Tunable morphology and its influence on electrical, thermal and mechanical properties of carbon nanostructure-buckypaper

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\textbf{A B S T R A C T}

In this study, free-standing buckypapers (BPs) made up of a three-dimensional network of multi-walled carbon nanotubes (MWCNTs) that are branched, cross-linked, and share common walls, defined henceforth as multi-walled carbon nanostructures (MWCNSs) are characterized to obtain their electrical, thermal and mechanical properties. Two types of free-standing sheets are studied, namely uncompressed buckypapers (UBPs) and compressed buckypapers (CBPs). Both are processed via a vacuum filtration method, with subsequent compressive force applied between platen presses to obtain CBP. CBPs are found to exhibit improved thermal and electrical conductivities since higher density of CNT networks leads to more conductive pathways. However, mechanical properties of CBPs and UBPs are approximately the same. Local anisotropic behavior of both BPs allows them to have either positive or negative Poisson’s ratio. While UBP is suitable for membrane applications due to its mechanical stability and well-defined pore characteristics, the CBP offers a compact structure that can reduce penetration of the polymer matrix in composite applications such as for integrated circuit packaging and EMI shielding.

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1. Introduction

Carbon nanotubes (CNTs) have attracted remarkable attention due to their excellent mechanical, thermal and electrical properties [1]. They have been utilized to fabricate macroscopic CNT sheets, commonly referred to as ‘buckypapers’ (BPs). These BPs are suitable for both light-weight structural and functional applications. These free-standing sheets are cohesively bound by van der Waals’ interactions among entangled CNTs. The central idea behind the fabrication of BP is to utilize the outstanding properties of individual CNTs in macroscopic form. This macroscale CNT sheet is advantageous to facilitate easier handling of CNTs and to improve the safety of using CNTs in industrial scale. Although the performance of BPs is lower than that of individual CNTs due to the low interaction energy, densely-packed and inter-entangled structures of CNTs offer multi-functional capabilities owing to their mechanical stability, flexibility, high electrical and thermal conductivity [2–5]. These characteristics make BPs suitable for membranes [6–8], electrodes [3,9,10], actuators [4,11–14], sensors [15–18], heat conductors [19–21] and for structural reinforcement in polymer composites [5,22–27].

BPs are commonly prepared by dispersing CNTs or chemically modified CNTs in a solvent medium followed by vacuum filtration process through a microporous membrane [5,28]. Through this process, CNTs are generally oriented randomly in the BP unless specific techniques such as the application of strong magnetic fields are used to align them [29,30]. Mechanical spinning of CNT from the CNT arrays or flakes may also result in a BP with aligned CNTs [21,31]. It has been demonstrated that properties of BPs are affected by many parameters such as CNT type (single- or multi-walled) [32], dimension (length and diameter) [19], purity [14,33,34], chemical modification of CNTs [28] and degree of alignment [21]. Smaller diameter and higher purity of CNT usually lead to BP with higher tensile strength, whereas larger CNT diameter results in BP with higher porosity and lower tensile strength [8]. Functionalization of CNT can improve inter-CNT interactions in the BP and thus improves their mechanical performance and conductivity to a certain extent [28,35,36]. The solvent medium used for the CNT dispersion and the synthesis conditions as well as post-treatments have a significant effect on the pore size distribution and nanotube packing density and hence on the final properties of the BP [21,24,37].

Through careful selection of CNT types and processing conditions, desired characteristics and properties of the resulting BPs can be achieved. However, fabricating macroscopic form of CNT networks with controlled geometry, porosity and properties is challenging. In addition to the experimental efforts, computational modeling strategies are important to develop tools so as to optimally design the CNT...
systems and configurations for specific targeted behavior and properties. Modeling of BPs based on a coarse-grain model of carbon nanotubes, with possible variation in parameters including initial CNT density and ratio of CNT type (single- and double-walled) was performed by Cranford and Buehler [38]. Through manipulation of the parameters, the porosity and Young’s modulus of the BP can be tuned over a range of 0.3–0.9 and 0.2–3.1 GPa, respectively. Yang Li et al. [39] have developed an algorithm that combines molecular dynamics and Monte Carlo methods to equilibrate initial structure of BP. Initial structure of BP was generated by applying random walk theory. Entanglement and bundling mechanisms are found to influence the pore size and mechanical properties of the BP.

