Composites: Part B 54 (2013) 377–382

Contents lists available at SciVerse ScienceDirect

Composites: Part B

journal homepage: www.elsevier.com/locate/compositesb

Effects of electron-beam and sulfur crosslinking of epoxidized natural rubber on the friction performance of semimetallic friction materials

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ARTICLE INFO

Article history:
Received 9 July 2012
Received in revised form 25 March 2013
Accepted 26 May 2013
Available online 7 June 2013

Keywords:
B. Wear
B. Surface properties
B. Porosity
E. Cure
Friction coefficient

ABSTRACT

Semimetallic friction composites (SMFCs) consisting of epoxidized natural rubber (50 mol% epoxidation, ENR 50), alumina nanoparticles, steel wool, graphite, and benzoxazine were prepared via melt mixing using a Haake internal mixer at 90 °C and 60 rpm rotor speed. The composites were vulcanized using sulfur and electron-beam (EB) crosslinking systems. The SMFC samples were then subjected to friction, hardness, porosity, and density tests to determine their friction and wear properties. The morphological changes in the samples were also observed under a scanning electron microscope. The friction and wear properties of SMFCs crosslinked via the EB irradiation and sulfur vulcanization systems were compared. The friction coefficients in normal and hot conditions, as well as the hardness and density of the irradiated SMFC, were higher than those of the sulfur-vulcanized samples at all applied doses. The porosity of the irradiated SMFC at 50, 100, and 150 kGy was higher than that of the sulfur-vulcanized samples; however, the irradiated SMFC exhibited a descending trend at 200 kGy. On the other hand, the specific wear rates of the irradiated samples were lower than those of the sulfur-vulcanized samples at all applied doses. The sample crosslinked via EB irradiation at 150 kGy exhibited the greater tribological property compared with the sulfur-vulcanized SMFC, as indicated by the higher friction coefficient (approximately 0.461) and lower wear rate achieved at 150 kGy irradiation.

1. Introduction

Brake friction materials are vital to the braking system because these convert the kinetic energy of a moving vehicle to thermal energy via the generated friction during the braking process [1]. A friction material is a heterogeneous material composed of a few elements. Each element has its own function, including improving the friction property at low and high temperatures, increasing strength and rigidity, prolonging life, reducing porosity, and reducing noise. Changes in the element types or weight percentages of the elements in the formulation may change the physical, mechanical, and chemical properties of the brake friction materials to be developed [2–5]. The four main components of a brake pad are the reinforcing fibers, binders, fillers, and frictional additives.

Fillers play an important role in modifying the characteristics of a brake friction material. The actual selection of fillers depends on the particular components in the friction material as well as the type of inorganic fillers. Rubber is an example of a commonly used organic filler in brake materials and is usually incorporated into brake pads to reduce brake noises because of their superior viscoelastic characteristics [6]. The physical properties of rubber are affected by vulcanization. Normally, rubbers are vulcanized using sulfur- or peroxide-based systems [7,8]. The common feature of these systems is that they all require an activation energy in the form of heat. However, at high temperatures (150–180 °C), the final properties of the finished product may be affected by the occurrence of several uncontrolled side reactions. The electron beam (EB) crosslinking of rubbers has a number of technical advantages over thermal curing, such as the absence of various noxious chemical additives, rapid curing process, effective penetration of the beam inside the sample, and uniformity and ease of curing [9–15]. Furthermore, radiation curing differs from thermal curing in that the final curing is performed at ambient temperature under closely controlled conditions, such as the radiation dose rate and penetration depth. This form of curing ultimately results in a more well-defined end product. Radiation can produce crosslink densities similar to those obtained through sulfur curing; however, while these methods exhibit some similarities, their net effects are not identical. The type of carbon–carbon crosslink formed in radiation curing improves the mechanical properties at higher temperatures [16]. In addition, Basfar and Silverman [17] showed

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http://dx.doi.org/10.1016/j.compositesb.2013.05.036

that higher abrasion and ozone resistances can be achieved upon irradiation of styrene butadiene rubber.

In this study, the effects of sulfur vulcanization and radiation crosslinking on the hardness, porosity, roughness, friction, and wear properties of semi-metallic friction composites (SMFCs) that contain epoxidized natural rubber (ENR) were investigated.

2. Experimental method

2.1. Materials

ENR 50 with 50% epoxidation was obtained from the Malaysian Rubber Board under the trade name ENR 50. The average Mooney viscosity (measured at ML (1 + 4) 100 °C) was 85.5 MU and the average specific gravity at approximately 25 °C was 0.9366. Alumina nanoparticles with 30–80 nm average diameter were supplied by Nanostructured and Amorphous Materials, Inc. (USA). Other reagents such as sulfur, zinc oxide, and stearic acid were purchased from System/Classic Chemicals Sdn. Bhd.; tetramethylthiuram disulfide (TMTD) was acquired from Aldrich Chemicals; and *N*-cyclohexylbenthiazyl sulfenamide (CBS) and *N*-(1,3-dimethylbutyl)-*N*′-phenyl-*p*-phenylenediamine (6PPD) were supplied by Flexsys America (USA).

