Electrical Conductivity Model Evaluation of Carbon Fiber Filled Liquid Crystal Polymer Composites

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ABSTRACT: Electrically conductive resins may have applications as fuel cell bipolar plates. The current trend in this technology is a thermosetting polymer as the matrix containing high concentrations of various types of fillers. These fillers are carbon based and electrically conductive powders, particles, or fibers. In this study, we utilized two composite formulations of polyacrylonitrile fibers (Fortafil 243 and Panex 30) in a liquid crystal polymer (Vectra A950RX) with increasing concentrations. Electrical conductivity tests were performed and modified Mamunya and additive models were applied to the experimental data. These models fit the entire range of data for each composite tested. Four alternate models were also produced: linear, quadratic, exponential, and geometric, with a restricted range of electrical conductivity data greater than 10^{-2} S/cm. The exponential and the geometric resulted in the best fits over this restricted data range. These particular models may allow researchers to extrapolate beyond the maximum filler concentrations studied here. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 2456–2462, 2007

Key words: liquid crystalline polymers (LCP); fillers; modeling; composites

INTRODUCTION

Composite materials have gained recognition for various applications due to their ease of manufacturing, low cost, and uniform reproducibility.[1] Our research group has been investigating polymer-based composites as bipolar plates in the fuel cell. A typical high power fuel cell can contain hundreds of electrically conductive bipolar plates. Currently, 70–90 wt % of a single type of graphite powder in a thermosetting resin is typically being used for bipolar plates.[2] Current research initiatives and manufacturing practices have investigated numerous matrix and carbon filler materials in the production of bipolar plates for fuel cells.

Polymers are naturally insulating materials. Typically polymers exhibit electrical conductivities in the range of 10^{-14}–10^{-17} S/cm. Thermosetting polymers are often being used for the matrix material. Thermosetting polymers have disadvantages as compared with thermoplastic polymers such as longer molding times and the inability to be remelted. Metallic plates have also been utilized but these have several disadvantages relative to conductive resins including higher cost and weight and less corrosion resistance. Thus, thermoplastic polymer resins have been investigated for the use as bipolar plates in fuel cell applications.

The electrical conductivity of a polymer can be enhanced by the addition of electrically conductive carbon fillers or metals.[3–15] Electrical conductivities of typical conductive carbon fillers range from 10^2 to 10^5 S/cm. The combination of a polymer and electrically conductive filler(s) may result in an electrically conductive resin effective for fuel cell bipolar plate applications. It is noted that the current department of energy target value for the electrical conductivity of fuel cell bipolar plates is 100 S/cm.[16]

Several types of fillers have been utilized in the research and development of electrically conductive composites such as powders, particles, and fibers. In this study two electrically conductive carbon fibers were added to Vectra A950RX in increasing concentrations. Each sample formulation was prepared and the electrical conductivity was measured. The fillers used in this project are polyacrylonitrile-based carbon fibers. Only single fillers were added to the polymer in this experiment. There were no combinations of fillers studied.

A correlation exists between the electrical conductivity of the various polymer composites and the carbon filler concentration of the composite. At low filler loadings the electrical conductivity is equivalent to the electrical conductivity of the insulating

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TABLE I
Properties of Ticona's Vectra A950RX[21]

Melting point	280°C
Tensile modulus (1 mm/min)	10.6 GPa
Tensile strength at break (5 mm/min)	182 MPa
Tensile strain at break (5 mm/min)	3.4%
Flexural modulus at 23°C	9.1 GPa
Notched Izod impact strength at 23°C	95 KJ/m^2
Density at 23°C	1.40 g/cc
Volumetric electrical conductivity at 23°C	10^{-15} S/cm
Surface electrical conductivity	10^{-14} S
Thermal conductivity at 23°C	0.2 W/mK (approx.)
Humidity absorption (23°C/50%RH)	0.03 wt %
Mold shrinkage-parallel	0.0%
Mold shrinkage-normal	0.7%
Coefficient of linear thermal expansion-parallel	0.04×10^{-4}/°C
Coefficient of linear thermal expansion-normal	0.38×10^{-4}/°C

polymer. As the filler concentration increases there is a critical volume fraction at which the electrical conductivity increases by several orders of magnitude. This is called the percolation threshold. Upon further increase of the carbon filler content, the electrical conductivity only shows a slight increase until a plateau is observed.[17] This relationship has been verified through the thermodynamic model developed by Mamunya, et al.[18,19] and additive model by Clingerman, et al.[20] These electrical conductivity models are a function of the surface energies of both the polymer and filler, the critical volume fraction (percolation threshold), and the aspect ratio (length/diameter) of the filler. In this study, we produced composite samples and measured the electrical conductivity in order to apply the Mamunya and additive models as well as several new models (linear, quadratic, exponential, and geometric) for our carbon fiber composites.