In this paper, both UBPs and CBPs are characterized in terms of its surface morphology, pore characteristics, thermal, electrical and mechanical properties. These BPs are synthesized from MWCNS. The MWCNS are produced in an industrial scale using chemical vapor deposition (CVD) method, capable of making nanotubes in high-throughput. Both BPs are processed by vacuum filtration method, with subsequent compressive force applied between platen presses to obtain CBP.

2. Experimental procedures

2.1. Synthesis of multi-walled carbon nanostructures and buckypapers

Multi-walled carbon nanostructures (MWCNSs) have been developed by Applied Nanostructured Solutions, LLC through a unique CVD process specifically characterized by their low cost/ high volume production process [40]. Unlike the conventional CNT processes that mostly focus on production of high purity CNT, the MWCNS process allows rapid and continuous growth of CNT on a moving substrate. While the conventional CNT growth rate is typically in the order of several microns per minute, the MWCNS process allows rapid and continuous growth of CNT on a moving substrate. As a result, the nanotubes within the MWCNS are more defective than those in a conventional CNT flakes. MWCNSs consist of bundles of aligned MWCNTs. Inner walls are intact, but the outer most wall that has 5 or 7 member of “C” rings which are covalently bonded with adjacent similar structure of other MWCNTs are defective. These defective features are characterized by its highly entangled, branched, cross-linked, and wall-sharing architecture.

The vacuum filtration method was used to fabricate the BPs. The UBP was prepared by adding the MWCNS flakes into an ethanol/distilled water mixture, with subsequent gentle stirring and sonication with 1/2" tip diameter at maximum amplitude for 10 min. The ethanol/distilled water volume ratio and the MWCNS suspension concentration were set to 1:1 and 0.175 g/L, respectively. Afterwards, the MWCNS suspension was vacuum-filtered through a glass fiber filter. After the free-flowing liquid was vacuumed and a damp MWCNS sheet was formed, the BP was peeled off from the filter and then put in a convection oven and dried fully. The CBD was fabricated using the same filtration method, with subsequent application of mechanical pressure between platen presses at 750 psi after peeling it off from the filter. The MWCNS suspension concentration was adjusted to obtain a uniform thickness of 30–40 μm for both BPs. Since MWCNSs are branched and covalently bonded with adjacent nanotubes, applied pressure of 750 psi was found to be optimum to obtain good consolidation of CBPs. Lower pressures didn’t yield a good consolidation of CBPs while higher pressures mechanically damaged the resultant BPs.

2.2. Characterization of multi-walled carbon nanostructures and buckypapers

Two different scanning electron microscopes (SEMs), namely Quanta 250 and Nova NanoSEM were used to elucidate the surface morphology of the BPs. Quanta 250 examines the sample at 10 mm working distance, whereas Nova NanoSEM which utilizes 5 mm working distance is capable of observing the sample at higher magnification. Transmission electron microscope (TEM) Technai TF20 was used to characterize the MWCNS. Quanta 3D dual beam SEM/FIB was used to evaluate the cross section morphology of the BPs. To obtain cross-sectional image of the BP, the sample was firstly tilted at 52°. Afterwards, the cross section was milled by focused ion beam (FIB) using a high beam current (7 nA) followed by several smoothing steps using lower beam current (0.3 nA). Finally, the resulting smooth cross-sectional area was observed by SEM.

Pore size distribution of the BPs was measured by two different methods; SEM image analysis and capillary flow porometer (CFP). Image analysis of the top surface of SEM image of the BPs was conducted by image segmentation of the pores and MWCNS via gray level thresholding technique using ImageJ software. The areas of all pores were obtained and the diameters of the pores were calculated by considering the pores as circular pores. The CFP measurement (Porous Materials, Inc.) was performed based on wet/dry flow method. The circular sample of the BP was wetted with Galwick, a wetting liquid with low surface tension of 15.9 dyn/cm, and then placed into the porometer sealed chamber through which a gas flows. Subsequently, the pressure of the gas was increased gradually and the through flow was recorded. The pressure needed for gas to flow through the wet sample and to remove the wetting liquid completely was used to measure the pore size. In wet/dry flow method, the porometer only considers one diameter per pore, which is the throat (most constricted) diameter across the through-thickness path of the BP. In the current work, the maximum applied pressure was 200 psi, which is adequate for detecting smallest pore diameter of 33 nm.

