Thermal and Electrical Conductivity of Carbon–Filled Liquid Crystal Polymer Composites

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ABSTRACT: The thermal and electrical conductivity of resins can be increased by adding conductive carbon fillers. One emerging market for thermally and electrically conductive resins is for bipolar plates for use in fuel cells. In this study, varying amounts of five different types of carbon, one carbon black, two synthetic graphites, one natural flake graphite, and one calcined needle coke, were added to Vectra A950RX Liquid Crystal Polymer. The resulting composites containing only one type of filler were then tested for thermal and electrical conductivity. The objective of this work was to determine which carbon filler produced a composite with the highest thermal and electrical conductivity. The results showed that composites containing Thermocarb TC-300 synthetic graphite particles had the highest thermal and electrical conductivity. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 99: 1552–1558, 2006

Key words: composites; thermal properties; thermoplastics; electrical conductivity

INTRODUCTION

Most polymer resins are thermally and electrically insulating. Increasing the thermal and electrical conductivity of these resins allows them to be used in other applications. One emerging market for thermally and electrically conductive resins is for bipolar plates for use in fuel cells. The bipolar plate separates one cell from the next, with this plate carrying hydrogen gas on one side and air (oxygen) on the other side. Bipolar plates require high thermal and electrical conductivity (to conduct heat and to minimize ohmic losses), low gas permeability, and good dimensional stability.

Typical thermal conductivity values for some common materials are 0.2–0.3 for polymers, 234 for aluminum, 400 for copper, and 600 for graphite (all values in W/mK). Electrical resistivity (1/electrical conductivity) values in Ω-cm for various materials are typically 10^{14}–10^{17} for polymers, 10^{-2} for carbon black, 10^{-2}–10^{-5} for high purity synthetic graphite, and 10^{-6} for metals such as aluminum and copper. One approach to improve the electrical and thermal conductivity of a polymer is through the addition of a conductive filler material, such as carbon and metal.[1–15] Currently, a single type of graphite powder is typically used in thermosetting resins (often a vinyl ester) to produce a thermally and electrically conductive bipolar plate material.[16–19] Thermosetting resins cannot be remelted.

In this work, researchers performed compounding runs followed by injection molding of carbon filled Vectra A950RX. Vectra is a thermoplastic that can be remelted and used again. One carbon black, two synthetic graphites, one natural flake graphite, and one calcined needle coke were studied. Characterization tests for materials included volumetric electrical resistivity, thermal conductivity, and optical microscopy to determine the filler orientation. The goal of this project was to determine which carbon filler produced a composite with the highest thermal and electrical conductivity.

Materials and experimental methods

Materials

The matrix used for this project was Ticona's Vectra A950RX Liquid Crystal Polymer (LCP), which is a highly ordered thermoplastic copolymer consisting of

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TABLE I
Properties of Ticona's Vectra A9SORX (20)

Melting point	280°C
Tensile modulus (1mm/min)	10.6 GPa
Tensile stress at break (5mm/min)	1 82 MPa
Tensile strain at break (5mm/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 resistivity at 23°C	10^{15} Ω-cm
Surface electrical resistivity	10^{14} Ω
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

73 mol % hydroxybenzoic acid (HBA) and 23 mol % hydroxynaphtholic acid (HNA). This LCP has the properties needed for bipolar plates, namely high dimensional stability up to a temperature of 250°C, extremely short molding times (often 5–10 s), exceptional dimensional reproducibility, chemically resistant in acidic environments present in a fuel cell, and a low hydrogen gas permeation rate.[20,21] The properties of this polymer are shown in Table I.[20]

The first filler used in this study was Ketjenblack EC-600 JD, which was primarily used to improve composite electrical conductivity. This is an electrically conductive carbon black available from Akzo Nobel, Inc. The highly branched, high surface area carbon black structure allows it to contact a large amount of polymer, which results in improved electrical conductivity at low carbon black concentrations (often 5–7 wt %). The properties of Ketjenblack EC-600 JD are given in Table II.[22] The carbon black is in the form of pellets that are 100 μm–2 mm in size and, upon mixing into a polymer, easily separate into primary aggregates 30–100 nm long.[22]

