

Hysteretic damping in XNBR – MWNT nanocomposites at low and high compressive strains

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ABSTRACT

In this study, compressive hysteretic damping properties of carboxylated nitrile rubber (XNBR) and its composites containing multiwalled carbon nanotubes (MWNT) were investigated at high strains by Universal Testing Machine (UTM) and at low strains by Dynamic Mechanical Analyzer (DMA). Degree of damping is less than the neat XNBR at low MWNT loading and it increases significantly at higher loading. At constant MWNT loading, the extent of damping depends on applied strain. Mullins effect is observed in both neat XNBR and composites due to the presence of ionic clusters. Differential Scanning Calorimetry (DSC) and X-ray diffraction analysis (XRD) confirm the reversible nature of the ionic cluster formation/deformation. Deformation of ionic clusters and nanotube agglomerates contribute to the overall increase in damping. Morphology characterization by Transmission Electron Microscope (TEM) reveals agglomeration of nanotubes at higher loading levels. A schematic diagram about the structural changes due to application of heat and stress is proposed. The results would be of great assistance in the design of application specific composites for various engineering applications.

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1. Introduction

Reinforcement using fillers is an important area of research in rubber compound development. A variety of fillers are being used to get substantial improvement in critical properties. The property enhancement is found to depend on the content, size and aspect ratio of the fillers as well as on the extent of dispersion. Rubber nanocomposites have gained great importance in recent times on account of their wide range of potential applications in many areas [1]. Due to the high surface area of nanofillers, one can achieve enhanced filler–matrix interaction at a significantly lower level of filler loading compared to the conventional fillers. Nanomaterials such as carbon nanotubes (CNT), carbon nanofibers, nano clays, nano silica, nano zinc oxide and nano calcium carbonate are being attempted as fillers in rubber compounds. Among these, CNTs, due to their very high mechanical properties and other exotic functional

properties, have become a primary choice for researchers to develop advanced multifunctional nanocomposites for many engineering applications [2]. Single walled nanotubes (SWNT) and multi walled nanotubes (MWNT) are the two major types of carbon nanotubes. MWNTs have attracted many researchers to develop new generation composite materials, due to low production cost and high performance [3,4].

Filler-reinforced rubber compounds when subjected to cyclic deformations show many interesting nonlinear effects like Payne effect, Mullins effect and hysteresis energy loss [5,6]. Development of rubber compounds for engineering and structural applications requires a thorough understanding of these phenomena. Payne effect relates dynamic modulus to filler–filler interaction. Our previous study has shown that MWNT filled XNBR rubber exhibits Payne effect at higher loading levels [7]. Mullins effect is a strain induced softening behavior observed in filled as well as gum rubber systems [8]. This is associated with a reduction in stress (softening) at a given strain in a cyclic deformation, during the unloading path as compared to the loading path. Further, when previously unstrained rubber is subjected to strain cycles at constant peak value,

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the first few oscillations result in reducing peak stress values. The effect is irreversible at any particular temperature, though a partial recovery is possible at an elevated temperature. This phenomenon is attributed to the mechanical hysteresis of the filler particles due to the debonding from each other or from the polymer chains. As a result, highly filled systems exhibit a prominent reduction in stiffness as compared to less filled systems [9,10]. The extent of hysteresis loop area provides an idea about the energy loss during cyclic deformation. Hysteresis loss in filled rubber systems had been investigated extensively at high strain levels (>100%) in tensile mode [11,12]. Hysteresis in CNT composites has been modeled numerically considering the slippage of nanotubes in the host matrix under shear stress [13]. Heat generation under cyclic deformation is found to be more in the case of MWNT-filled rubber composites due to the poor interaction and higher friction between MWNT and matrix [14]. Mullins effect has been observed in MWNT filled natural rubber composites due to the strain induced debundling of nanotube agglomerates under tensile load [15]. Numerous papers report hysteresis under tensile or shear load, whereas in practical scenario, in many engineering applications, rubber compounds are subjected to cyclic compressive loads resulting in low to high strains. A large number of applications in the field of shock and vibration damping utilize rubber in diverse configurations, undergoing different type and extent of strains [16]. A detailed investigation of the hysteresis in nanocomposites at low as well as high strains is necessary to comprehend the mechanism and origin of damping.

Hence, in the present work, the hysteresis behavior of MWNT-filled carboxylated nitrile rubber (XNBR) is thoroughly analyzed in compression mode in a dynamic mechanical analyzer at low strain and in a universal testing machine at high strain. XNBR is a high performance specialty rubber having pendent carboxyl groups at random along the chain. It has superior mechanical, and damping properties in addition to better abrasion resistance. The presence of reactive and polar functional groups makes it a suitable candidate to study interactions between rubber matrix and various fillers [17].

