Effect of Reinforced Conductive Multiwall Carbon Nanotube on the Dynamic Mechanical Properties of Closed Cell Microcellular silicone elastomer nanocomposites

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Abstract— Elastomers filled with hard nanoparticles are of great technical importance for the rubber industry. Dynamic mechanical analysis of conductive multiwall carbon nanotube reinforced microcellular silicone elastomer nanocomposites were used to study the structural analysis of viscoelastic materials as a function of temperature (room temp to 100ºC). The effect of filler and blowing agent loadings (0, 2, 4, 6 phr) on storage modulus (E') and loss tangent (tanδ) characteristics has been investigated. Relative storage modulus decreases with decrease in higher temperature. Both E' and tan δ were found to be dependent on temperature. The Morphological as a function of filler and blowing agent loading has also been reported.

Key words—Blowing agent, conductive multiwall carbon nanotube, dynamic mechanical properties, microcellular elastomers.

I. INTRODUCTION

The dynamic viscoelastic response of closed cell Microcellular silicon rubber nanocomposites can be used as packaging material in sophisticated electronic equipment to absorb shock or vibration during storage, handling and transportation. Dynamic mechanical analysis of closed cell microcellular rubber is an important tool to study the viscoelastic behavior of the material, for evaluation of its use in various engineering applications.

The linear relation between log shear modulus and log density of closed cell microcellular EPDM [1] and polyurethane foams [2], [3] has been reported by researchers. The values of storage modulus have been shown as follows:

$$\text{Storage modulus } (E') = a.\rho^{b} \qquad (1)$$

where a and b are the constants at a particular temperature and vary with the change of temperature, and ρ is the density. Turner et al [4]. Studied the dynamic mechanical response of flexible polyurethane foam specimens as well as of compression-molded plaque of the same material and characterized the morphological structure in the foam. Carbon nanotubes (CNTs) have exceptional electrical, mechanical, electrochemical, chemical, thermal and thermo electric properties [5]. Since the discovery of CNTs by Lijima [6] in 1991, many potential applications have been proposed that exploit these extraordinary properties, such as nano electronics, chemical and physical sensors, biosensors, actuators, composites, integrated circuit manufacturing and scanning probes [7]. MCNTs have high aspect ratio (length to radius ratio), high conductivity and an added advantage of achieving percolation at lower concentration than spherical fillers, which makes them an excellent choice for electrically conducting composites. There are some studies on the applicability of multiwall carbon nanotube as reinforcing fillers in rubbers, such as natural rubber [8], PMMA [9], fluoro elastomer [10], styrene butadiene rubber[11], epoxy [12]. To probe the mechanical properties of interest, the dynamic mechanical analysis (DMA), which is an effective tool to study the viscoelastic properties of the elastomer and to directly measure the frequency and temperature dependency of the modules [13], was used.

The objective of the present work is to study the dynamic viscoelastic properties of conductive multiwall carbon nanotube reinforced microcellular silicone elastomer nanocomposites closed cell microcellular rubber vulcanizates. The effects of blowing agent, temperature on dynamic viscoelastic properties of the vulcanizates were studied. The purpose of the study is also to predict how morphological properties of a suspension correlate to dynamic mechanical properties.

II. EXPERIMENTAL

A. Materials

Silicone rubber, conductive MWCNT, curative, Dicumyl Peroxide (DCP), blowing agent, Azo di carbonamide (ADC-21), Zinc oxide, stearic acid and Paraffinic are the materials and ingredients used in this study.

B. Sample Preparation

The rubber was compounded with the ingredients (Table.1) in a laboratory size two-roll mixing mill at room temperature according to ASTM D3182-74. The friction ratio used was 1:1.2. The curing agent and blowing agent added at the end of the mixing in the mill. The mixed materials were molded at 160 ° C to 80% of their respective optimum cure times in an electrically heated hydraulic press at a pressure of 5.0 MPa with a specially designed mold for microcellular vulcanizates. All sides of the mold were tapered to 30 ° to facilitate the expansion of the microcellular rubber and to achieve better mold release. Expanded microcellular sheets were post cured at 100 ° C for one hour in an electrically heated air oven.

