorganic borate whiskers, which possess many superior properties such as high modulus, high strength, high antiwear abilities, etc. Consequently, in the present investigations, MgBWs were utilized to improve the antiwear and mechanical performance of NR. Figure 1 shows the micrographs of MgBW. The length of the MgBW varied from several micrometers to tens of micrometers and its diameter was hundreds of nanometers.

Figure 2 shows the specific wear rate of NR/silica/whisker vulcanizates. With the addition of MgBW, the wear resistance of the vulcanizates increased significantly and decreased gradually when the MgBW content exceeded 7.5 phr. From Fig. 2 it is suggested that the optimum content of MgBW was 7.5 phr, at which the specific wear rate was 0.0394 mm^3/N m, an 15.87% reduction compared to that of vulcanizates without MgBW. It is believed that the improvement in antiwear properties resulted from the incorporation of MgBW with high wear resistance and high strength. In the process of friction, a certain amount of MgBW with high wear resistance and high strength plays the role of bearing attrition and shear cut. As a result, the abrasion performance of the vulcanizates increased significantly. In addition, the mechanical performance of the vulcanizates increased to some extent with the inclusion of MgBW, which will be discussed in the section of "Effects of Surface Modification on the Antiwear and Mechanical Performance of NR."

Effects of Surface Modification by Si-69 of MgBW (OMgBW) on the Antiwear and Mechanical Performance of NR

As discussed above, inclusion of small amount of MgBW improves the abrasion performance of NR to some extent.

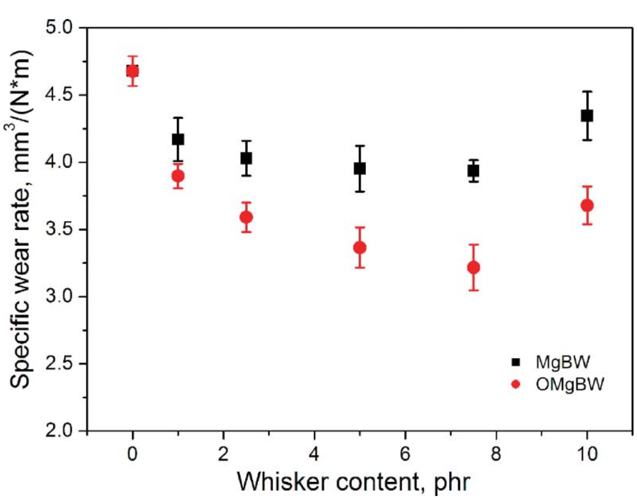


Fig. 2—Specific wear rate of NR/silica/whisker composites. (color figure available online.)

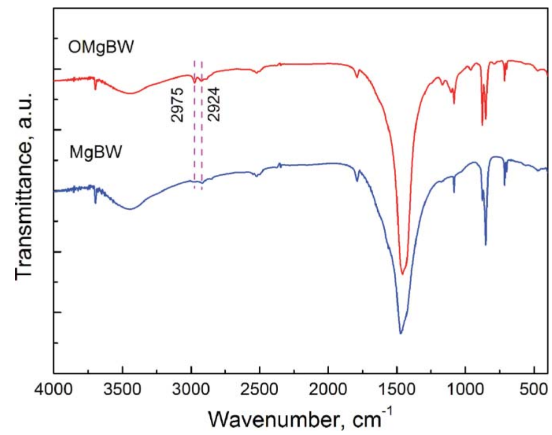


Fig. 3—FTIR spectra of MgBW and OMgBW. (color figure available online.)

However, there is a lack of reactive groups on the surface of MgBW toward the NR matrix and, therefore, it is expected that improvement of the inferior interface between the MgBW and NR matrix can further increase the antiwear property of the vulcanizates. In this investigation, Si-69, a typical coupling agent, was introduced to enhance the interfacial interactions between MgBW and NR matrix.

The MgBW and OMgBW were characterized by Fourier transform infrared spectroscopy (FTIR) and the results are shown in Fig. 3. After the treatment, typical dissymmetrical and symmetrical stretching vibration peaks of -CH_2- around 2925 and 2850 cm^{-1} were observed in the spectrum of OMgBW, indicating the organic surface modification of MgBW by Si-69. In order to further identify the presence of Si-69 on the surface of OMgBW, TGA of MgBW and OMgBW was performed under a nitrogen atmosphere. As shown in Fig. 4, there was almost no decrease in weight within 700°C of the MgBW, indicating its high thermal stability. However, the weight of OMgBW decreased slightly when the temperature increased from 200 to 700°C, which was attributed to the thermal decomposition of the coupling agent grafted to the surface of the OMgBW. In brief, FTIR and TGA