MATERIALS AND EXPERIMENTAL METHODS

Materials

This article focuses on the comparison of two electrically conductive carbon-based fibers within a thermoplastic polymer. The polymer is the Vectra liquid crystal polymer (LCP), manufactured by Ticona. Vectra A950RX is a highly ordered thermoplastic copolymer consisting of 73 mol % hydroxybenzoic acid and 27 mol % hydroxynaphtholic acid. This LCP has the desired properties for bipolar plates, namely high dimensional stability up to temperatures of 250°C, extremely short molding times, exceptional dimensional reproducibility, is chemical resistant in the acidic environments present in a fuel cell, and has a low hydrogen permeation rate.[21,22] In addition, Vectra can be molded into thin walls needed to reduce the volume and weight of a fuel cell assembly. The volumetric electrical conductivity of the Vectra polymer is 10^{-15} S/cm.[21] Table I shows the properties of this liquid crystal polymer.

Two electrically conductive polyacronitrile (PAN) based carbon fibers were used in this study. The first carbon fiber used in this study was Fortafil 243, sold by Toho Tenax America, Inc. Fortafil 243 is a 3.2 mm chopped and pelletized carbon fiber that is often used to improve the electrical and thermal conductivity and the tensile strength of a resin. Fortafil 243 was surface treated and then formed into pellets by the manufacturer. A proprietary polymer (sizing) is used as a binder for the pellets that also promotes adhesion with the matrix. Table II shows the properties of this carbon fiber, which is 95.0 wt % carbon.[23]

Table III shows the properties of Zoltek's Panex 30 milled 150 μm long high purity carbon fiber. This carbon fiber is electrochemically surface treated but not sized. Panex 30 is produced by a high temperature batch graphitization process that produces fibers that are 99.5 wt % carbon.[24]

The concentrations (shown in wt % and the corresponding vol %) for all of the single filler composites tested in this research are shown in Table IV. Because of increases in resin viscosity, the maximum amount of fiber that could be extruded and injection molded into test specimens were 50 wt % for both Fortafil 243 and Panex 30.

Test specimen fabrication

For this entire project, the fibers were used as they were received. Vectra A950RX was dried in an indirectly heated dehumidifying drying oven at 150°C, and then stored in moisture barrier bags.

The extruder used was an American Leistritz Extruder Corporation Model ZSE 27. This extruder has a 27-mm co-rotating intermeshing twin screw with 10 zones and a length/diameter ratio of 40. The

TABLE II
Properties of Fortafil 243 Carbon Fiber[23]

Carbon content	95 wt %
Electrical conductivity	600 S/cm
Thermal conductivity	20 W/mK (axial direction)
Tensile strength	3800 MPa
Tensile modulus	227 GPa
Specific gravity	1.74 g/cc
Fiber diameter	7.3 μm
Fiber shape	Round
Fiber mean length	3.2 mm (entire range is 2.3 mm–4.1mm)
Binder content	2.6 wt % proprietary polymer that adheres pellet together and promotes adhesion with nylon matrix
Bulk density	356 g/L

TABLE III
Properties of Panex 30 Milled High Purity Carbon Fiber[24]

Carbon content	99.5 wt %
Electrical conductivity	700 S/cm
Thermal conductivity	22 W/mK (axial direction, approximate)
Tensile strength	3600 MPa
Tensile modulus	207 GPa
Specific gravity	1.75 g/cc
Fiber diameter	7.4 μm
Fiber shape	Round
Fiber mean length	150 μm
Bulk density	445 g/L

screw design is shown elsewhere.[25] The design was chosen to obtain a minimum amount of fiber degradation to allow for maximum electrical conductivity, while still dispersing the fillers well in the polymers. The polymer pellets (Vectra) were introduced in Zone 1. The side stuffer, utilized to introduce all the fibers into the polymer melt, was located at Zone 5. Two Schenck AccuRate gravimetric feeders were used to accurately control the amount of each material added to the extruder.