The electrical resistivity of the BPs was measured using a four point probe (LakeShore, USA) according to the Van der Pauw method. The electrical contact was made by placing the contact needles onto the four corners of the 1.1 × 1.1 cm² size of the BP sample. To facilitate the electrical contact, silver conductive ink was manually painted onto the four corners of the sample. The electrical conductivity (σ) was computed by \( \sigma = 1/\rho \), where \( \rho \) is the electrical resistivity of the sample.

The Netzsch LFA 457 Microflash was used to determine the through-thickness thermal diffusivity (α) of the BPs according to ASTM E-1461 [41]. Each sample was tested with a reference sample for the specific heat (\( C_p \)) calculation. The density (ρ) was evaluated from the measured weight and volume of the sample. From α, ρ and \( C_p \) measurements, the thermal conductivity (κ) was determined by

\[
\kappa = \alpha \times \rho \times C_p
\]

2.3. Mechanical tests

Quasi-static tensile tests of the BPs with rectangular dimension of 60 × 6 × (thickness) mm² were conducted in displacement mode with a crosshead speed of 20 μm/min in Zwick Roel 2005 universal tensile machine with 20 N load cell. The thickness of both BPs ranges from 30 to 40 μm. The average thickness at three locations of each tensile sample was used for the stress evaluation. Unloading-reloading tests were performed with the same crosshead speed. To avoid high stress concentration in the grip zone and to ensure effective clamping, adhesive foam tape was used as a specimen tab.

The evolution of the strain field on the surface of the BP during loading was evaluated by digital image correlation (DIC) technique. Similar to the use of extensometer, the DIC technique is important to obtain valid strain measurements. Micro deformation in the grip zones can under-/overestimate the measured strain depending on the shape of the test sample and the material behavior. This effect can be eliminated from the measurement using DIC technique. Moreover, Poisson’s ratio and strain contour during the test can be evaluated.
The DIC setup consists of CCD Camera with spatial resolution of 2448 × 2048 pixels, Schneider Xenoplan 2/3″ (11 mm) lens, and two light source lamps. The camera was mounted on a tripod with built-in spirit level to ensure the horizontal level. To perform DIC measurement, random white speckle patterns were produced over the inherent black specimen surface of the BPs. Air brush (Harder & Steenbeck GmbH & Co. KG.) of 0.6 mm nozzle diameter and high flow acrylic paint of titanium white color (Golden Artist Colors, Inc.) were used for this purpose. The speckle size was optimized to be more than three pixels to prevent image aliasing and to ensure accuracy of the DIC analysis [42]. By the current DIC setup, it is assured that the measurement error is <1%. It should be noted that even though the BP has nanoporous structure, the white speckle produced by the spray paint does not penetrate into the BP due its relatively high surface tension. This was verified by directly comparing the mechanical properties of the un-speckled and speckled samples. To further ensure that the adhered white speckle has no effect on the mechanical properties of the BPs, the tensile tests were performed within an hour of spray paint application.

The software from correlated Solutions, Inc.; Vic-Snap and Vic-2D were used respectively to capture images during tests and to perform data correlation analysis. Since there is no built-in synchronization device between the tensile machine and DIC system, load display of the tensile machine was also captured by the camera to ensure data correspondence between both systems. Optical image recognition was used to extract the load values. The area under the stress-strain curve which represents the energy to break the sample was determined to estimate the toughness of the BPs.

### 3. Results and discussion

#### 3.1. Morphology of carbon nanostructures and buckypapers

The MWCNS flakes produced from the continuous, in situ CVD synthesis process is presented in Fig. 1a–b. This MWCNS flake micrograph was captured after removing the MWCNS flakes from the substrate. It can be seen that the synthesis process results in an oriented MWCNS with fibers attached to each other. One can see from Fig. 1a–b that the length of the MWCNS is approximately 30 μm. The MWCNSs were extracted from the MWCNS flake for further analysis by transmission electron microscope (TEM). It was observed that the currently studied MWCNSs have number of walls varying from 2 to 9 and outer diameter ranging from 4 to 14 nm. An example of the TEM image of MWCNS with different number of walls as well as the dimension of the inner and outer diameters of the nanotubes can be seen in Fig. 1c.