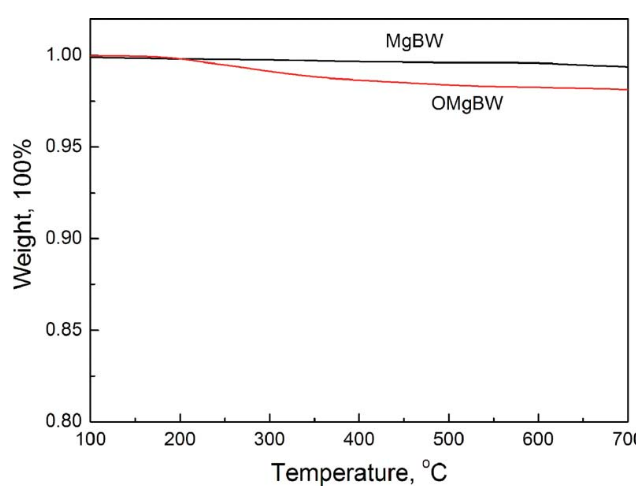


Fig. 4—TGA curves of MgBW and OMgBW. (color figure available online.)

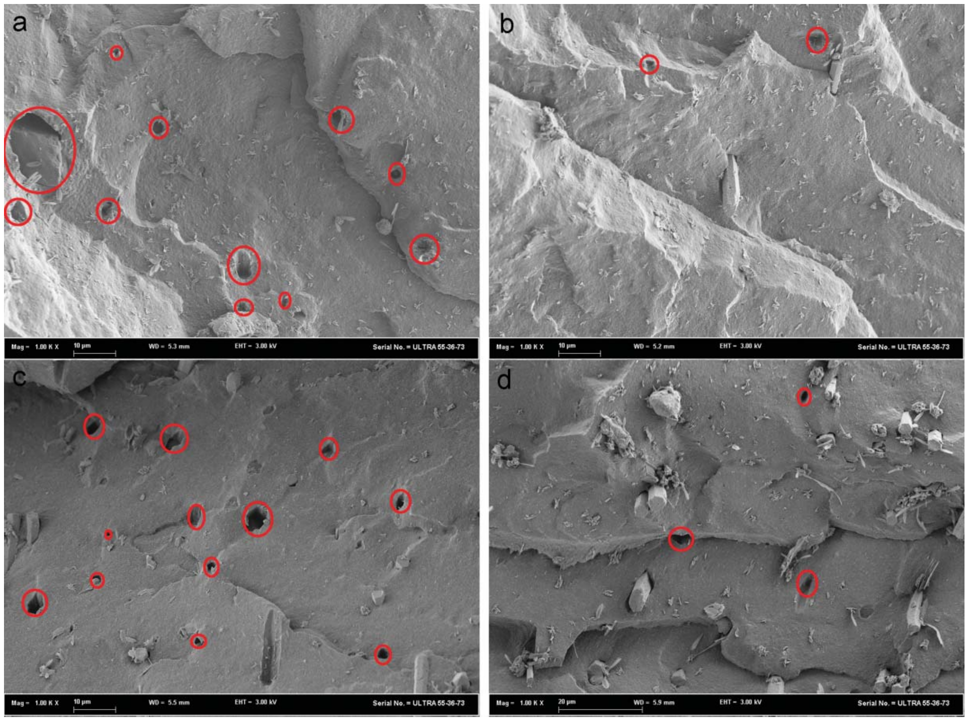


Fig. 5—SEM micrographs of NR/silica/whiskers vulcanizates: (a) NR/silica/MgBW (100/30/2.5) 1,000×; (b) NR/silica/OMgBW (100/30/2.5) 1,000×; (c) NR/silica/MgBW (100/30/7.5) 1,000×; and (d) NR/silica/OMgBW (100/30/7.5) 1,000×. (color figure available online.)

results confirmed that the surface of the MgBW was effectively modified by Si-69, which was expected to be compatible with the NR matrix.

Figure 5 shows the SEM micrographs of the tensile fractured surface of NR/silica/MgBW and NR/silica/OMgBW vulcanizates. Obviously, the fracture surface of NR/silica/MgBW vulcanizates had more caverns formed by the pulling out of whiskers than that of NR/silica/OMgBW vulcanizates, which may have resulted from the relatively weak interface combination. The micrographs illustrate that the interfacial interactions between the whisker and matrix were significantly enhanced after surface modification with Si-69.

