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Loading dependence similarities on the cure time and mechanical properties of rubber ferrite composites containing nickel zinc ferrite

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Abstract

Composite magnetic materials have the unique advantage of property modification for tailoring devices for various applications. Rubber ferrite composites (RFCs) prepared by incorporating ferrites in rubber matrixes have the advantage of easy mouldability and flexibility. RFCs containing various loadings of nickel zinc ferrite (NZF) ($Ni_{1-x}Zn_xFe_2O_4$) in a natural rubber matrix have been prepared. The cure characteristics and the mechanical properties of these composites were evaluated. The effect of loading on the cure characteristics and tensile properties were also evaluated. It is found that the loading dependence on the cure time and mechanical properties exhibit an identical pattern.

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Keywords: Rubber ferrite composites (RFCs); Nickel zinc ferrites; Cure time

1. Introduction

Rubber ferrite composites are composite materials with ferrite fillers as one of the constituents and natural or synthetic rubber as the base matrix. In these magnetic polymers, the magnetic powder as well as the polymer will affect the processability and other physical properties of the final product. In tailoring composites for various applications, it is necessary to select the proper filler and a matrix. The physical and chemical properties of the magnetic composites will possibly be influenced by the interactions with the filler and the matrix.

Incorporation of polycrystalline ferrites in elastomer matrixes will lead to rubber ferrite composites (RFC) [1–5]. An appropriate selection of magnetic filler and matrix can result in RFCs with required properties for different applications. Rubber ferrite composites (RFCs) can be synthesized by the incorporation of ferrite powders in natural or synthetic rubber as matrixes. This can not only bring in economy but also can produce flexible magnets. Factors like percolation limit or nature of the matrix, like saturated/unsaturated/polar rubber all influences the final properties of the composites. A systematic study of these composites may be of help in tailoring materials for various applications.

The evaluation of cure characteristics throws light on the processability of these composites. The mechanical properties are one of the most important properties of plastic materials because all applications

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involve some degree of mechanical loading. A tensile test is a measurement of the ability of a material to withstand the forces that tend to pull it apart and to determine to what extent the material stretches before breaking. Different types of plastic materials are often compared on the basis of their tensile strength, elongation and modulus. Hence the evaluation of these properties also assumes significance in making devices based on RFCs [6–10]. In this article, the effect of magnetic filler loading on the cure parameter and the impact on the mechanical properties of the composite is reported. The similarities observed on the loading dependence on cure time as well as on mechanical properties are also highlighted in this communication.

2. Experimental

2.1. Preparation of ceramic nickel zinc ferrite (NZF)

Nickel zinc ferrite (NZF) $Ni_{1-x}Zn_xFe_2O_4$, for various x, ranging from 0 to 1 in steps of 0.2 was prepared by using conventional ceramic techniques [11–13]. Ferrous oxalate dihydrate, nickel oxide, zinc oxide, etc., were used as precursors. These precursors taken in appropriate ratio were mixed thoroughly in an agate mortar to produce a homogeneous mixture of fine particles. Repeated sintering at 500 °C and mixing of this powder were continued till single phasic spinel NZF was obtained. This pre sintered powder was then finally sintered at 1000 ± 15 °C for several hours.

2.2. Incorporation of ferrites in natural rubber

Pre characterized NZF was then incorporated in a natural rubber (ISNR-5 supplied by Rubber Research Institute of India, Kottayam) matrix by adding appropriate amounts of ZnO, Stearic acid, Tetra Methyl Thiuram Disulphide (TMTD), Mercapto Benzo Thiazole Sulphide (MBTS) and Sulpher [3,6]. The mixing was done in a Brabender Plasticorder (Torque Rheometer) model PL 3S. Rubber ferrite composites were prepared for loadings of 30 to120 parts per hundred grams of rubber by weight (phr) in steps of 30 phr. Further, it was moulded into thin sheets of about 1 mm thickness at 150 °C at their respective cure time in accordance with ASTM D 3188 using a hydraulic press.