III. TESTING

A. SEM Studies

SEM studies were carried out for understanding the cell structure using JEOL JSM 5800 SCANING ELECTRON MICROSCOPE. Razor cut surfaces from microcellular sheets surfaces of the tensile specimen were used as samples for SEM studies. The surfaces were gold coated before being studied. The function of this electron microscope is similar to its optical counterpart except that a focused beam of electrons is employed instead of light to

Table.1 Formulations of unfilled and MWCNT filled

mix.no.	SM1	SM2	SM3	SM4
Silicone Rubber	100	100	100	100
MWCNT	0	2	2	2
Blowing Agent	0	2	4	6

Each mix contains ZnO-5 Phr; Stearic Acid-1.5 Phr and Dicumyl peroxide-1 Phr

"image" the specimen and to gain information on its structure and composition.

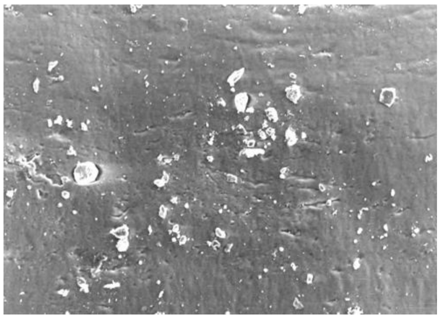
B. Dynamical Mechanical Analysis (DMA)

DMA measurements were done with TA Instruments DMA Q800 equipment. The maximum force was 18 N and maximum frequency 200 Hz. The measurements were done in ambient pressure using Film/Tension measuring head. The DMA frequency range was at 1Hz with a temperature range from room temperature to 100^0C at a strain of 1%. DMA is a technique that is employed in the structural analysis of viscoelastic materials, that is, materials displaying both elastic and dissipative components of deformation. Storage modulus (E') and loss tangent (tanδ) curves was taken in DMA for different blowing agent concentration.

IV. RESULTS AND DISCUSSION

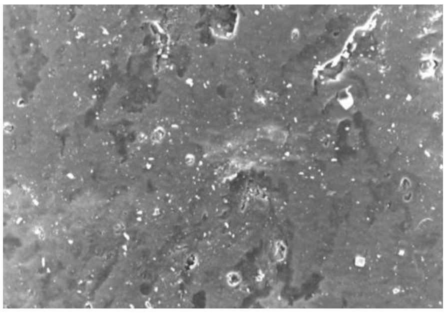
A. Morphology of Razor Cut Surfaces

SEM Photomicrographs of razor cut surfaces of various unfilled and filled microcellular rubber are shown in figure.1.The number of cell increases as the blowing agent loadings increase. Average cell size decreases with increase in blowing agent loading from 2, 4, 6 phr. Photomicrographs shows that with filler loading the average cell size increases. The micro-bubbles formed by decomposition of the blowing agent defused and collapsed with each other due to decrease of the cure rate with increase in filler loading[15]. Thus with increase in filler loading average cell size as well as maximum cell size increase. The number of cell increases with blowing agent loading.

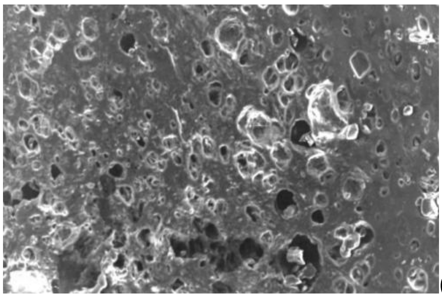


(a)

(b)



(c)



(d)

Fig.1. SEM photomicrographs of razor cut surfaces of microcellular Silicone elastomer nanocomposites: effect of Filler and Blowing agent loadings (a) 0 phr; (b) 2 phr; (c) 4phr; (d) 6phr.

B. Temperature-dependent dynamic properties

Figure 2 shows the variation of storage modulus E', at 1 Hz, with temperature for 2 phr multiwall carbon nanotube reinforced microcellular silicone elastomer nanocomposites, with different blowing agent loadings.

The nature of storage modulus–temperature curve of closed cell microcellular rubber is similar to that of solid rubber. The decomposed gas is enclosed in closed cells. The deformation of enclosed gas in closed cell is elastic in nature and has little effect on the storage modulus [14]. Hence, E' decreases continuously with increase in blowing agent loading because of the decrease in solid rubber content, that is, with decreasing density.