As discussed above, the inclusion of MgBW obviously increased the antiwear property of NR and, as expected, as shown in Fig. 2, the wear loss was further improved with the surface modification. When the content of OMgBW was 7.5 phr, the specific wear rate was 0.0322 mm^3/N m, a 31.24% reduction compared to that of the vulcanizates without MgBW. The effects of MgBW and surface modification on the mechanical performance of NR vulcanizates were investigated and the results are shown in Table 1. From the table the following results were obtained: (a) As the whisker content increased, the tensile strength of the two vulcanizates increased to some extent and when whiskers content exceeded 2.5 phr, the tensile strength declined. Compared to NR/MgBW vulcanizates, the tensile strength of NR/OMgBW vulcanizates was further improved. The optimum MgBW and OMgBW contents were both 2.5 phr with tensile strengths of 26.53 and 29.39 MPa, respectively. (b) With the increased whisker content, the tear strength of the NR/MgBW vulcanizates increased gradually. Similar to tensile strength, the vulcanizates with OMgBW exhibited further increased tear strength. (c) When the whiskers were added gradually, the hardness of the vulcanizates show a slight increasing trend. (d) With the addition of whiskers, the elongation at break of the both vulcanizates increased and then decreasd. We attribute the reinforcement of MgBW on the NR matrix to the high strength and modulus of the inorganic whiskers and, of course, surface modification can further increase the mechanical performance due to the improved interfacial interactions between the whiskers and matrix.

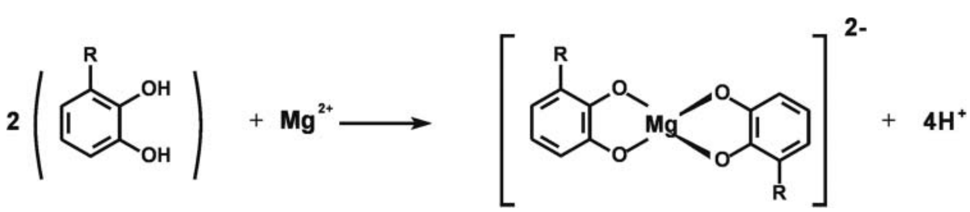


Fig. 6—Complexation reaction between TP and MgBW.

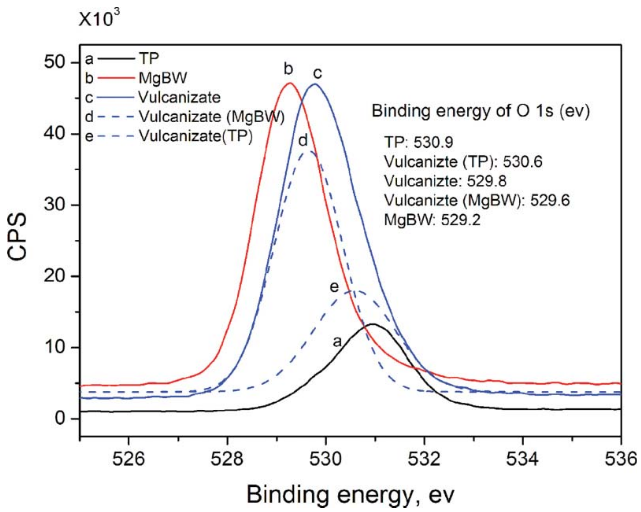


Fig. 7—High-resolution XPS spectra of O atoms in MgBW and vulcanizate. (color figure available online.)

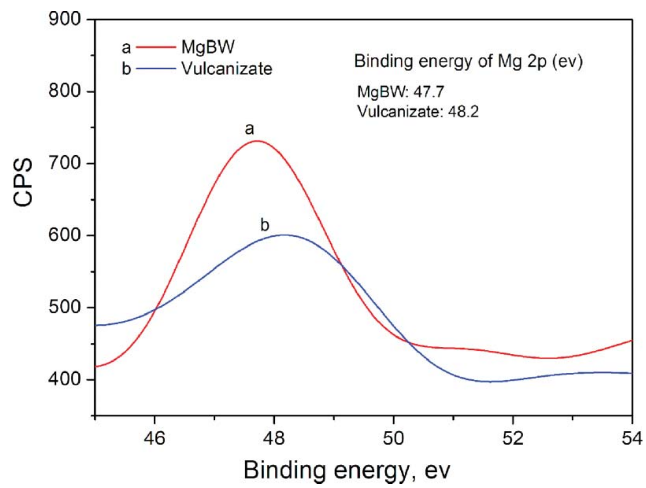


Fig. 8—High-resolution XPS spectra of Mg atoms in MgBW and vulcanizate. (color figure available online.)