2.3. Cure characteristics

The cure parameters of RFC for various loadings of NiZn Ferrite was studied by using Gottfert Elastograph model 67.85 at 150 °C. A test specimen of the vulcanisable rubber compound is inserted into the curometer test cavity and closed under positive pressure [14]. The cavity is maintained at 150 °C. The rubber totally surrounds the biconnical disc after the dies are closed. The disc is oscillated 3° amplitude and this action exerts a shear strain on the test specimen. The force required to oscillate the disc to the maximum amplitude is continuously recorded as a function of time, with the force being proportional to the shear modulus or stiffness of the test specimen at the test temperature. This stiffness first decreases as it warms up and it increases as a result of vulcanisation. The test is completed when recorded torque either reaches a maximum when predetermined time is elapsed. The time required to cure the test specimen is a function of cure characteristics of the rubber compound and at the test temperature. Thus from the cure curve obtained the cure parameters such as minimum torque, maximum torque, and cure time are evaluated. The compounds are then vulcanized at 150 °C on an electrically heated laboratory hydraulic press up to the respective cure time to make sheets of the sample. The entire test is carried out according to the ASTM standards.

2.4. Evaluation of mechanical properties

Tensile strength, modulus and elongation are some of the most important indications of the strength of the material. Mechanical properties of the representative samples of the RFCs were determined using the universal testing machine (UTM) model Instron 4500. Dumb-bell shaped samples were cut from the prepared RFCs containing $Ni_{1-x}Zn_xFe_2O_4$ (x = 0.2,0.6 and 1.0) at various loadings of 30, 60, 90 and 120 phr. Parameters namely tensile strength, 100% modulus and elongation at break were determined for the samples containing NZF at different loadings.

3. Results and discussion

3.1. Cure characteristics

The vulcanization characteristics of the prepared rubber compounds were determined by using the Gottfert elastograph. Cure characteristics were plotted for all composites and from the cure characteristics, parameters, namely, maximum torque minimum torque, cure time, etc., were evaluated. A representative cure characteristic curve is shown in Fig. 1. Cure rate curve shows the rate of cure as a function of time. Initially as time increases cure rate also increases and reaches a maximum and then it decreases to zero, when the curing of the compound is completed.

Graphs showing the variation of maximum torque and minimum torque with filler loading are also plotted and are shown in Figs. 2 and 3. It is seen that the maximum torque increases with loading for almost all compositions. Maximum torque gives an idea about the shear modulus of the fully vulcanized compound at the vulcanization temperature [6,8]. It is evident from the graph (Fig. 2) that the maximum torque increases with loading for all compositions of zinc in the NZF series.

Minimum torque is an indirect measure of the viscosity of the compound [6,8]. Or it can be generally treated as the measure of the stiffness of the unvulcanised test specimen taken at the lowest point of the curve. Minimum torque also shows an increasing trend for lower loading and saturates at higher loadings. The variation pattern is by and large the same for $x=0.8$ and 0.2. It may also be noted that the minimum torque values for $x=0.0$, 0.2 and 1.0 coincides.

Cure time is defined as the time required for optimum vulcanization of the samples. This is an important parameter as far as the vulcanization is concerned.

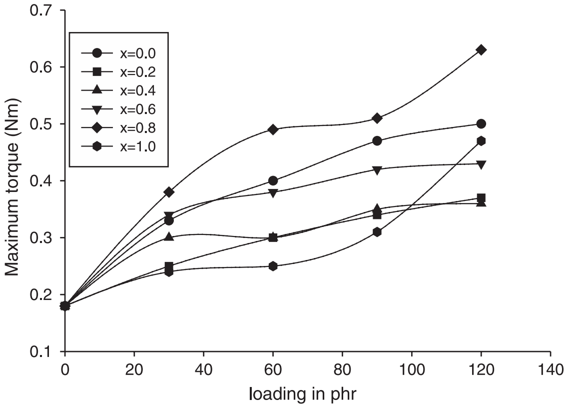

Fig. 2. Variation of maximum torque with loading $Ni_{1-x}Zn_xFe_2O_4$ in NR.