Table III shows the properties of the synthetic graphites, natural flake graphite, and calcined coke used.[23] Two different synthetic graphites from Asbury Carbons were studied: Thermocarb TC-300 (previously sold by Conoco) and grade 4012. These synthetic graphites have excellent thermal and electrical conductivity. Grade 4012 has been successfully used in thermosetting vinyl ester resins for bipolar plates in the past.[16] Thermocarb TC-300 is produced by nonconventional thermal processing and contains less impurities than grade 4012.[23] One natural flake graphite grade 3160 from Asbury Carbons was used. This flake graphite is a naturally occurring mineral. One Asbury Carbons' calcined needle coke F108A was used as a filler. Calcined needle coke is produced from a thermally treated highly aromatic petroleum feedstock and is typically calcined at ~1350°C. Although calcined needle coke is a conductive carbon, it does not have a graphitic crystalline structure. Natural flake graphite and synthetic graphite grade 4012 and Thermocarb TC-300 do have a graphitic crystalline structure, which increases their thermal and electrical conductivity above that of calcined petroleum coke (an amorphous carbon material).[7,13,23]

Thermal and electrical conductivity were measured on composites containing varying amounts of these carbon fillers in Vectra A950RX. The concentrations (shown in wt % and the corresponding vol %) for these single filler composites are shown in Table IV. Prior work in nylon 6,6 and polycarbonate has shown that the concentrations selected for these fillers will yield highly thermally and electrically conductive resins.[24–27] For bipolar plate applications, typically 60–70 wt % graphite is used.[16] Often 5–7 wt % of Ketjenblack EC-600 JD is added to a polymer to dramatically improve the composite electrical conductivity. The maximum amount of Ketjenblack EC-600 JD that could be extruded and injection molded into test specimens was 15 wt %. At higher concentrations of this carbon black, the material is too viscous. To the authors' knowledge, this is the first time in the open literature that these fillers have been used in Vectra A950RX to produce a thermally and electrically conductive resin.

Test specimen fabrication

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

The extruder used was an American Leistritz Extruder Corp. Model ZSE 27. This extruder has a 27 mm corotating intermeshing twin screw with 10 zones and a length/diameter ratio of 40. Figure 1 shows the screw design. It was chosen to allow a large concentration of filler to mix with the matrix material and to allow maximum possible conductivity. The Vectra polymer pellets were introduced in Zone 1. A side

TABLE II
Properties of Akzo Nobel Ketjenblack EC-600 JD (22)

Electrical resistivity	0.01–0.1 Ω-cm
Aggregate size	30–100 nm
Specific gravity	1.8 g/cm^3
Apparent bulk density	100–120 kg/m^3
Ash content, max	0.1 wt %
Moisture, max	0.5 wt %
BET surface area	1250 m^2/g
Pore volume	480–510 cm^3/100 g

TABLE III
Properties of Graphite and Calcined Coke (23)

Filler	Thermocarb synthetic graphite	4012 synthetic graphite	3160 natural flake graphite	Calcined needle coke F108A
Carbon content (wt %)	99.91	99.67	99.30	99.10
Ash (wt %)	<0.1	<0.5	0.7	0.3
Sulfur (wt %)	0.004	<0.1	<0.1	0.5
Density (g/cc)	2.24	2.24	2.24	2.1
Thermal conductivity at 23°C (W/mK)	600 in "a" crystallographic direction	600 (approx) in "a" crystallographic direction	600 (approx) in "a" crystallographic direction	10–20 (approx) along axial direction
Electrical resistivity of bulk carbon powder at 150 psi, 23°C, parallel to pressing axis (Ω-cm)	0.020	0.021	0.046	0.093
Particle shape	acicular	Acicular	Flake	acicular
Particle aspect ratio	1.7	1.7	4.8	2.3
Sieve analysis (wt %)				
+600 μm	0.19	0	0	0.13
+500 μm	0.36	0	0	—
+425 μm	—	0	0	4.16
+300 μm	5.24	0	0	19.05
+2l2 μm	12.04	0	0	42.84
+180 μm	8.25	0.22	1.9	17.56
+150 μm	12.44	0.86	9.7	10.64
−150 μm	—	—	—	5.62
+75 μm	34.89	71.65	50.4	—
+44 μm	16.17	24.43	21.6	—
−44 μm	10.42	2.82	16.4	—