After passing through the extruder, the polymer strands (3 mm in diameter) entered a water bath and then a pelletizer that produced nominally 3-mm long pellets. After compounding, the pelletized composite resin was dried and then stored in moisture barrier bags prior to injection molding.

A Niigata injection molding machine, model $NE85UA_4$, was used to produce test specimens. This machine has a 40-mm diameter single screw with a length/diameter ratio of 18. The lengths of the feed, compression, and metering sections of the single screw are 396, 180, and 144 mm, respectively. A four cavity mold was used to produce 3.2-mm thick ASTM Type I tensile bars (end gated) and 6.4-cm diameter disks (end-gated). The electrical conductivity values of all formulations were determined. Before the electrical conductivity tests were performed, the samples were conditioned at 23°C and 50% relative humidity for 88 h.[26]

In plane electrical conductivity test method

The volumetric in-plane (also called longitudinal) electrical conductivity was measured on all samples with an electrical conductivity $>10^{-4}$ S/cm. Test specimens were cut from the center gauge portion of a tensile bar, were surface ground on all sides, and then cut into sticks 2-mm wide by 2-mm thick by 25.4-mm long. Typically for each formulation, a total of six specimens were cut from a single tensile bar, and three tensile bars were typically used to obtain a total of 18 test specimens. These samples were then tested using the four-probe technique. This technique measures conductivity by applying a constant current (typically 5–10 mA) and measuring the voltage drop over the center 6 mm of the sample. A Keithley 224 Programmable Current Source and Keithley 182 Digital Sensitive Voltmeter were used.[27] Equation (1) below is then used to calculate the electrical conductivity.

$$\mathrm{EC} = \frac{(i)(L)}{(\Delta V)(w)(t)} \tag{1}$$

where EC, Electrical Conductivity, S/cm; ΔV, Voltage drop over center 0.6 cm of sample, volts; w, sample width, cm; t, sample thickness, cm; i, current, amps; L, length over which ΔV is measured (0.6 cm).

Through plane electrical conductivity test method

For samples with an electrical conductivity $<10^{-4}$ S/cm, through-plane (also called transverse), volumetric electrical conductivity test was conducted. In this method, a constant voltage (typically 100 V) was applied to the as-molded test specimen, and the resistivity was measured according to ASTM D257 using a Keithley 6517A Electrometer/High Resistance Meter and an 8009 Resistivity Test Fixture.[28] The Keithley 6524 High Resistance Measurement Software was used to automate the conductivity measurement. For each formulation, a minimum of six specimens were tested. Each test specimen was an injection molded disk that was 6.4 cm in diameter and 3.2 mm thick.

Fiber length and orientation test method

To determine the length of the carbon fiber in the molded test specimens, diethylenetriamine was used to dissolve the matrix. The fibers were then dispersed onto a glass slide and viewed using an Olym-

TABLE IV
Single Fiber Loading Levels

Fiber wt %	Fortafil vol %	Panex vol %
5.0	4.1	4.0
7.5	6.1	6.1
10.0	8.2	8.2
15.0	12.4	12.4
20.0	16.8	16.7
25.0	21.2	21.1
30.0	25.5	N/A
35.0	30.2	30.1
40.0	34.9	34.8
45.0	39.7	39.9
50.0	44.6	44.4

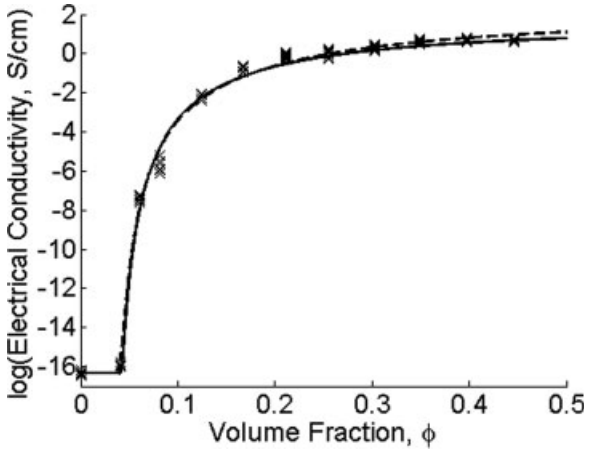

Figure 1 Electrical Conductivity Results for Fortafil 243. The symbols represent the data points, the solid line represents the Mamunya model, and the dashed line represents the additive model.