Effects of TP on the Interface and Properties of NR Vulcanizates

TP is a type of condensed tannin and the main chemical composition is flavonoids. Recently, researchers have found that TP possesses many characteristics such as antioxidants (Nakagawa and Yokozawa *(22)*), reacting with protein (Freitas and Mateus *(23)*), complexing with metal ions (Richard, et al. *(24)*), etc. On the basis of the chemical structure with polyphenols and complexing with metal ions, TPs were chosen to improve the interfacial interactions between the MgBW and NR matrix to further improve the mechanical properties and especially the antiwear performance. In the present investigations, as illustrated in Fig. 6, it is proposed that hydroxyls in the TP will combine with Mg^{2+} in the MgBW to form stable multiring structures (Nathan and Julia *(25)*) and, consequently, the relatively inert surface of MgBW will become much more chemically active. In addition, the TP may complex with the metal ions in the curing additives (such as Zn^{2+}) to increase the interfacial interactions between MgBW and the rubber matrix. An XPS survey was performed to substantiate the complexation reaction between TP and Mg^{2+}. The XPS survey aims at the binding energy of the O and Mg atoms, because a complexation reaction occurs between the two types of atoms and induces a change in the bonding energy of the two types of atoms. As shown in Fig. 7, the binding energies of the O atoms in TP and MgBW were 530.9 and 529.2 eV, respectively. However, the binding energy of the O atom shifted to 529.8 eV in the vulcanizate. The spectrum of the O atom in the vulcanizate was deconvoluted using Origin 7.0 software (Origin Lab, USA) into two peaks according to the two kinds of chemical environmentsin the vulcanizate. The peaks at 530.6 and 529.6 eV were assigned to the O atoms in the TP and MgBW, respectively. Compared to the binding energy of O in TP and MgBW, it can be seen that both the binding energy of O in TP and MgBW changed evidently, indicating the complexation reaction between TP and MgBW. Similarly, as shown in Fig. 8, there was an evident shift in the binding energy of the Mg atom, which shifted from 47.7 eV in the MgBW to 48.2 eV in the vulcanizate, suggesting a second complexation reaction between TP and MgBW. In addition, a large number of hydroxyl groups in the TP also enhanced the surface activity of the MgBW. Consequently, it is believed that the complexation reaction between the TP and MgBW will enhance the interfacial action between the MgBW and NR matrix, which may be beneficial to the antiwear property and mechanical performance of NR vulcanizates.

As shown in Fig. 9, the wear resistance of the vulcanizates increased significantly with the incorporation of TP. When the TP content was 7.5, the specific wear rate was 0.0335 mm^3/N m, a 28.9% reduction compared to that of the vulcanizates without MgBW. The morphology of the worn surfaces of the

TABLE 1—MECHANICAL PROPERTIES OF NR/SILICA/WHISKERS VULCANIZATES

Samples	Tensile Strength (MPa)	Tear Strength (KN/m)	Elongation at Break (%)	Hardness (Shore A)
NR/silica (100/30)	20.19	51.67	900	69
NR/silica/MgBW (100/30/1)	22.89	52.46	905	70
NR/silica/OMgBW (100/30/1)	25.63	56.78	880	71
NR/silica/MgBW (100/30/2.5)	26.53	52.18	978	70
NR/silica/OMgBW (100/30/2.5)	29.39	59.32	922	71
NR/silica/MgBW (100/30/5)	23.48	54.75	949	71
NR/silica/OMgBW (100/30/5)	27.62	58.60	819	72
NR/silica/MgBW (100/30/10)	21.83	55.71	916	72
NR/silica/OMgBW (100/30/10)	25.16	56.41	825	72

Data in parentheses are standard deviations.

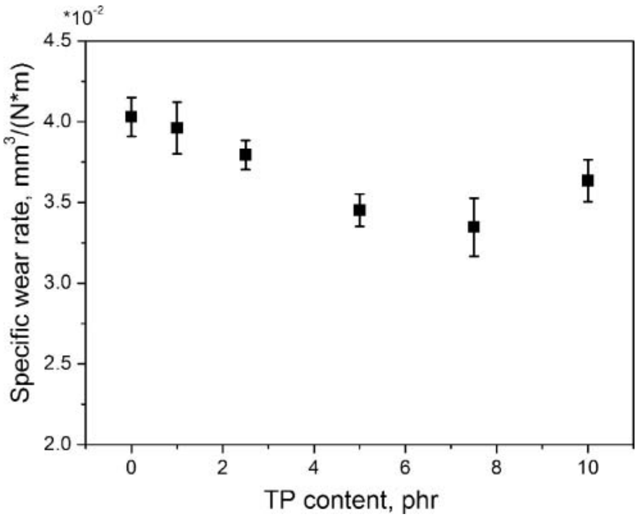


Fig. 9—Specific wear rate of NR/silica/whisker/TP vulcanizates.

