

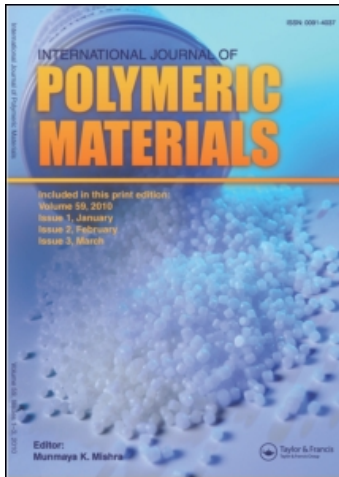
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The Magnetism Properties and Surface Microstructure of NBR/Fe₃O₄ Composites

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For preparing the good magnetism properties of rubber, NBR/Fe₃O₄ composites are prepared. In the experiment, the surface microstructure of NBR/Fe₃O₄ and distribution of nano-Fe₃O₄ particles are analyzed. The results showed that after the addition of nano-Fe₃O₄, the surface microstructure of NBR/Fe₃O₄ is greatly improved. The interface bonding of nano-Fe₃O₄ and NBR are close-knit. With adding different mass fractions of nanoparticles, the maximum elongation 300% stress at definite elongation and the tensile strength of composites are decreased, but the shore A hardness of composites is improved. The magnetic property of composites can only rely on mass fraction of nano-Fe₃O₄, while uncorrelated to the degree of scatter of nano-Fe₃O₄.

Keywords magnetic properties, nano-Fe₃O₄, NBR, physical-mechanical properties, surface microstructure

INTRODUCTION

Magnetic rubber is prepared by filling magnetic materials in rubber and vulcanization molding. Magnetic rubber has fair magnetism and maintained the advantages of rubber [1–5]. The traditional metal magnetic materials are replaced by magnetic rubber because its own characteristics, such as high saturation magnetic induction, low coercivity, high permeability, low

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high-frequency loss and so on, can be widely used in the automobile, petroleum and chemical industries. Nitrile rubber (NBR) has an excellent resistance to oil, burning oil, aromatic solvents, heat and corrosion due to the polar hydrogen in a molecule of NBR. Therefore NBR was widely used in the seal products. Rubber is filled with nanoferrite particles with low relative density and is suitable to be processed into products with high precision and complex shape [6–8]. The application scope of NBR composites was expanded due to its special properties when there is a need for reliability and performance [9–11].

In this paper, in order to further study on the properties of magnetic rubber, NBR/Fe₃O₄ was prepared by mixing nano-Fe₃O₄ particles, which have good magnetic properties, in the widely used NBR. The surface microstructure, physical-mechanical properties and magnetic properties of NBR/Fe₃O₄ were studied, and the relationship of micro-structure and the magnetic properties of composites was investigated.

EXPERIMENTS

Material Preparation

The size of nano-Fe₃O₄ particles was 50 ~ 100 nm because they could be well-distributed during compounding. The magnetic powder size was small and had a large specific surface area. The magnetic density of magnetizing products was well-proportioned and not demagnetized when nanoparticles were uniformly dispersed in rubber. NBR and all accessory ingredients were mixed in the XK-150 mixing machine at $50 \pm 5^\circ\text{C}$ for 26 min. The basic formulation was unvulcanized NBR(N41) 420 ± 0.2 g; ZnO 20 g; sulfur 6 g; accelerant DM 4 g; stearic acid 4 g; black carbon 160 g. Meanwhile, 15, 20, 25 and 30% mass fraction of nano-Fe₃O₄ particles were added by contrasting with the mass of material used. After the completion of mixing, the specimens were put on a smooth and clean metal plate for 6 h before vulcanization. Last, the specimens were vulcanized in the Y33-50A plate vulcanizing press at $145.0 \pm 0.5^\circ\text{C}$, 10 Mpa for 45 min. The curing time of NBR/Fe₃O₄ composites and standard rubber was the same while has nothing to do with the content of magnetic powder.

