DISPERSION AND MECHANICAL/ELECTRICAL CHARACTERISATION OF CARBON NANOTUBE POLYMER FOAMS

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Keywords: Carbon Nanotubes Polymer Foam

Abstract

The mechanical and electrical properties of Multi-Wall Carbon Nanotube (MWCNT) Polyurethane (PU) foam composites were studied with nanotube loading in the range 0.125-1 wt%. The mechanical properties of Young's modulus and energy absorbed showed improvements over reference samples and indicated the effectiveness of the dispersion methods. The measurements of electrical properties indicated that at these nanotube loadings the composite is an insulator.

1 Introduction

The mechanical and electrical properties of Carbon Nanotubes (CNT) composites have been well documented in polymers such as epoxies, but challenges remain in achieving measurable performance in foam networks. In particular the formation of a conducting network within a foam composite has proved difficult with conventional filler materials [1], thus the exceptional electrical properties of CNT's could be employed to overcome these difficulties.

A great deal of work has been carried out on the dispersion of CNT's for their use in polymer composites, using a variety of solvents, surfactants, mixing and ultrasound techniques [2]. Ultrasound is the most popular method of dispersion, but realistically is limited to small batches for lab scale work [3]. In this work an industrial, scalable methodology for the dispersion of Multi-Walled Carbon Nanotubes (MWCNT) in polyurethane foam (PU) is presented. This method is based on high shear fluid processing, enabling consistent batch production of CNT polymer foam composites. Assessment of the method is carried out directly on the dispersed samples, and with respect to the final MWCNT/PU foam composites.

2 Experimental procedure

2.1 Materials

MWCNTs of diameter 110-170 nm, length 5-9 micron, of 90+ purity were supplied by Sigma-Aldrich inc. and were dispersed in a Polyurethane (PU) foam. The PU foam was a 2-part water blown foam named Cellante, supplied by International Polyurethane foam Ltd.

Two different methods were chosen to achieve an adequate dispersion of the nanotubes in the foam matrix. The first method used an ultrasonic bath to achieve nanotube dispersion in the PU foam, this being a commonly employed and proven research method to disperse nanotubes within a polymer, and a second method to assess the possibility of using high shear mixing to achieve nanotube dispersion. The second method, while less commonly used, represents a methodology that, unlike the ultrasonic method, is one which is industrially scalable.

2.2 Ultrasonic dispersion

The MWCNTs were first weighted and manually stirred into Part A of the polyurethane foam, them the samples were dispersed using a Branson 2510 Ultrasonic bath. The MWCNTs and Part A foam mixture was then sonicated at full power for 1 hour at to achieve dispersion. Part B of the polyurethane foam was added to the MWCNT/PU mix, and manually stirred for 30 seconds until the foaming process began. Samples were then poured into an open mould and allowed to freely expand in an atmospheric environment. The nanotube foam composite was then left to achieve a full cure at room temperature for 24 hours.

2.3 High shear mixing

High shear mixing was carried out with a Silverson mixer, using the lowest speed available for 30 minutes. Longer mixing times were ruled out as the temperature increase and subsequent foaming of the MWCNT/PU foam mix was considered likely to be of detriment to the final composite.
The addition of Part B and subsequent curing of the foam composite was carried out with the same methodology as the previous ultrasonically dispersed system.

2.4 Sample preparation

Samples were prepared in a range of MWCNT loadings by weight as follows 0%, 0.125%, 0.25%, 0.5%, 0.75% and 1%, in both the sonicated and high shear mixing methods. The MWCNT/PU composites were then prepared into cylindrical samples with diameter of 17.5 mm and 20 mm length for mechanical and electrical characterisation. Figure 6 below shows the sonicated samples, in increasing MWCNT weight % from left to right. Visually the nanotube loading is clearly illustrated, with the colour change indicating increasing levels of nanotubes.

Figure 6: Nanotube samples during preparation

2.5 Mechanical characterisation

Mechanical characterisation was carried out using a Lloyd Instruments TA500 texture analyser. This equipment was set up to perform compressive testing, as is common with the testing of foam materials. A 500 N load cell was used, and tests carried out using the following parameters:

- Pre-load: 0.1 N
- Cross head speed: 5mm/min
- A continuous load was applied until 10mm deflection was achieved

2.6 Electrical characterisation

Measurement of the electrical conductivity was carried out using the four probe method. Samples were prepared by slicing the same 20mm diameter samples as shown in figure 1 into 5 mm thick specimens. The outer surfaces of the samples were then coated with silver conducting paint with 2 conducting strips on each side and left to cure for 24 hours before testing. Electrodes were connected to a power supply (rapid SPS 9602) and a digital multi-meter.

3 Results and discussion

Young’s Modulus and Energy absorbed were calculated from the compression tests carried out to characterise the MWCNT/PU foam composites. Energy (Joules) is work done, and thus is the area under the curve achieved by integration. In both cases the software supplied with the test equipment carried out the calculations and presented the data in the form of Young’s Modulus (MPa) and Energy (J).

Both sets of data were normalised with respect to density, to reduce the effects of geometric variation, and the variability in the foaming mechanism, the density was calculated from measuring the volume, and weighing each sample.