pus SZH10 optical microscope with an Optronics Engineering LX-750 video camera. Additional details of this method are shown elsewhere.[29]

To determine the orientation of the carbon fiber, a polished composite sample was viewed using an optical microscope. For each formulation, an in-plane electrical conductivity sample was cast in epoxy so that the direction of flow induced during the injection molding process, which was also the direction of electrical conductivity measurement (lengthwise direction), would be viewed. For the through-plane conductivity samples, the center portion was cut out of a disk and set in epoxy such that the through the sample thickness (3.2 mm) face could be viewed. The samples were then polished with a Buehler Ecomet 4 polishing wheel fitted with an Automet 2 polishing head. The polished sample was viewed using an Olympus BX60 reflected light microscope at a magnification of 100× or 200×. The images were then processed using Adobe Photoshop 5.0 and the Image Processing Tool Kit version 3.0. For each formulation, the orientation was determined by viewing typically 1000–2000 particles.

RESULTS

Fiber length and orientation results

For the molded test specimens containing both carbon fibers, the fiber length was typically 70 μm. The corresponding fiber aspect ratio (length/diameter) was 9. For the through-plane conductivity samples, the fibers are primarily oriented transverse to the conductivity measurement direction. For the in-plane electrical conductivity samples the fibers are primarily oriented in the electrical conductivity measurement direction.

Electrical conductivity results

Figure 1 show the logarithm of electrical conductivity (S/cm) as a function of the volume fraction (ϕ) for the Fortafil carbon fiber. In each figure, the entire collection of experimental data is shown. The electrical conductivity curve generated by the data is characteristic with previous work.[7,17] According to Figure 1, the Fortafil fiber is effective at increasing the electrical conductivity of a polymer composite at lower loadings. This observation is apparent from the low value of the percolation threshold of 4.1 vol %. At the maximum filler loading of 44.6 vol %, the resultant electrical conductivity is 4.7 S/cm.

The Panex fiber, shown in Figure 2, is not as effective as Fortafil at increasing the electrical conductivity in the polymer composite. This is illustrated by the comparison of the values of the percolation threshold and the electrical conductivity at the maximum filler concentration with those of the Fortafil carbon fiber. The value of the percolation threshold is 12.4 vol % and at the maximum loading of 44.4 vol %, the electrical conductivity is 1.9 S/cm.

Electrical conductivity modeling results

For the electrical modeling portion of the study, our research group focused our attention on the simplified thermodynamic models developed by Keith et al.[30] These simplified models are modified version of the thermodynamic model created by Mamunya et al.[18,19] and the additive model of Clingerman et al.[20] The modified Mamunya model, which calculates the electrical conductivity, σ (S/cm), of the polymer composite and is described by eqs. (2) and (3):

$$\log(\sigma) = \log(\sigma_p) + [\log(\sigma_f) - \log(\sigma_p)]\left(\frac{\phi - \phi_c}{F - \phi_c}\right)^k \quad (2)$$

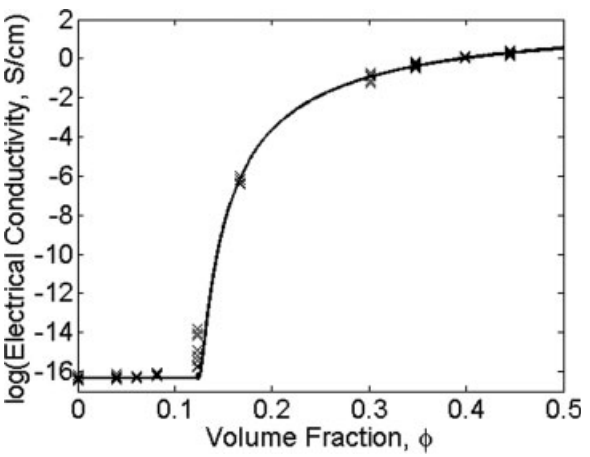

Figure 2 Electrical Conductivity Results for Panex 30. The symbols represent the data points, the solid line represents the Mamunya model, and the dashed line represents the additive model.