Performance Test

The environmental temperature was $23 \pm 2^\circ\text{C}$, and the relative humidity was 30% ~ 50%. The surface microstructure of NBR/Fe₃O₄ and distribution of nano-Fe₃O₄ particles were analyzed by using a JSM-5600LV electron microscope and energy dispersive X-ray spectrometer. The tensile strength,

maximum elongation and 300% stress at definite elongation of the specimens was measured after they were vulcanized with a DXLL-10000 electronic tensile tester. The shore A hardness was characterized using a MC010-TH200 rubber durometer. Mooney viscosity were measured by Mooney Viscometer. The magnetic property was tested by using a U5-10 magnetizer, and magnetization time was 20 min.

RESULTS AND DISCUSSION

Surface Microstructure

Because the molecular chain of NBR has strong polarity and a low degree of flexibility, which causes bad compatibility of the polymers, the surface of ordinary NBR is rough with obvious surface defects and much pits as seen in Figure 1(a). After adding nano- Fe_3O_4 particles, the surface microstructure is smoother with less surface defects and pits in Figure 1(b). This is because the nano- Fe_3O_4 particles have good chemical and thermal stability and a large specific surface area [9,13]. Thus, we can know that nano- Fe_3O_4 particles have a high-energy surface, while NBR has a low-energy surface. When mixing the two materials, the low-energy surface of NBR is inclined to be strongly adsorbed on the high-energy surface of nano- Fe_3O_4 particles. This kind of adsorption can compensate the imbalance force field on the high-energy surface [8,9,11]. At the same time, nano- Fe_3O_4 particles can fill surface defects of NBR, which represents that nano- Fe_3O_4 particles can better reinforce the NBR. And the two-phase interface of NBR and nano- Fe_3O_4 are fuzzy in the NBR matrix, which indicates nano- Fe_3O_4 particles and NBR matrix had good affinity in Figure 1. The polymer mixed process is completed in a sticky flow condition which can be seen as nano- Fe_3O_4 dissolves in rubber. Through comparison, we can know that the surface structure of NBR/ Fe_3O_4 composites is better when the mass fraction of nano- Fe_3O_4 is 15%. However, with increasing the level of filling, the surface properties gradually reduced and there was a more granular convex body which means greater roughness in Figures 1(c)–(e). This is because the integration degree of NBR and nano- Fe_3O_4 particles

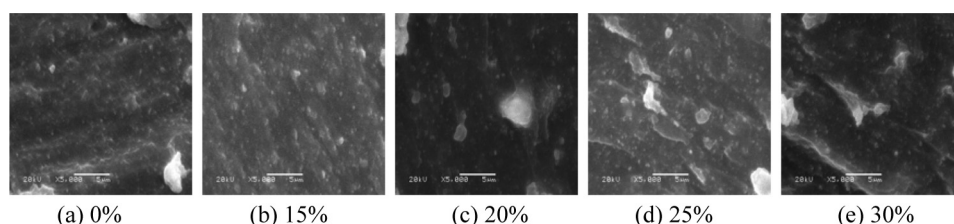


Figure 1: SEM pictures of NBR with a different mass fraction of nano- Fe_3O_4 (5000 \times).

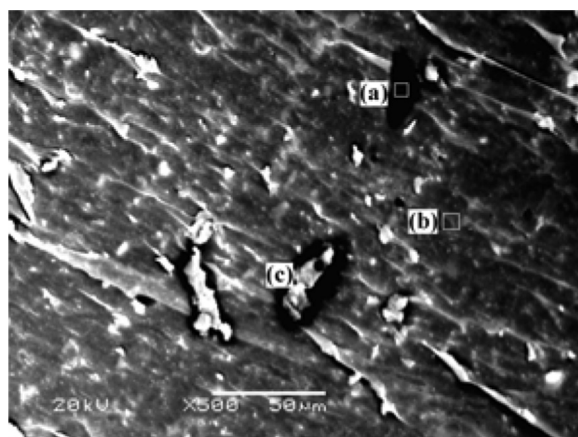


Figure 2: Microscopic surface backscattered image (mass fraction of nano-Fe₃O₄ particles 25%).

decreased, and the irregular phase structure increased due to the bond strength variation of the two-phase interface, which led to the composites strength reduction when the addition is over 20%.