stuffer located at Zone 5 was used to introduce the carbon fillers into the polymer melt. 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 again and then stored in moisture barrier bags prior to injection molding.

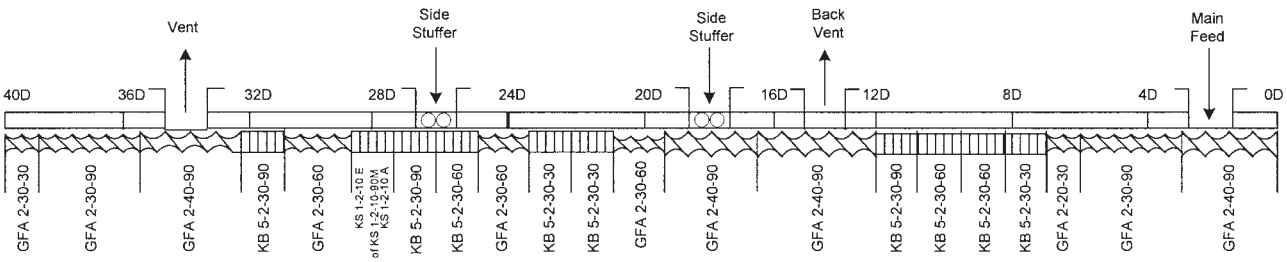
A Niigata injection molding machine, model $NE85UA_4$, was used to produce test specimens. This machine has a 40 mm diameter single screw with a length/diameter ratio of 18. The lengths of the feed, compression, and metering sections of the single screw are 396, 180, and 144 mm, respectively. The temperature profile typically used was 280°C in Zone 1 (nearest feed hopper), 307°C in Zones 2 and 3, and 315°C in Zone 4. 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 thermal and electrical conductivity of all formulations were determined. Prior to conducting any tests, the samples were all conditioned at 23°C and 50% RH for 88 h and then tested.[28]

Through-plane thermal conductivity test method

The through-plane thermal conductivity of a 3.2 mm thick, 5 cm diameter disc-shaped test specimen was measured at 55°C using a Holometrix Model TCA-300 Thermal Conductivity Analyzer, according to the

TABLE IV
Single Filler Loading Levels in Vectra A95ORX

Filler	Filler concentrations	
	Wt %	Vol %
Ketjenblack EC-600 JD	2.5, 4.0, 5.0, 6.0, 7.5, 10.0, 15.0	1.9, 3.1, 3.9, 4.7, 6.0, 8.0, 12.1
Thermocarb TC-300 synthetic graphite	40.0, 60.0, 70.0	29.3, 48.4, 59.3
4012 synthetic graphite	40.0, 60.0, 70.0	29.3, 48.4, 59.3
3160 natural flake graphite	40.0, 60.0, 70.0	29.3, 48.4, 59.3
Calcined needle coke F108A	40.0, 60.0, 70.0	30.8, 50.2, 61.4



For Screw Type Elements
GFA-d-ee-ff
G = co-rotating
F = conveying
A = Free-Meshing
d = number of threads
ee = pitch (length in millimeters for one complete rotation)

ff = length of screw elements in millimeters

Kneading disks
KBj-d-kk-ll
KB = kneading block
j = number of kneading segments
d = number of threads
k = length of kneading block in millimeters
l = twisting angle of the individual kneading segments