Both UBP and CBP studied in this work are flexible, lightweight and glossy (Fig. 2). The glossy nature of BPs qualitatively represents smooth and uniform surface such that it has the ability to reflect light. Observation by scanning electron microscope at different magnifications shows that MWCNSs are randomly oriented (Fig. 3a–d). The random orientation of MWCNS is also confirmed from the cross-sectional SEM images which show no specific orientation of the CNS fibers (Fig. 3e–f). Even though the BPs were fabricated from aligned flakes, randomly oriented MWCNSs within the BPs were achieved due to the mechanical stirring and sonication procedures during the preparation of the MWCNS suspension.
The in-plane SEM images at low and high magnifications (Fig. 3a–d) and cross-sectional SEM images (Fig. 3e–f) show that the CBP has a more closely-packed fiber arrangement and lower porosity than those of the UBP. In all SEM images, the darkest zone represents the porosity of the BP. In both UBP and CBP, MWCNS’ wall-sharings and wall separations (branching) were observed (Fig. 3c–d). This indicates the unique nanostructure of the currently studied BPs. According to TEM analysis, the diameter of MWCNS in the BP ranges from 4 to 14 nm. SEM images (Fig. 3) show that several MWCNSs have diameters larger than 14 nm. This is due to wall sharing of two or more MWCNS (i.e., the alignment of two fibers of lower diameter). The average length of the randomly oriented BP cannot be evaluated through 2-D SEM images because every individual fiber is oriented both in-plane and through-thickness directions. However, the MWCNS length in the BPs can be estimated by observing the MWCNS length in its flake form, which is around 30 μm (Fig. 1a–b).

3.2. Pore size distribution and density of the buckypapers

Pore characteristics can be evaluated by several methods such as SEM, mercury intrusion porosimetry (MIP), capillary flow porometer (CFP) and N₂ adsorption isotherm technique. SEM imaging of the BP surface followed by image analysis is useful for evaluating “apparent surface” pore size [7,35,43]. MIP involves the use of toxic mercury and
In the current work we used two methods; SEM image analysis and CFP to evaluate the pore characteristics of the BPs. It should be noted that the current image analysis is only performed on the top surface of the SEM image of the BPs and therefore the analysis does not contain information across the thickness of the BPs. Consequently, the measured pore diameter should be considered as apparent pore diameter. In general, pore diameters of UBP are higher than those of CBP. The pore diameter of UBP varies from 5 to 70 nm while that of the CBP ranges from 5 to 50 nm. The average pore diameters of UBP and CBP are 38 and 27 nm, respectively. The applied compression load stacks together the nanotubes in a closer arrangement and thus it reduces the pore diameter of the BP. We further characterized the pore diameter by CFP method. The current CFP machine configuration allows maximum pressure of 200 psi, to detect a minimum pore size of 33 nm. This pressure is not high enough to cause change in microstructure of the BPs during the test, as discussed earlier in Section 2.1. It can be seen in Fig. 4b that normal distribution of pore diameter is observed in UBP, with pore diameters varying from 33 to 48 nm and average pore diameter being 38 nm. For CBPs, no pores were detected within the measurement resolution of the machine (minimum pore diameter of 33 nm). This indicates that pore diameters for the CBPs are <33 nm. This finding confirms and supports the SEM image analysis discussed earlier. Number of relatively large pore diamters (>40 nm) in UBP observed by CFP method is statistically small because CFP only measures the constricted diameter along the through-thickness path and thus it doesn’t consider many larger pore diameters situated within the same through-thickness path of the BPs. The CFP method is especially useful when accurate estimate of pores that contribute to the through-thickness transport (i.e. for filtration membrane applications) is needed. Further confirmation through density evaluation of the BPs by measuring their mass and volume was performed. The measured density of the UBP and CBP are 0.4 and 0.8 g/cm³, respectively. The results indicate that the pore size and density of the BP can be tailored depending on the desired end properties by fine tuning the process parameters such as the compressive force applied on the BP.

### 3.3. Tensile properties

In this section, tensile properties of both UBPs and CBPs are discussed. All strain measurements were evaluated by DIC method. The computation zone for the DIC covered approximately 90% of the rectangular sample area, leaving only small area near the grip zone (Fig. 5). The strain reported in this work is the average value of the strain in the computation zone.