In order to accurately study the distribution of nano-Fe₃O₄ particles, components of the surface elements were analyzed. Microscopic surface backscattered image of the NBR/Fe₃O₄ composites is shown in Figure 2. As in Figure 2, we selected areas a, b and c to analyze the distribution of nano-Fe₃O₄. The results are shown in Table 1.

We can see from Table 1 that the percentage composition, atom percentage composition of Fe, and counting rate of x-ray are basically in line with the addition of nano-Fe₃O₄. There are minute differences between different regions owing to the inhomogenous mixing. Surface microstructure of area c has more defects due to more nano-Fe₃O₄. However, in terms of overall effect, the mixing is relatively uniform.

Physical-Mechanical Properties

The change curve of 300% stress at definite elongation, tensile strength and maximum elongation of the NBR/Fe₃O₄ composites filled with different mass fraction of nano-Fe₃O₄ in Figure 3.

Table 1: The distribution of Fe elements of composites.

Surveyed area	Line	Weight %	Cnts/s	Atomic %
a area	Ka	22.03	42.83	12.27
b area	Ka	23.17	45.27	13.17
c area	Ka	27.32	53.88	17.91

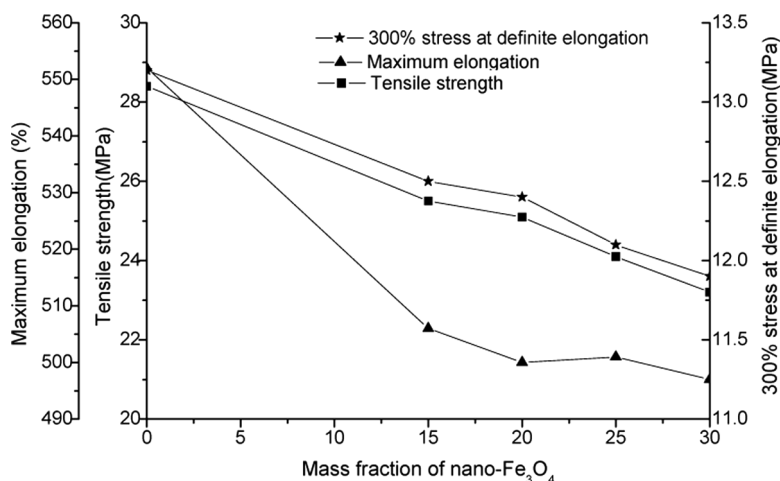


Figure 3: Change curve of 300% stress at definite elongation, tensile strength and maximum elongation of the NBR/ Fe_3O_4 with a different mass fraction of nano- Fe_3O_4 .

It can be seen from Figure 3 that 300% stress at definite elongation, tensile strength and maximum elongation of NBR/ Fe_3O_4 composites are slightly lower than ordinary NBR. In order to obtain better strength for polymer composites, a key factor is the uniformity dispersion of nano-particles; the other is the interface structure of rigid particles surface in the matrix [3,5,9]. With the addition of nano- Fe_3O_4 particles increasing for composites, interfacial thickness is decreased due to the spacing of particles being reduced between the particles. The wrapped degree of nano-particles is weakened in the NBR matrix, and bond strength of two-phase interface is broken up, imbalance force is concentrated to cause strength of composites is decreased. With addition of nano- Fe_3O_4 increasing, the homogeneous degree of nano-particles gradually declined and agglomeration increased. Binding force is decreased due to a large area of phase interface existing between agglomeration of nano- Fe_3O_4 particles and the rubber matrix in Figure 1.

The change curve of shore A hardness of NBR/ Fe_3O_4 composites filled with different mass fraction of nano- Fe_3O_4 in Figure 4. It can be observed that shore A hardness of NBR/ Fe_3O_4 composites is larger than ordinary NBR. Shore A hardness of unfilled NBR was 63 degrees, while the shore A hardness of NBR/ Fe_3O_4 composites was 74 degrees when the mass fraction of nano- Fe_3O_4 was 30%. The reason for the increase of hardness, on one hand, was that the hardness of nano- Fe_3O_4 was bigger than that of unfilled NBR, which deservedly increased the hardness of the composites [4,12,14], nano- Fe_3O_4 particles filled the cavity of NBR, which increased the hardness of NBR/ Fe_3O_4 composites as well; on the other hand, the good interfacial bonding and a certain thickness of the interfacial layer exist around evenly distributed rigid particles