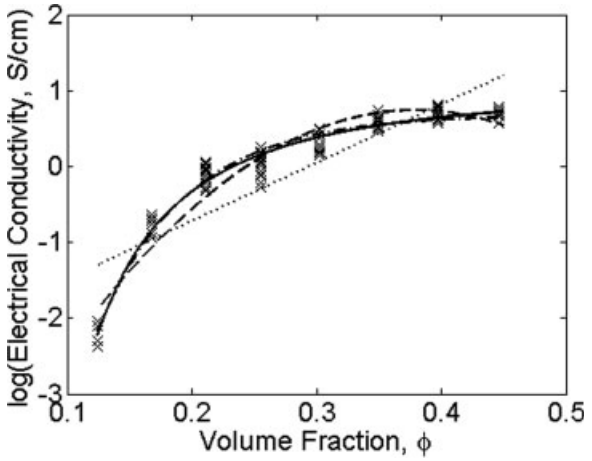

Figure 3 Alternate Electrical Conductivity Models for Fortafil 243. The symbols represent the data points, the dotted line represents the linear model, the dashed line represents the quadratic model, the dash-dot line represents the exponential model, and the solid line represents the geometric model.

$$k = \frac{K\phi_c}{(\phi - \phi_c)^n} \tag{3}$$

where σ_p is the electrical conductivity of the pure polymer (S/cm). It is assumed that the composite electrical conductivity is equal to σ_p for volume fractions less than or equal to the percolation threshold ϕ_c. At the maximum filler loading, F, the electrical conductivity is σ_f (S/cm). The two model parameters are K and n. For all formulations, $\sigma_p = 4.57 \times 10^{-17}$ S/cm. The results for the Fortafil/Vectra composites are shown in Figure 1, with $\sigma_f = 4.73$ S/cm, $F = 0.45$, $\phi_c = 0.0406$, $K = 0.89$ (dimensionless), and $n = 0.48$ (dimensionless). The results for the Panex/Vectra composites are shown in Figure 2, with $\sigma_f = 1.91$ S/cm, $F = 0.44$, $\phi_c = 0.124$, $K = 0.46$ (dimensionless), and $n = 0.46$ (dimensionless).

Equation (4) is the modified additive model. It also predicts the electrical conductivity of polymer composite and is dependent on four model parameters H, G, n, and E.

$$\log(\sigma) = \log(\sigma_p) + H(\phi - \phi_c)^{\frac{G}{(\phi-\phi_c)^n}} + E \tag{4}$$

The results of the modified additive model for both the Fortafil 243 and Panex 30 carbon fiber composites are also shown in Figures 1 and 2, respectively. For all formulations, $\sigma_p = 4.57 \times 10^{-17}$ S/cm. The model parameters for the Fortafil/Vectra composites are $H = 17.13$ S/cm, $G = 3.46 \times 10^{-2}$ (dimensionless), $n = 0.47$ (dimensionless), and $E = 0.97$ S/cm. The model parameters for the Panex/Vectra composites are $H = 17.71$ S/cm, $G = 3.65 \times 10^{-2}$ (dimensionless), $n = 0.51$ (dimensionless), and $E = 0.22$ S/cm. As a final note, both the Mamunya and additive models show good agreement over the entire range of volume fractions for both composites, producing nearly identical results.

In addition to the modified models, we developed new models which may be more accurate for interpolation over a limited range of electrical conductivities. Because of the applicability of composites for fuel cell bipolar plates, the alternative models are restricted to electrical conductivity values greater than 10^{-2} S/cm, resulting in eight data points for the Fortafil composites and four data points for the Panex 30 composites. These models are given by the following linear, quadratic, exponential, and geometric equations:

$$\log \sigma = a\phi + b \tag{5}$$

$$\log \sigma = c\phi^2 + d\phi + e \tag{6}$$

$$\log \sigma = f + g\exp(-h\phi) \tag{7}$$

$$\log \sigma = m + j\phi^p \tag{8}$$

The graphical results are shown in Figure 3 for Fortafil/Vectra composites (for $\phi > 0.12$) and Figure 4 for the Panex/Vectra composites (for $\phi > 0.30$). The model constants from Eqs. (5)–(8) are listed in Table V. For each composite the exponential and geometric models produced a better fit with the experimental data. We note that for Fortafil/Vectra composites, the exponential and geometric models have a residual $\sim 10^1$ times smaller than that measured for the Mamunya and additive models. Furthermore, for Panex/Vectra composites the exponential