The DIC result demonstrates the evolution of strain field of the sample as the load increases during the tensile test (Fig. 5). High variation of the strain profile of the BP shows that the randomly distributed, web-like MWCNSs in the BP generate non-uniform strain fields in the sample. The strain variation is due to the local MWCNS arrangement, such as the presence of fiber entanglement, wall sharing, branching and crosslinking. The DIC measurement offers a more accurate result since it can discard the strain effects at the grip zones, such as the ones caused by the clamping effect and mechanical fastenings. As shown in Fig. 6a, the strain measured by DIC is lower than the strain measured by UTM machine. Here the strain from the UTM machine is calculated by the displacement over the initial grip to grip distance.

The mechanical tests of the BPs with varying crosshead speed; (12, 0.2, 0.02) mm/min) were performed to examine the sensitivity of the BPs to strain-rate. It can be seen from Fig. 6b that the tensile properties of the BPs are insensitive to the strain-rate. For the subsequent tensile tests, we have used the crosshead speed of 0.02 mm/min to better capture the stress-strain behavior of the BPs.

Table 1 and Fig. 7 show the average value and standard deviation of Young’s modulus, tensile strength, strain to failure, toughness and Poisson’s ratio. Based on the published literatures on the mechanical properties of BPs, tensile strength of 2–94 MPa, tensile modulus of 2.1 MPa–3.84 GPa, and failure strain of 0.3–2% have been reported [3, 6,7,14,23,24,28,37,46–48]. The large difference in the measured properties originates from various parameters such as CNT type, CNT diameter and length, chemical treatment, processing condition, alignment, density, etc. In general, the currently studied BPs possess tensile properties within the range of typical randomly oriented BPs. However,
the reported values are much lower than those of individual CNTs due to the low inter-bundle junction interactions via van der Waals forces. The individual CNTs can have Young’s modulus as high as 1.2 TPa and a tensile strength of about 30–200 GPa [49,50].

Despite the fact that the CBPs have higher fiber density than that of the UBPs, the mechanical properties of both types of buckypapers are approximately the same. It can be inferred that the change in pore size and density of buckypaper due to compression doesn’t significantly alter the macroscopic mechanical response. This is because the applied compressive force doesn’t change the van der Waals’ interaction between random MWCNSs that contribute to the mechanical properties. The applied compression reduces the distance between MWCNS. Since the MWCNSs are randomly oriented, the effective increase of van der Waals’ interaction only occurs at the intersection points between adjacent MWCNS and thus one cannot see any improvement in macroscopic mechanical properties. The case could be different for aligned CNTs. The decrease in separation distance between aligned CNTs would lead to significant increase in van der Waals’ interaction since the van der Waals’ interaction between CNTs occur along the entire length of the CNTs.

We observed that the Poisson’s ratio of the BPs can have a positive or negative value (Fig. 8a). This was found in both UBP and CBP. If the instantaneous in-plane Poisson’s ratio is defined as

\[ \nu = \frac{\varepsilon_y}{\varepsilon_x} \]

where \( \varepsilon_x \) and \( \varepsilon_y \) are the strain in transverse and longitudinal directions, respectively, and \( \sigma \) is the stress in the longitudinal direction, one can see that the initial Poisson’s ratio can be positive or negative, and then reaches a stabilized value as the strain increases (Fig. 8b). Usually, solid materials experience transverse deformation with positive Poisson’s ratio. However, based on the published literatures, BP can also have negative Poisson’s ratio [51], or can change the Poisson’s ratio from negative to positive as the load increases [52,53]. The currently studied BPs are randomly oriented. However, local arrangement of MWCNS can lead to anisotropic response, allowing the BPs to initially have either positive or negative Poisson’s ratio. The realignment of MWCNS as the load increases leads to a stabilized positive Poisson’s ratio. If we only consider the stabilized value, one can see that UBP
and CBP possess average Poisson’s ratio of 0.13 and 0.14, respectively, as reported in Table 1 and Fig. 7.