NR/silica/whiskers vulcanizates is shown in Fig. 10. As shown in the figure, the worn surface of the NR/silica vulcanizate was rather rough; however, with the surface modification of MgBW by Si-69 and incorporation of TP to improve the interfacial interactions, the worn surfaces of the NR vulcanizates became smoother, indicating an improvement in the antiwear properties. The effects of TP on the mechanical properties of NR/silica/MgBW vulcanizates are summarized in Table 1. The data indicate that the inclusion of TP had an obvious reinforcement effect on the vulcanizates. The mechanical properties, especially tensile strength and tear strength, increased to some extent with the incorporation of a number of TPs. The incorporation of a number of TPs also contributed to some extent to the elongation at break. However, the hardness of the composites containing TP decreased slightly.

CONCLUSIONS

MgBWs were utilized to improve the antiwear and mechanical performance of NR. The effectiveness of MgBW and improvement of the interface by Si-69 and TP on the mechanical and antiwear performance of NR and the relationship of structure and the properties were explored. The results suggested that the wear resistance of the vulcanizates increased significantly and the mechanical performance of the vulcanizates increased to some extent with the inclusion of MgBW. It is believed that the improvement of antiwear properties resulted from the high antiwear property of the whiskers and the improved mechanical performance of the vulcanizates. The SEM observations suggested that the interfacial interactions between the whiskers and matrix were obviously enhanced after surface modification. With the incorporation of TP, the wear resistance of the vulcanizates increased significantly and the mechanical properties, especially tensile strength and tear strength, increased remarkably.

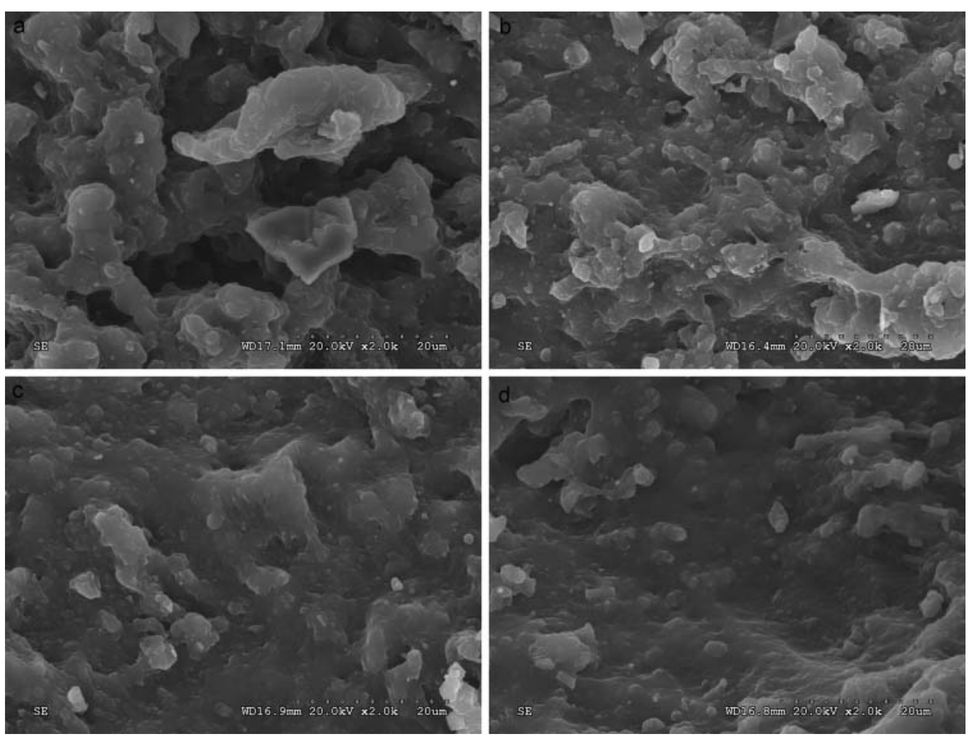


Fig. 10—SEM micrographs of the worn surface of the NR/silica/whiskers vulcanizates: (a) NR/silica/MgBW (100/30/5); (b) NR/silica/OMgBW (100/30/5); (c) NR/silica/MgBW/TP (100/30/2.5/1); and (d) NR/silica/MgBW/TP (100/30/2.5/5).

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