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Material Properties

Epoxidized natural rubber for vibro-acoustic isolation

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ABSTRACT

The performance of an elastomeric vibro-acoustic isolation system is governed by the dynamic properties of the material under service temperature and frequency conditions. This paper investigates the effect of epoxidation level on the dynamic properties of Epoxidized Natural Rubber (ENR). Dynamic properties of natural rubber with 0, 25 and 50 mol% epoxidation levels were measured in simple shear over a range of temperatures and frequencies and a master curve for their dynamic properties was generated based on the time-temperature superposition principle. These master curves and the Cole-Cole plot of their dynamic properties shows that the epoxidation level does not affect the relationship between the storage modulus and loss modulus, but merely shifts the master curves for natural rubber along the frequency axis. These observations would be of value for designing a vibration isolation system with an optimum level of epoxidation to achieve the desired storage modulus (or loss modulus) at the given service conditions. A reduced temperature nomogram is presented for the prediction of appropriate epoxidation level required for such design purposes. The 5-parameter fractional derivative model by Pritz [J. Sound & Vib., 265, 935–952 (2003)] was fitted to the master curves for the complex dynamic modulus of natural rubber with different epoxidation levels.

1. Introduction

Viscoelastic materials such as rubber are used extensively for vibro-acoustic isolation. The dynamic mechanical properties of these materials are dependent on the temperature and frequency. Therefore, it is necessary to test rubber compounds used in vibration and noise control applications in a range of environmental conditions and frequencies.

A chemically modified natural rubber (NR); Epoxidized Natural Rubber (ENR), is the focus of this study. ENR is produced by epoxidation of natural rubber with peracetic acid at the latex stage. A known percentage of its double bonds are reacted to form epoxide groups. As the epoxide groups are introduced, ENR shows a higher glass transition temperature (T_g) as compared to natural rubber by approximately 1 °C for every mol% epoxidation [1]. This will strongly affect the dynamic mechanical properties of ENR [2].

ENR provides good resistance to oils and non-polar solvents, high damping at room temperature and low air permeability. These properties result from the high polarity of epoxide groups, which are randomly distributed along the natural rubber backbone [3,4].

There are two commercial grades available for ENR which have different epoxidation levels: 25 mol% and 50 mol% [2], referred as ENR-25 and ENR-50. Rubber with any level of epoxidation can be

developed under the right reaction conditions, which are mainly controlled by the peracetic acid concentration and epoxidation temperature [5].

A number of authors have reported on the dynamic mechanical properties of ENR. The materials investigated so far include blends of ENR and polymers with different T_g s such as polyvinyl chloride [6], ethylene vinyl acetate [7], natural rubber [8], polychloroprene [9] and ENR blends with ethylene propylene diene monomer rubber [10]. Materials consisting of ternary blends have also been reported such as ENR/polyvinyl chloride/chlorinated polyethylene [11] and ENR/natural rubber/cellulose [12]. Several studies on the performance of dynamic mechanical properties of ENR blends using different mixing approaches have been conducted. For example, Rooj et al. [13] developed a mixing technology based on an in-line electron induced reactive processing technique. Wang et al. [14] introduced wet masterbatch technique instead of using the traditional dry mixing method.

There is a relatively small amount of published work on dynamic mechanical properties of materials based on ENR alone as the main matrix. Furthermore, the effect of the epoxidation level on the dynamic properties of ENR has not been fully investigated. One particular study on the dynamic properties of ENR alone has been reported by Lu and Li [15]. They implemented an alternative approach in producing ENR

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with effective damping in a broad temperature range. It was shown that the application of a different crosslinking agent, phenolic resin, in the ENR matrix helps to achieve an effective damping with the loss factor of $\tan \delta > 0.3$ and in the temperature range from $-48\text{ }^{\circ}\text{C}$ to $100\text{ }^{\circ}\text{C}$. However, the research they conducted focused only on a single epoxidation level of 40 mol%. The earlier work reported by Ahmadi et al. [16] has particularly addressed the effect of different epoxidation levels on the dynamic mechanical properties of ENR matrix. They have included a more interesting finding when they generated master shear modulus and loss factor curves of 0, 25 and 50 mol% epoxidation levels. The master curves obtained were found to be similar in shape.

In this study, the effect of different epoxidation levels on the dynamic properties of ENR as a single matrix was investigated for a wide range of temperatures and frequencies. Three types of natural rubber with different epoxidation levels of 0, 25 and 50 mol% were studied. The master curves of the rubbers were compared and nomogram relating the reduced frequency to the complex moduli at different temperatures was obtained. The influence of the epoxidation level on the relationship between the two dynamic moduli of the material was also studied using a Cole-Cole plot. Finally, the dynamic properties obtained were fitted by fitting the 5-parameter fractional derivative model by Pritz (Eq. (38) in Ref. [17]) to the master curves for the complex dynamic modulus.