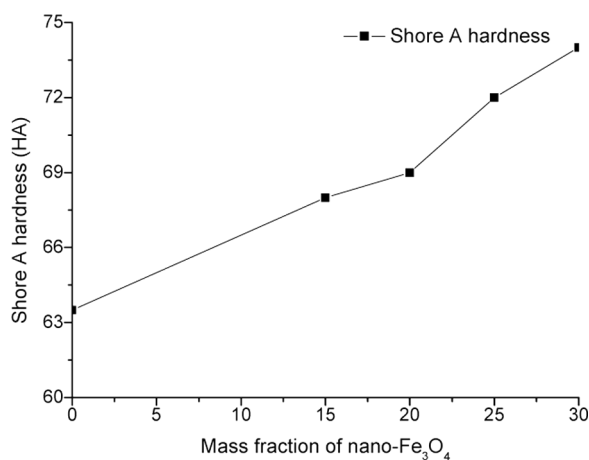


Figure 4: Change curve of shore A hardness of NBR/Fe₃O₄ with a different mass fraction of nano-Fe₃O₄.

which causes craze when damage are produced [1,15], which causes a large amount of impact to be consumed and suffer stress is well-transmitted.

The change curve of Mooney viscosity of NBR/Fe₃O₄ composites filled with different mass fraction of nano-Fe₃O₄ is in Figure 5. The Mooney viscosity of NBR was slightly increased after adding nano-Fe₃O₄ particles, but the processing was affected little. From the above analysis we can know that the low mass fraction of nano-Fe₃O₄ and the change of physical-mechanical properties was small.

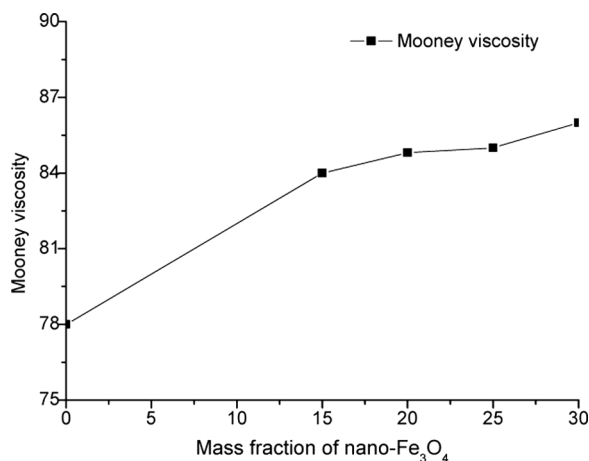


Figure 5: Change curve of Mooney viscosity of NBR/Fe₃O₄ with a different mass fraction of nano-Fe₃O₄.

Magnetic Properties

The magnetic hysteresis loops of NBR/Fe₃O₄ composites are shown in Figure 6. Clearly, by increasing the mass fraction of nano-Fe₃O₄, magnetic properties of the NBR/Fe₃O₄ composites monotonously increase, and the saturation magnetization strength of the composites increases constantly. The composites show typical characteristics of paramagnetism. Due to internal defects in rubber materials, wall energy is reduced and the effect of magnetization is influenced, so the magnetic density needed during the magnetization process is relatively larger [8,9]. With the magnetic intensity increasing, saturation magnetization strength of the NBR/Fe₃O₄ composites increases due to the microstructure of composites weakened with the addition of nano-Fe₃O₄ increasing. When the magnetic strength increased to 10,000 (Oe), the value of saturation magnetization strength tends basically to steady.