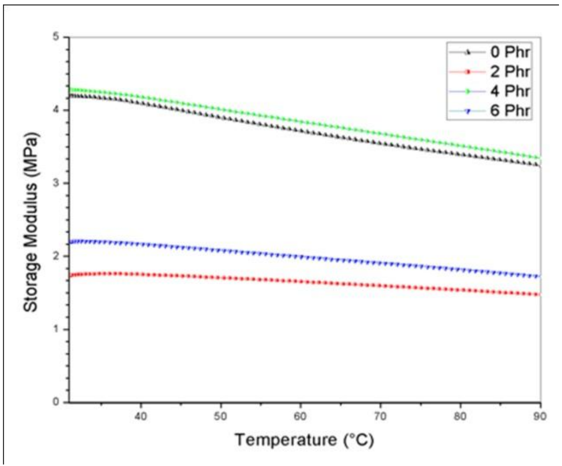


Fig.2. Effect of blowing agent loading variation on storage modulus in 2 phr MWCNT reinforced microcellular silicone elastomer nanocomposites.

The variation of loss tangent (tan δ) with temperature for 2 phr multiwall carbon nanotube reinforced microcellular silicone elastomer nanocomposites is shown in Figure 3. It exhibits the damping behavior of closed cell microcellular vulcanizates, with variation of blowing agent loadings. The higher tan δ at higher blowing agent loading may be due to induced strain by decomposed gas pressure. In rubbery region, i.e., above 25–30°C, the tan δ values decreases at lower blowing agent loading up to 2 phr. Whereas at higher blowing agent loading, that is, at 6 phr tan δ values increases. It is because the enclosed gas in closed cell has little contribution toward the damping property [1] For closed cell microcellular rubber at a higher blowing agent loading, the decomposed gas pressure increases inside the cell [16]. But in case of open cell, the damping behavior is largely affected by the viscosity of the fluid [17]. Owing to the increase in decomposed gas pressure, the cell membrane remains in a strained condition. This increase in strain increases the tan δ value, i.e. damping to some extent.

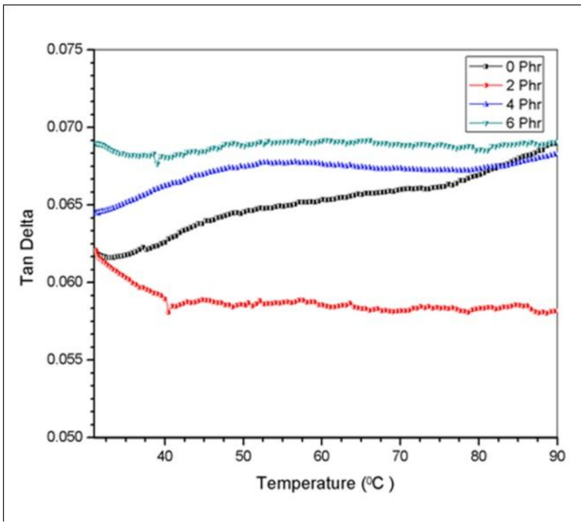


Fig.3. Effect of blowing agent loading variation on loss tangent (tan δ) in 2 phr MWCNT reinforced microcellular silicone elastomer nanocomposites.

V. CONCLUSIONS

Multiwall carbon nanotube MWCNT reinforced silicone elastomer nanocomposites are prepared. SEM micrographs show dispersion of multiwall carbon nanotube homogenous in the polymer matrix and microbubbles will be increased as filler loading increased, which is clearly observed from the SEM photomicrograph of sample containing 6 phr filler loading. The storage modulus (E') or stiffness of multiwall carbon nanotube reinforced microcellular silicone elastomer nanocomposites decreases concomitantly with increase in blowing agent loading. The Loss tangent (tan δ) value of microcellular silicone elastomer nanocomposites increases with increase in blowing agent loading in rubbery region at lower blowing agent loading. tan δ value decreases at higher blowing agent loading, and again it increases due to increase of decomposed gas pressure inside the cell.

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