Kneading disks
KS1-d-hh-i
KS1 = Kneading disc
d = number of threads
h = length of kneading disc in millimeters
i = A for initial disc and E for end disc

Zones
0D to 4D is Zone 1 (water cooled, not heated)
4D to 8D is Zone 2 and Heating Zone 1
8D to 12D is Zone 3 and Heating Zone 2
12D to 16D is Zone 4 and Heating Zone 3
16D to 20D is Zone 5 and Heating Zone 4
20D to 24D is Zone 6 and Heating Zone 5
24D to 28D is Zone 7 and Heating Zone 6
28D to 32D is Zone 8 and Heating Zone 7
32D to 36D is Zone 9 and Heating Zone 8
36D to 40D is Zone 10 and Heating Zone 9
Nozzle is Heating Zone 10

Figure 1 Extruder screw design.

ASTM F433 guarded heat flow meter method.[29] For each formulation, four samples were tested.

Through-plane electrical resistivity test method

For samples with an electrical resistivity $>10^4$ Ω-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 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.[30] 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.

In-plane electrical resistivity test method

The volumetric in-plane (also called longitudinal) electrical resistivity was measured on all samples with an electrical resistivity $<10^4$ Ω-cm. Test specimens 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.[31] These samples were then tested using the four probe technique. This technique measures resistivity by applying a constant current (typically 5–10 mA) and measuring the voltage drop over the center 6 mm of the sample.[8] A Keithley 224 Programmable Current Source and Keithley 182 Digital Sensitive Voltmeter were used. Equation (1) below is then used to calculate the electrical resistivity.

$$ER = \frac{\Delta V w t}{i L} \tag{1}$$

where ER is the electrical resistivity in Ω-cm, ΔV is the voltage drop over center 0.6 cm of sample in volts, w is the sample width in cm, t is the sample thickness in cm, i is the current in amps, and L is the length over which ΔV is measured (0.6 cm)

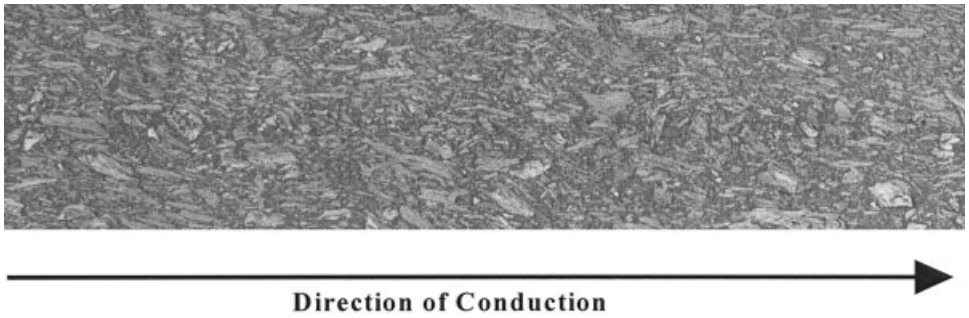


Figure 2 Optical micrograph by reflected light of an in-plane electrical resistivity sample containing 60 wt % thermocarb TC-300 synthetic graphite in Vectra A950RX at ×200 magnification.

Filler orientation test method

To determine the orientation of the carbon fillers, a polished composite sample was viewed using an optical microscope. Because of the small size of the carbon black (aggregates 30–100 nm in size), the orientation of only the synthetic graphite, natural flake graphite, and calcined coke particles were determined. For each formulation, an in-plane electrical resistivity sample was cast in epoxy so that the direction of flow induced during the injection molding process, which was also the direction of ER measurement (lengthwise direction), would be viewed. For the through-plane thermal 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 and 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

Filler orientation results

As discussed previously, the filler orientation angle was measured by optical microscopy. The angle of interest was the deviation of the filler away from the direction of conductivity measurement. All of the angles will be between 0° and 90°. An angle of 0° signifies that the fillers are aligned parallel to the measurement direction. An angle of 90° means that the fillers are perpendicular (transverse) to the measurement direction.