2. Experimental procedures

2.1. Materials and sample preparation

Three types of natural rubber, SMR-CV60, ENR-25 and ENR-50 were obtained from the Malaysian Rubber Board. The basic details of the materials are shown in Table 1. All other ingredients were provided by Tun Abdul Razak Research Centre (TARRC), United Kingdom. All of the materials were commercial grade. The formulations used are given in Table 2. The formulations are denoted by R0, R25 and R50, where the numbers stand for the epoxidation level of the rubber.

The materials were compounded on a laboratory 2-roll-mill machine maintained below $40\text{ }^{\circ}\text{C}$. The rubber was loaded at the beginning of the compounding process. The other ingredients were mixed together and then loaded gradually. Finally, a smooth and uniform sheet was obtained. The compounding time was kept below 20 min. The compounded natural rubber was left for 24 h before being compression moulded into the rubber disc shaped of the double bonded shear test pieces for 20 min using an electrically heated hydraulic press at $150\text{ }^{\circ}\text{C}$. The curing time was selected according to the crosslinking time at which 95% of cure has taken place observed in the rheometer curve. The curve was analysed using a Monsanto Rheometer, MDR 2000.

The rubber pieces were bonded to stainless-steel surfaces during the curing process. The first step for bonding was to clean the stainless steel by dry sandblasting (air-blasting). Then, the stainless steel was immersed in acetone to remove the loose particles for 5 min. Finally, a

Table 1
Rubber materials used in the experiments.

Material	Type	Characteristic	
		Epoxidation Level ^b (%)	Glass Transition Temperature, T_g ($^{\circ}\text{C}$)
Natural rubber	SMR-CV60 ^a	0	-72.0
Epoxidized natural rubber	ENR-25	29.4	-45.5
	ENR-50	52.1	-25.1

^a Standard Malaysian Rubber constant viscosity: grade 60.

^b As analysed by Proton Nuclear Magnetic Resonance ($^1\text{H NMR}$).

^c Estimated based on the epoxide levels analysed by $^1\text{H NMR}$ from relationship described by Davey and Loadman [1].

Table 2
Formulations in parts per hundred of rubber (phr).

Ingredient	R0	R25	R50
SMR-CV60	100	–	–
ENR-25	–	100	–
ENR-50	–	–	100
Sulfur	2.5	2.5	2.5
Zinc Oxide	4	4	4
Stearic Acid	4	4	4
Wingstay L	1	1	1
CBS	1	1	1
PVI	0.3	0.3	0.3

rubber-metal bonding system, Chemosil[®] 211 (primer) and Chemosil[®] 225 (covercoat) were applied to the stainless-steel surfaces which was to be bonded to the rubber pieces during the curing process. Drying time of 30 min at room temperature for primer bonding agent was allowed before applying the covercoat bonding agent.

2.2. Dynamic mechanical characterisation

The dynamic properties of the three vulcanizates were measured using a Metravib DMA + 1000 dynamic mechanical analyser. Tests were carried out over a frequency range of 0.1–170 Hz and a temperature range of -40 to $50\text{ }^{\circ}\text{C}$ with a dynamic strain of 0.1%. Double bonded shear test pieces (Fig. 1) were used. The nominal dimensions of the rubber discs were 10 mm in diameter and 2 mm thickness.

3. Results and discussion

3.1. Effect of temperature on the dynamic mechanical properties

Fig. 2 illustrates the dependence of the storage modulus (G') as a function of temperature in the range of $-40\text{ }^{\circ}\text{C}$ to $50\text{ }^{\circ}\text{C}$ for natural rubber with different epoxidation levels. These data were taken at 170 Hz excitation. This choice of frequency is the best to capture the development of the loss factor peak for the R0, R25 and R50 rubbers studied in this work.

It can be seen that all the materials exhibit high G' in the low temperature region. G' decreases with the increasing temperature. In this region the mobility of the polymer chains was increasing [12], so that a reduction in the material stiffness was expected. The storage modulus eventually converges for all three materials at around $50\text{ }^{\circ}\text{C}$.

Fig. 3 shows the loss factor ($\tan \delta$) curves under the same conditions. This parameter gives a measure of the energy loss in the material due to the molecular rearrangement and internal friction in the natural rubber matrix. Above $5\text{ }^{\circ}\text{C}$, $\tan \delta$, and therefore damping, increases with the increasing epoxidation level. Fig. 3 also indicates that T_g increases as the epoxidation level is increased, suggesting that the epoxide groups result in restricting movement the natural rubber chains. As a result, a higher temperature is needed to overcome the chain rigidity and interactions and allow rubbery behaviour.

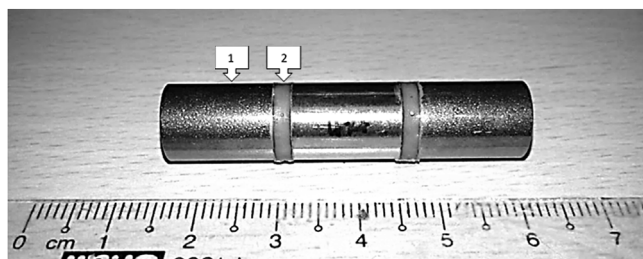


Fig. 1. A double bonded shear test piece. 1: stainless steel; 2: rubber.

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