The changing curve of saturation magnetization strength (B_s), residual magnetic flux density (B_r), and coercive force (H_c) with different mass fraction of nano-Fe₃O₄ particles are shown in Figure 7. As seen in Figure 7, H_c of the NBR/Fe₃O₄ composites is small. When the mass fraction of nano-Fe₃O₄ is 15%, the value of H_c is 14 emu/g which is relatively smaller. B_s and B_r keep good linear relationship with the mass fraction of nano-Fe₃O₄. By increasing the mass fraction of nano-Fe₃O₄, B_s and B_r continuously become larger. This is because magnetic properties of the NBR/Fe₃O₄ composites do not depend on the way nano-Fe₃O₄ particles scatter, and also do not depend on physical processes between particles and substrate or particles and particles [4,8,12], but only depend on the mass fraction of nano-Fe₃O₄. Meanwhile, nano-Fe₃O₄

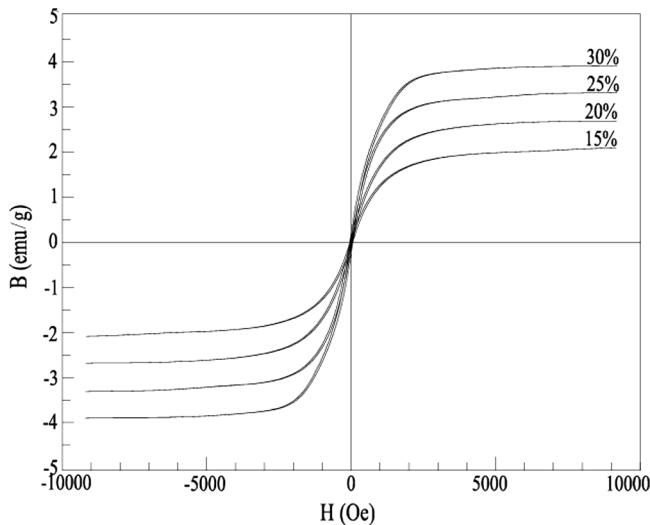


Figure 6: Magnetic hysteresis loops of NBR/Fe₃O₄ composites.

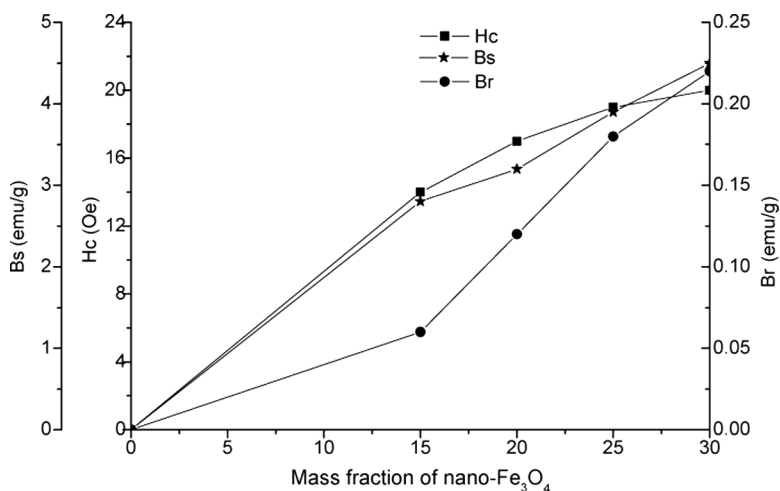


Figure 7: Changing curve of saturation magnetization strength (B_s), residual magnetic flux density (Br) and coercive force (H_c) with different mass fraction of nano-Fe₃O₄.

particles are ferrimagnetics which have typical characteristic of high frequency, therefore hysteresis losses are lesser. Compared with [8], we can see that NBR/Fe₃O₄ has good soft magnetism performance. These all lay a foundation for further application of NBR/Fe₃O₄ composites.

CONCLUSIONS

1) The NBR/Fe₃O₄ was prepared by blending. Surface microstructure of NBR/Fe₃O₄ was improved due to the addition of nano-Fe₃O₄ and was smoother when the addition of nano-Fe₃O₄ was 15%.

2) Nano-Fe₃O₄ particles were well-inoculated in the NBR matrix, and the distribution of nano-Fe₃O₄ was well-proportioned. With increasing the mass fraction of nano-Fe₃O₄ 300% stress at definite elongation, tensile strength and maximum elongation of the NBR were decreased while shore A hardness was increased.

3) With the addition of nano-Fe₃O₄ increasing, the microstructure of composites has more pits which lead to saturation magnetization strength increasing. The magnetic properties of composites can only rely on the mass fraction of nano-Fe₃O₄ particles, while uncorrelated to the degree of scatter of nano-Fe₃O₄ particles. The NBR/Fe₃O₄ shows good soft magnetism performance.

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