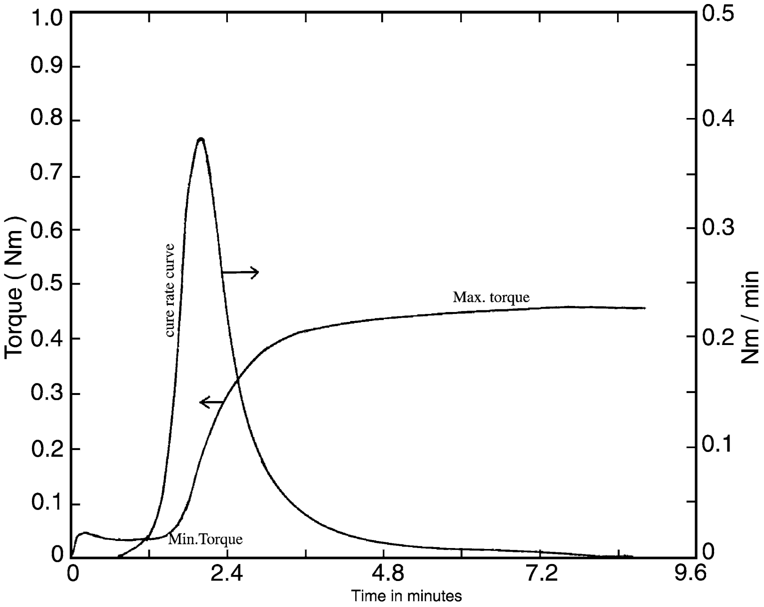

Fig. 1. Representative cure characteristic curve for RFC.

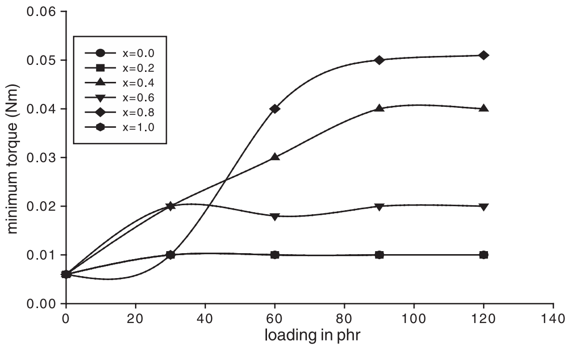

Fig. 3. Variation of minimum torque with loading $Ni_{1-x}Zn_xFe_2O_4$ in NR.

Evaluation of cure time is a prerequisite for moulding the compounds. The variations in cure time for different loadings were studied for RFCs containing NZF in natural rubber matrix. The variations are shown in Fig. 4.

From Fig. 4 it is seen that cure time sharply decreases for initial filler loadings (30 phr) and the change in cure time is only marginal for additional loadings of the filler. The above observations confirm that the filler addition does not affect the processability of the composites considerably.

3.2. Mechanical properties

The mechanical properties, namely the tensile strength, elongation at break and 100% modulus were determined and their variation with loading was also studied. The variation of tensile strength with loading is shown in Fig. 5. It shows that the tensile strength first decreases with initial loading up to 30 phr and thereafter it increases with loading. It is known that stress-induced crystallization will increase the tensile strength and hence blank NR has got a higher tensile strength [6–10]. But the addition of filler will inhibit the stress-induced crystallization, which in turn will result in a decreased tensile strength for the initial filler loading of 30 phr [6–10,15,16]. For further loadings of the filler, the tensile strength shows an increasing trend. This means that the filler NZF is acting as a semi-reinforcing filler [16].

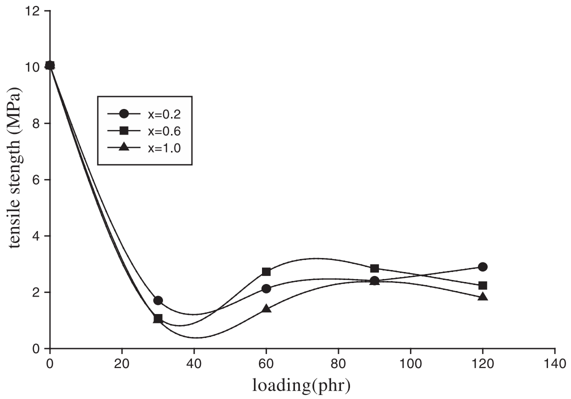

Fig. 5. Variation of tensile strength with loading $Ni_{1-x}Zn_xFe_2O_4$ in NR.