Figure 2 shows a photomicrograph of an in-plane electrical resistivity sample containing 60 wt % Thermocarb TC-300 Synthetic Graphite in Vectra A950RX. This figure clearly shows that most of the fillers are oriented in the direction of electrical conductivity measurement (mean orientation angle was 24°). This was the case for all the samples in this study and it agrees with prior work.[31]

Figure 3 shows a photomicrograph of a through-plane thermal conductivity sample containing 40 wt % calcined needle coke F108A in Vectra A950RX. This figure clearly shows that most of the fillers are oriented transverse to the thermal conductivity measurement direction (mean orientation angle was 52°). This was the case for all the samples in this study and it agrees with prior work.[32]

Thermal conductivity results

Figure 4 shows the mean through-plane thermal conductivity as function of filler volume fraction. These formulations correspond to those shown in Table IV. The mean thermal conductivity of the pure Vectra A950RX was 0.22 W/mK. The standard deviation was typically less than 5% of the mean.

Several observations can be made from the results shown in Figure 4. First, the composites containing carbon black had a lower thermal conductivity as compared to the others. Second, the composites containing both synthetic graphites and the natural flake graphite had similar thermal conductivity at 40 and 60 wt % (29.3 and 48.4 vol %) filler. However, at the 70 wt % (59.3 vol %) filler, the composites containing Thermocarb TC-300 had a higher thermal conductivity of 2.3 W/mK versus 2.0 W/mK for the other two graphites. The composites containing calcined coke had a thermal conductivity approximately half that of the composites containing graphite. This result was expected because calcined coke has a lower thermal conductivity, since it does not have a graphitic crystalline structure.

Electrical resistivity results

Figure 5 shows the log (electrical resistivity in Ω-cm) for composites containing varying amounts of single fillers as a function of filler volume fraction. In this figure, all the data points have been plotted. Figure 5

Figure 3 Through-plane thermal conductivity sample containing 40 wt % calcined needle coke F108A in Vectra A950RX at ×100 magnification.

follows the typical electrical resistivity curve. At low filler loadings, the electrical resistivity remains similar to that of the pure polymer. Then at a point called the percolation threshold, the resistivity decreases dramatically over a very narrow range of filler concentrations. At higher filler loadings, the electrical resistivity begins to level out again at a value many orders of magnitude lower than that of the pure polymer.[5,33]

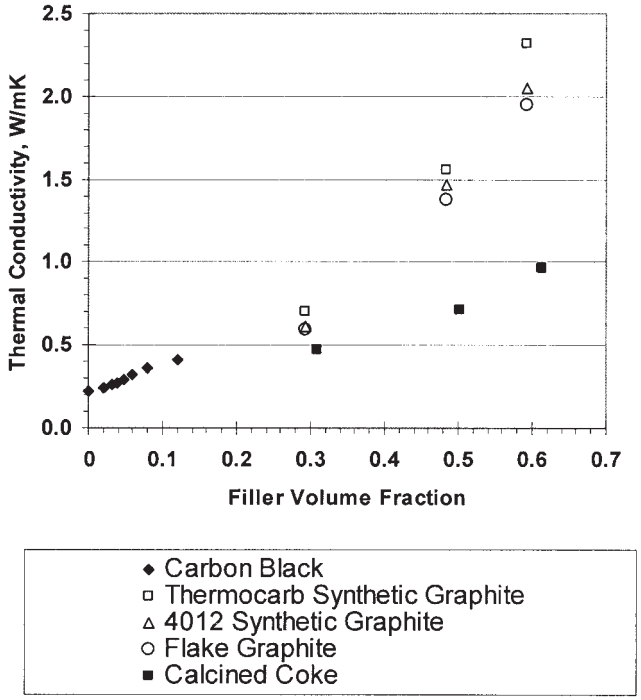


Figure 4 Through-plane thermal conductivity results.