3.1 Young’s modulus

Young’s modulus of the sonicated samples initially drops relative to the reference sample as the nanotube loading is increased, until loading of about 0.5% is achieved, figure 2. After reaching 0.5% nanotube loading the modulus increases at loadings of 0.75% and 1 %. The high shear samples show a marked increase of Young’s modulus over the reference sample, but increasing the nanotube loading does not appear to influence the properties out with the sample error. Comparing the 2 methods of dispersion, the high shear method clearly shows an improvement in the sample modulus over the reference sample, but increasing nanotube loading appears to have little influence, while the sonicated samples only show an improvement at higher loadings.

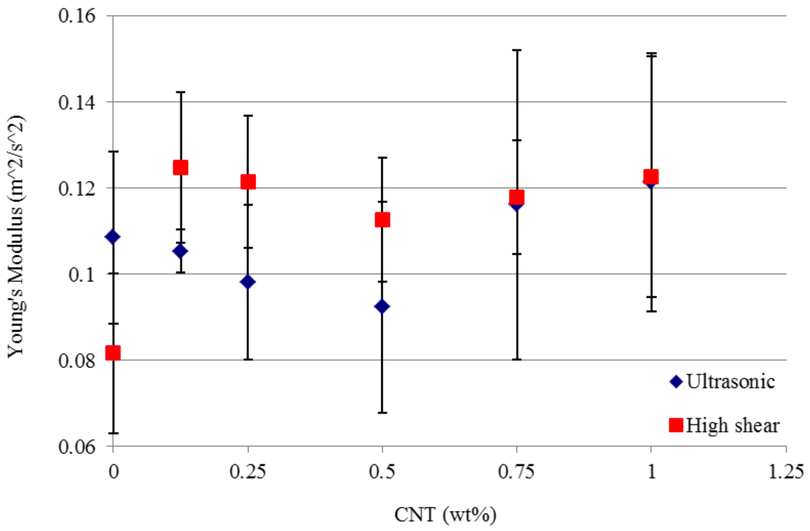

Figure 2. Normalised Young’s modulus for ultrasonic and high shear dispersion

3.2 Energy Absorbed

The energy absorbed plot figure 3 shows a more significant change in the properties of the MWCNT/PU foam composites, with up to a 30% increase in the energy absorbed in the sonicated sample at 0.25% nanotube loading. The high shear mixed sample showing approximately a 20 to 25% improvement over the reference sample. Both methods of dispersion clearly show improved performance in the 0.125 to 0.25% range, and the high shear samples show improvements all the way up to 0.75% nanotube loading. At 1% loading the high shear samples show a marked drop in the performance of the MWCNT/PU composite which indicates the increase in loading has become detrimental to the composite. It is likely at this point the nanotubes are forming agglomerates in the composite and are creating large unreinforced voids. It can be concluded from this assumption that the high shear dispersion is no longer suitable at these levels of nanotube loading. A similar drop in the energy absorption of the sonicated samples from 0.5% onwards indicates the dispersion is no longer as effective as indicated in lower loadings, but has not proven to be as detrimental to the extent seen in the high shear mixing samples at 1% loading.

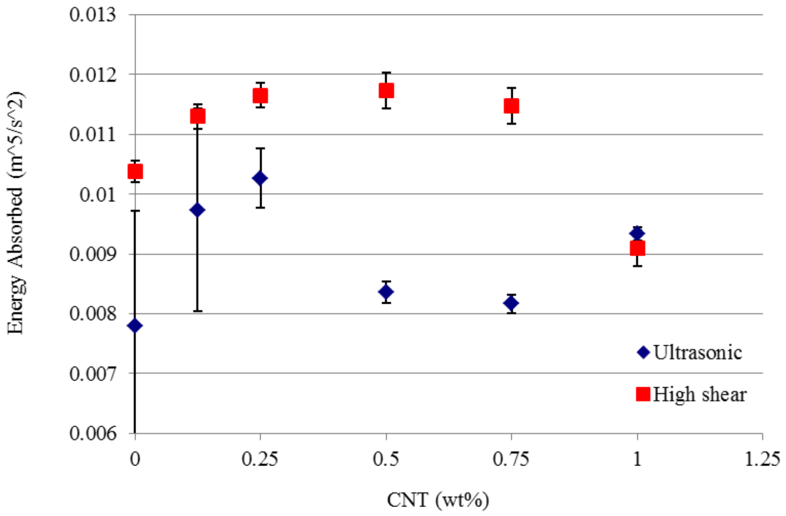

Figure 2. Normalised energy absorbed for ultrasonic and high shear dispersion

3.3 Electrical characterisation

A 4-probe method was used to assess the conductive performance of the foam materials. Unfortunately these tests indicate that no percolating network was possible at this level of nanotube loading.

4 Conclusions

Both methods of dispersion show improvements in the mechanical properties of the MWCNT/PU foam composites particularly at low nanotube loading. The high shear method clearly indicates an industrially scalable technique, which show improvements over the ultrasonic method for the preparation of MWCNT/PU foams. A percolation network was not achieved with these dispersion techniques.

References

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