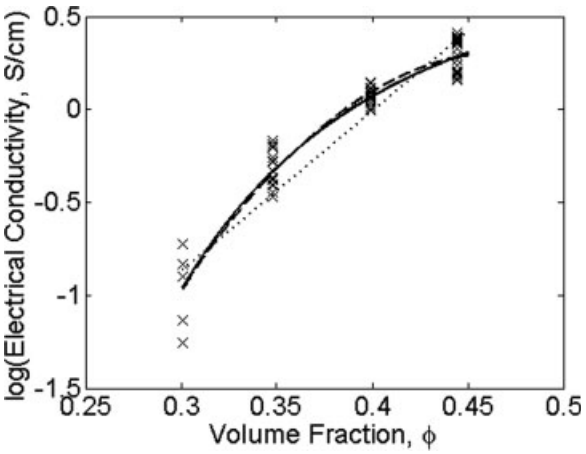

Figure 4 Alternate Electrical Conductivity Models for Panex 30. The symbols represent the data points, the dotted line represents the linear model, the dashed line represents the quadratic model, the dash-dot line represents the exponential model, and the solid line represents the geometric model.

TABLE V
Parameters for Alternate Electrical Conductivity Models

Parameter	Fortafil 243	Panex 30
a	7.67	8.63
b	−2.25	−3.46
c	−40.58	−45.34
d	30.77	42.43
e	−5.09	−9.62
f	0.68	0.61
g	−15.56	−42.13
h	13.70	10.91
m	1.03	0.91
j	-7.15×10^{-2}	-6.4×10^{-2}
p	−1.83	−2.81

and geometric models have a residual $\sim 10^2$ times smaller than that measured for the Mamunya and additive models. Thus, these models are more accurate for interpolating data for fuel cell bipolar plate applications. In addition, the models may find application for data extrapolation to higher filler loading levels.

Comparison of fiber composites with other carbon filled composites

In a comparison to previous work done by our research group the percolation threshold of the Fortafil/Vectra composites ($\sim$ 4 vol %) is comparable to that of Ketjenblack (a form of carbon black)/Vectra composites ($\sim$ 4 vol %).[25,31] However, the Ketjenblack composites has a significantly higher viscosity and lower electrical conductivity than the Fortafil composites,[25,31] indicating Fortafil may be a better choice for a bipolar plate containing multiple carbon fillers. Furthermore, the percolation threshold of Panex/Vectra composites ($\sim$ 12 vol %) is comparable to that of Thermocarb (a synthetic graphite)/Vectra composites ($\sim$ 15 vol %).[25,31] Thermocarb's outstanding effect on thermal conductivity enhancement[32] may make it suitable for a bipolar plate containing multiple carbon fillers.

CONCLUSION

In this study two PAN based carbon fibers, Fortafil 243 and Panex 30, were incorporated in a liquid crystal polymer, Vectra A950RX with increasing filler concentrations. The electrical conductivity of each sample composite formulation was obtained and the experimental data was fit to modified versions of the Mamunya and additive models. Both the modified Mamunya and additive models showed good agreement with the data for both composites.

The modified models were fit to the experimental data from the percolation threshold value to the maximum loading concentration for each composite. The Fortafil 243 composite has a percolation threshold, ϕ_c of 4.1 vol % and the maximum filler concentration tested was 44.6 vol %, with an electrical conductivity 4.7 S/cm. The Panex 30 composite has a ϕ_c of 12.4 vol % and the electrical conductivity at the maximum filler concentration of 44.4 vol % is 1.9 S/cm.

Alternate models were analyzed for the purpose of restricting the experimental data range to electrical conductivity values greater than or equal to 10^{-2} S/cm. The four models developed were the linear, quadratic, exponential, and the geometric. The exponential and geometric showed good agreement with both composites and outperformed the Mamunya and additive models over a data range that is appropriate for fuel cell bipolar plate applications. As a final note, the alternate models may be more suited to data extrapolation beyond the maximum filler concentration than the modified Mamunya and additive models.

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