Figure 5 shows that carbon black is effective at increasing the electrical conductivity (1/electrical resistivity) at low filler loadings. The pure Vectra A950RX has a mean electrical resistivity of 2.2×10^{16} Ω-cm (vendor literature states 10^{15} Ω-cm).[20] At the

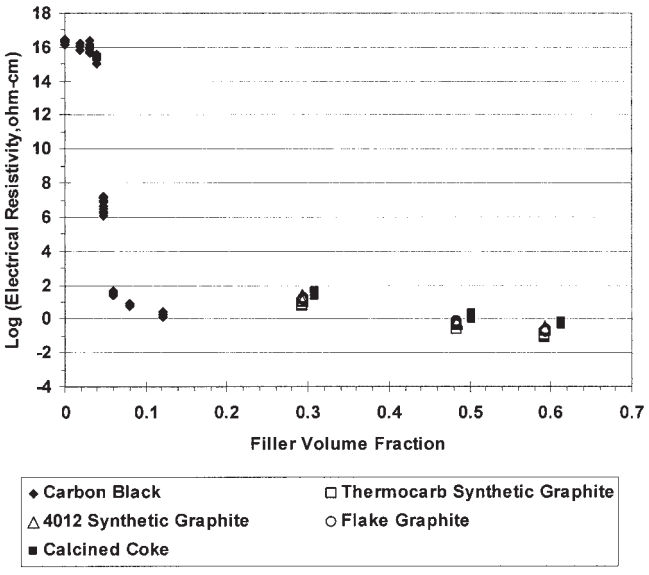


Figure 5 Electrical resistivity results.

highest filler concentration, the carbon black produced a mean composite resistivity of 2 Ω-cm (15 wt % = 12 vol %). These electrical resistivity results are similar to those reported elsewhere.[5,22]

Figure 5 shows that the Thermocarb synthetic graphite had the lowest mean electrical resistivity at 6.4, 0.26, and 0.11 Ω-cm for the 40, 60, and 70 wt % (29.3, 48.4, and 59.3 vol %) composites, respectively. The composites containing synthetic graphite 4012 had mean electrical resistivity values of 18, 0.59, and 0.28 Ω-cm for the 40, 60, and 70 wt % (29.3, 48.4, and 59.3 vol %) composites, respectively. The composites containing natural flake graphite had mean electrical resistivity values of 15, 0.55, and 0.21 Ω-cm for the 40, 60, and 70 wt % (29.3, 48.4, and 59.3 vol %) composites, respectively. Hence, the composites containing natural flake graphite and 4012 synthetic graphite had similar electrical resistivity values. The composites containing calcined coke had mean electrical resistivity values of 33, 1.4, and 0.57 Ω-cm for the 40, 60, and 70 wt % (30.8, 50.2, and 61.4 vol %) composites, respectively. These values for composites containing calcined coke are approximately twice that of the composites containing natural flake graphite or 4012 synthetic graphite. However, the electrical conductivity results for the composites containing calcined coke are higher than the authors expected because of the lack of a graphitic crystal structure.

CONCLUSIONS

The goal of this project was to determine which carbon filler produced a composite with the highest thermal and electrical conductivity. Carbon black did significantly increase the composite electrical conductivity at relatively low filler loadings (7.5, 10, and 15 wt %), but had little impact on composite thermal conductivity. Composites containing Thermocarb TC-300 synthetic graphite had the highest thermal and electrical conductivity. This result is quite likely due to the high purity and crystallinity of this synthetic graphite. Composites containing synthetic graphite 4012 and the natural flake graphite had similar thermal and electrical conductivity. Composites containing calcined needle coke, which does not have a graphitic crystalline structure, had thermal and electrical conductivity values about half that of the composites containing natural flake graphite and synthetic graphite 4012. The electrical conductivity results for the composites containing calcined needle coke are higher than that the authors expected.

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