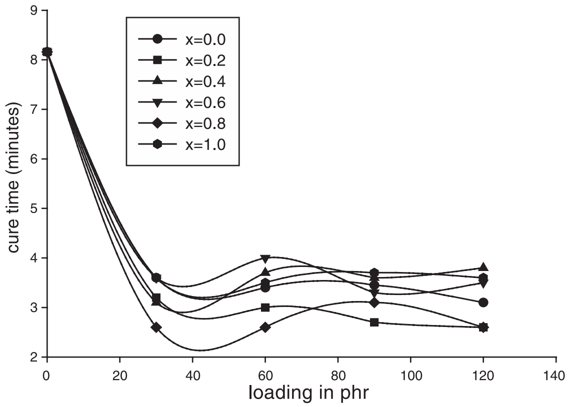

Fig. 4. Variation of cure time with loading $Ni_{1-x}Zn_xFe_2O_4$ in NR.

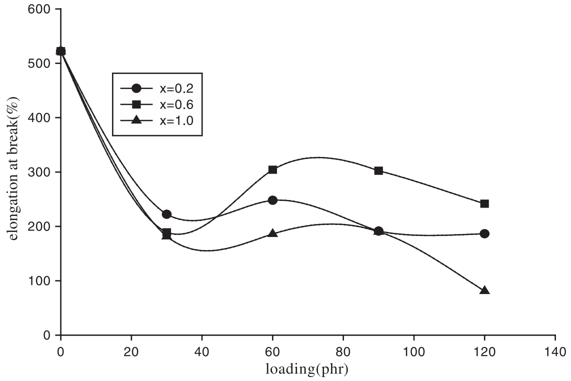

Fig. 6. Variation of elongation at break with loading $Ni_{1-x}Zn_xFe_2O_4$ in NR.

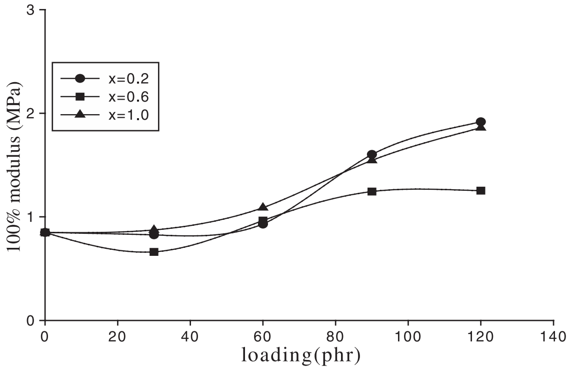

Fig. 7. Variation of 100% modulus with loading $Ni_{1-x}Zn_xFe_2O_4$ in NR.

Here it may be noted that the variation pattern for tensile strength with loading and cure time vs. loading (refer to Fig. 4) almost resembles. It has been studied and observed by many researchers that cross-linking between polymer molecules reduces their mobility and thus increases the modulus and strength [8–10]. Cross-linking generally affects the properties such as the tensile strength and elongation, which are determined by large molecular deformations. But greater interaction between other effects makes the ultimate property unpredictable. So as the cure time increases, cross-linking also increases, which in turn increases the tensile properties. Thus the cure time variation with loading indirectly gives an idea about the tensile strength variation with loading.

Elongation at break also shows a similar decrease for the initial loading and it remains by and large the same for higher loadings (Fig. 6). This can also be explained as due to the decrease in stress-induced crystallization [15,16]. During elongation rearrangement or orientation occur to form crystallites, and this stress-induced crystallization gives natural rubber (NR) high strength in the blank form. But the addition of filler will inhibit this rearrangement or orientation, thereby reducing the elongation at break as well as tensile strength. One hundred percent modulus shows an increasing trend with increase in loading (Fig. 7). This is evident from the cure characteristics too. It may also be noted that the filler addition will decrease the polymer molecular weight slightly and this will also cause a small decrease in the tensile as well as the modulus values.

4. Conclusion

Rubber ferrite composites (RFCs) containing $Ni_{1-x}Zn_xFe_2O_4$ in natural rubber matrix have been prepared. The cure characteristics reveal that the processability and flexibility of the matrix is not much affected even up to a maximum loading of 120 phr of the filler in this set of experiments. The mechanical properties of representative samples were studied and it was found that the filler modifies the mechanical properties. It is also found that the cure time is also a determining factor as far as the tensile properties are concerned. It is also observed and already published [3,14] that the compositions corresponding to $x = 0.4$ and 0.6 gives optimum strength and magnetisation and minimum hardness. The modification of these properties will aid in the design of composite materials for possible applications. The dependence of loading on the cure time exactly resembles the variation of tensile strength and elongation with loading for the NZF incorporated RFC.

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