2. Experimental

2.1. Materials

Carboxylated nitrile rubber (XNBR) used was Chemigum NX-146 supplied by M/s Eliochem, India, having Mooney viscosity: 45 ± 5 (method: ML 1 + 4[100 °C]), acrylonitrile content: 33% and carboxylic acid content: 1%. Multiwalled carbon nanotubes (MWNT), having 95% purity and average diameter of 20–30 nm was supplied by M/s Chemapol, Mumbai. The rubber was compounded in a 6" (150 mm) two-roll mill along with various loadings of MWNT, namely 0, 0.5, 1.5, 2.5, 3.5 and 4.9% (by volume) of the final composite. Other ingredients added were zinc oxide: 0.8%, stearic acid: 2.2%, N-cyclohexyl-2-benzothiazole-sulfenamide (CBS): 1.1%, sulfur: 1.1% and Vulcanox HS: 0.8% (all by volume %). The stocks were cured to 90% (t_{90} : 30 min) under pressure at 150 °C, based on a rheometric study in Monsanto Rheometer 2000.

2.2. Methods

Compression studies were carried out according to ASTM standard D 575-91. The high strain compressive experiment was carried out in a Universal Testing Machine (UTM), model 1476 of Zwick, Germany, at room temperature. The test specimens were of 28.6 ± 0.1 mm in diameter and 12.5 ± 0.5 mm in thickness. The specimen gauge length was 12.5 mm. The sample was kept in between the platens, along with sandpaper to prevent slippage. It was

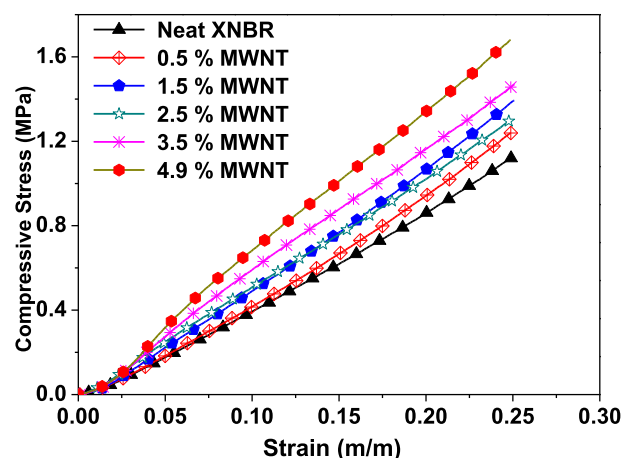


Fig. 1. Compressive stress–strain plots of neat XNBR and nanocomposites obtained from UTM experiment.

subjected to a compressive load at a constant crosshead speed of 10 mm/min up to 25% strain and consequently the load was removed at the same speed. A total of 10 cycles of loading and unloading were carried out. In all the cases the loop area became constant by the fifth cycle. Hysteresis loss of the first cycle and fifth cycle (constant area cycle) are taken for investigation.

The low strain compressive experiment was carried out in a Dynamic Mechanical Analyzer (DMA) model Q 800 of TA instruments, USA. The test specimens consisted of 13 ± 0.1 mm in diameter and 2 ± 0.1 mm in thickness. The specimen gauge length in DMA experiment was 2.0 mm. The sample was kept in between the platens and subjected to the compressive load up to 2% strain, at a strain rate of 10 $\mu\text{m}/\text{min}$, thereafter the load was removed at the same rate. The strain rate and the maximum strain are chosen based on the resolution of the optical sensor of the DMA. A total of 5 cycles of loading and unloading were carried out. Hysteresis loss of the first cycle and last cycle (constant area cycle) are taken for investigation.

The dispersion characteristics of CNT in the nanocomposites were analyzed using a JEOL 3010 Transmission Electron Microscope

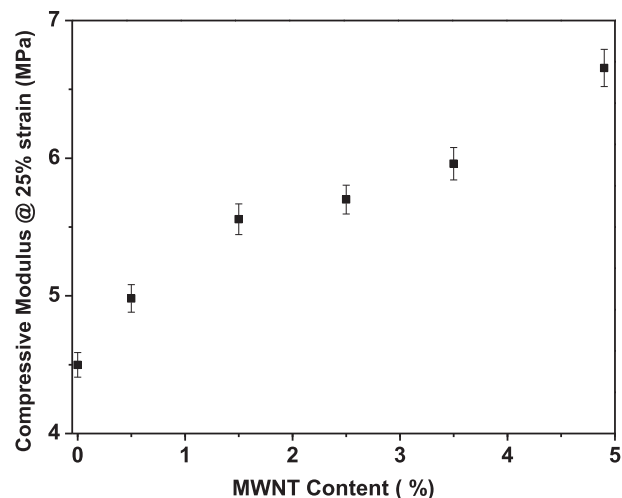


Fig. 2. Compressive modulus of XNBR-MWNT nanocomposites at 25% strain.

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