2.2. Preparation of friction materials

SMFCs were prepared by blending ENR 50, alumina nanoparticles, steel wool (as the main fiber reinforcement), graphite (as a lubricant), and benzoxazine resin (as a binder) in a Haake Rheomix Polydrive R 600/610 operating at 90 °C and a rotor speed of 60 rpm for 6 min. The complete list of components is given in Table 1.

The obtained composites were then subjected to compression molding under 14.7 MPa at 150 °C for 30 min to form 6 mm thick sheets. The sheets were immediately cooled between the two plates of a cold press at 25 °C.

For the sulfur crosslinking studies, the previously obtained composites were mixed with all curatives (Table 2) in two roll mills in accordance to ASTM D3184-80.

The cure characteristics (t90) of the vulcanized rubber were determined using an oscillating disc rheometer (ODR 2000).

2.2.1. Compression molding

All composites (with and without curatives) were molded in a hydraulic hot press (LabTech Engineering Ltd.) followed by cooling under pressure. The processing parameters are presented in Table 3.

2.2.2. Irradiation

The molded samples (without crosslinking agents) were irradiated in air at room temperature using a 3.0 MeV Cockroft Walton type EB accelerator (model NHV EPS-3000) at a dose range of 50–200 kGy. The acceleration energy, beam current, and dose rate were 3 MeV, 5 mA, and 50 kGy per pass, respectively. The doses were estimated based on machine parameters.

Table 1
Raw material content (in vol%) of the designed samples.

Raw material	Content (vol%)
Steel wool	50
Binder	23
Graphite	11
ENR50	14.55
Alumina	1.45

Table 2
Formulation of the mixes.

Ingredient	Loading (phr)[a]
ENR 50	100
Sulfur	1.6
Zinc oxide	2.0
Stearic acid	1.5
CBS[b]	1.9
TMTD[c]	0.9
IPPD[d]	2.0
Alumina	10

[a] Parts per hundred rubber.
[b] *N*-cyclohexylbenthiazylsulphenamide.
[c] Tetramethylthiuram disulfide.
[d] *N*-(1,3-Dimethylbutyl)-*N*′-phenyl-*p*-phenylenediamine.

Table 3
Processing parameters for compression molding.

Parameters	Samples	
	Radiation crosslinking	Sulfur vulcanization[a]
Temperature (°C)	150	150
Preheating time (min)	3	3
Pressing time (min)	30	30
Cooling time (min)	3	3
Pressure (MPa)	14.7	14.7

[a] The cure time, t90 were determined using an oscillating disc rheometer (ODR 2000).

2.3. Testing

The samples for the porosity tests were cut from the brake pad to a dimension of 25 mm × 25 mm × 5 mm according to JIS D 4418:1996 using a Tech-Lab Digital Heating Circulator HC 20. The surface was polished smoothly without applying abrasive powder. The test samples were then left in the desiccators at 90 °C for 8 h and finally cooled for 12 h at room temperature.

The test samples for the friction and wear tests were cut from the brake pad backing plate to a 25 mm × 25 mm × 6 mm size using a Link Chase machine according to MS 474 PART10:2003. The samples were glued to the braking plate and then attached to the brake clipper on the brake drum. The friction tests were conducted by pressing the test samples against the rotating brake drum. Five samples from each type of vulcanization were subjected to the friction and wear test according to the test program shown in Table 4. The average friction result was then calculated from the recorded result. In addition, the hardness test was performed using the Shore type D Zwick/Roell Durometer according to ASTM D2240.

The weights of the pads for each sample were recorded before and after each test; wear was determined using the mass method following the TSE 555 (1992) standard and was then calculated using the following equation:

$$w = (1/2\pi R) \times (1/f_m n) \times [(m_1 - m_2)/\rho)] \quad (1)$$

where w is the specific wear rate (cm^3/Nm), R the distance between the specimen center and the rotating disk center, m_1 and m_2 are the average weights of the specimen before and after the test (g), respectively, ρ the density of the brake lining (g/cm^3), and f_m is the average frictional force (N).

The friction surfaces of the samples after the friction tests were observed under a scanning electron microscope (SEM, Model Jeol-JSM-6700F) at a voltage of 20 kV. The SEM images were obtained using secondary and back-scattered electrons at an operating voltage of 20 kV.