We have also conducted quasi-static unloading-reloading tests considering several load increments for both UBP and CBP under the same strain-rate. It can be seen from Fig. 9 that the viscous effect results in different trajectories for loading and unloading, with the formation of the hysteresis loop. The viscoelastic-viscoplastic response of UBP and CBP under repetitive cyclic loading with increased amplitude is shown in Fig. 9a and b, respectively. Note that hysteresis area increases as the load level increases. One can also see irreversible strain as complete unloading doesn’t allow the sample to regain its original length. Increase of average separation distance between MWCNS and the reorientation of MWCNS occur during mechanical loading. The accumulation of plastic strain with cycling is believed to be due to the damage caused by the breakage of the MWCNS network in the form of relatively high inter-nanotubes separation distance in several localized areas of the sample.

### 3.4. Thermal and electrical properties

Fig. 10 shows that at room temperature, the thermal conductivity of UBP and CBP is 6.1 and 20 W/mK, respectively. The electrical conductivity of UBP is 118 S/cm while the CBP has $\sigma = 182$ S/cm. The current results demonstrate that the electrical and thermal conductivities are improved by increasing the BP’s density. The individual CNTs have very high electrical and thermal conductivities, which are $10^2$–$10^6$ S/cm [49] and 2000–6000 W/mK [54], respectively. The high disparity between the transport properties of our BPs and the individual CNTs is due to the discontinuity between the individual MWCNS and the low electrical and thermal conductance between MWCNS –

| Tensile properties of uncompressed and compressed buckypapers. The ± signs indicate standard deviation of the respective properties. |
|------------------|------------------|------------------|------------------|------------------|
| **Young’s modulus (GPa)** | **Maximum stress (MPa)** | **Strain to failure (%)** | **Toughness (×10^3 J m⁻³)** | **Poisson’s ratio** |
| UBP | 2.22 ± 0.22 | 11.02 ± 0.9 | 1.18 ± 0.27 | 110 ± 18 | 0.13 ± 0.014 |
| CBP | 2.15 ± 0.33 | 10.32 ± 0.83 | 1.23 ± 0.22 | 116 ± 29 | 0.14 ± 0.025 |

Fig. 7. Tensile properties of uncompressed and compressed buckypapers. Error bars indicate standard deviation of the respective properties.
MWCNS contacts. Compared with the thermal and electrical properties of other BPs, our BPs are within the range of the properties of BP with randomly aligned CNTs [14,29,31,55], but lower than those of the BP with aligned CNTs [21,29,55]. Generally, the thermal and electrical behavior of the BP depend on various parameters such as fabrication method, BP’s density and alignment, CNT type, treatment and dimension. Our results are within reasonable range of BP’s electrical and thermal properties. The use of high production rate of MWCNS synthesis for fabricating our BPs is of particular interest for industrial scale application.

4. Conclusion

Both UBPs and CBPs were successfully characterized in terms of their surface morphology, pore characteristics, thermal, electrical and mechanical properties. The currently studied BPs were synthesized from MWCNSs, that are branched, crosslinked and exhibit wall-sharing. Both BPs were fabricated by vacuum filtration process, with subsequent application of compressive force to obtain CBPs. It was shown from the morphological analysis by SEM that the MWCNSs were randomly oriented within the BPs. From the morphology, pore size distribution and density measurement analysis, it was found that the CBP has a more closely-packed fiber arrangement, lower porosity and higher density than those of the UBP. MWCNS entanglements, branches and wall-sharings were observed in the BPs, indicating a unique architecture of the currently studied BPs.

Improved thermal and electrical conductivities were found for CBP since higher density of CNT networks leads to more conductive pathways. However, mechanical properties of the CBPs and UBPs are approximately the same. The change in microstructure of buckypaper (pore size and density) due to compression doesn’t significantly alter the macroscopic mechanical properties. The van der Waals’ interaction between CNTs at nanoscale remains the main factor that contributes to the mechanical properties. Due to its mechanical stability and well defined pore characteristics, the UBP is suitable for separation membrane applications. On the other hand, the CBP offers a compact structure that can reduce penetration of the polymer matrix in composite materials, leading to a superior thermal and electrical conductivity while maintaining sufficient mechanical properties. These attributes are desirable for electronic applications such as for integrated circuit packaging and EMI shielding. The tunability of Poisson’s ratio of BPs (positive or negative) by carefully controlling the alignment or orientation of nanotubes is of particular interest for further investigation.

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Fig. 9. Unloading-reloading test of: a) uncompressed and b) compressed buckypapers.

Fig. 10. Electrical and thermal conductivities of uncompressed and compressed buckypapers.