Investigation of Elastomer Graphite Composite Material for Proton Exchange Membrane Fuel Cell Bipolar Plate

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The bipolar plate is an important and integral part of the proton exchange membrane (PEM) fuel cell and PEM fuel cell stacks. Currently bipolar plates represent more than 80% by weight and 40% by cost of the fuel cell stack. Traditional materials used for bipolar plates are primarily graphite and metal. Search for alternative materials to improve weight and cost considerations is needed. This paper discusses the results of an investigation of two elastomeric materials being developed for bipolar plate applications. Perceived advantages of the use of elastomers for this application include improved sealability without additional gasket material, reduction in the contact resistance between individual cells, improved formability, and weight reduction. The first elastomer investigated is a two component liquid silicone rubber, and the second is a polyolefin thermoplastic elastomer. These polymer matrix materials are made electrically conductive by the addition of conductive fillers including thermal graphite fibers (Cytec DKD & CKD), high surface area conductive carbon black nanoparticles (Cabot Black Pearls 2000), and graphite flakes (Asbury 4012). Electrical conductivity, processability, and elastic behavior measurements of the composites have been conducted. Some of silicone-graphite fiber composites material exhibit conductivity values comparable to those of the traditional graphite plate materials. Elasticity of all composites is maintained even at high filler concentrations. [DOI: 10.1115/1.3005580]

Keywords: PEM fuel cells, bipolar plates, elastomers

1 Introduction

Fuel cell is an energy conversion device that produces electricity directly through the electrochemical reaction of hydrogen and oxygen producing water as the only by-product. With the decrease in fossil fuel resources and increase in global warming, fuel cell technology has become an area of rising interest. Among different types of fuel cells, proton exchange membrane (PEM) fuel cells attract most research interests due to their low operating temperature, high power density, short start-up time, and nearly zero emission. Therefore, the PEM fuel cells are most desirable for the transportation application where portability and temperature are issues.

The bipolar plate is an important component of PEM fuel cells. Through the flow channel on the bipolar plates, the reactant gas enters fuel cells. The reactant gas then diffuses through the gas diffusion layer (GDL) to the catalyst layer to participate in the chemical reaction. Meanwhile, the water produced from the chemical reaction will diffuse back through the GDL and leave the fuel cell through the flow channel on the bipolar plate again. In addition, the bipolar plate can supply good electrical connections for multiple cells in stacks and provide sufficient support for the fuel cell components. Therefore, a suitable material for bipolar plates should have good electrical and thermal conductivity, impermeability to gases, and good mechanical, chemical, and thermal properties.

Materials that are typically used for bipolar plates are graphite, metals, and polymer composites. Graphite has been held as the benchmark material for bipolar plates due to its very good electrical and thermal conductivity, and good thermal stability. However, due to graphite's brittle behavior and expensive processing, investigation in alternative materials has increased. Metal plates are less expensive to fabricate and have higher mechanical strength than graphite plates. However, there are issues with corrosion, processing, and added weight. More emphasis has been placed on organic composites in recent years. Organic composites consist of a matrix material that is either a thermoset or thermoplastic that is combined with noncorrosive conductive filler. The use of organic composites allows for much more efficient processing for bipolar plates with even better mechanical strength and corrosion-resistances than the aforementioned materials. Organic composites developed for bipolar plate applications include thermoset composites based on vinyl ester [1,2], and phenolic resin [3,4], and composites based on thermoplastic resins including polypropylene [4,5], polyethylene [6], polyester [7,8], polyphenylene sulfide [5,8], polycarbonate [9,10], and polyvinylidene fluoride (PVDF) [6,11]. None of the composites discussed in literature are based on elastomeric materials.

Elastomers have been implemented for use as gasket material of fuel cells [12]; however, their use as bipolar plate material has yet to be explored. Current research explores the use of elastomer composite materials for bipolar plate applications. Base polymers being investigated are either silicone slurry or a polyolefin thermoplastic elastomer. Conductive fillers include graphite fibers, graphite flakes, and high surface area conductive carbon black nanoparticles. This paper represents a preliminary investigation of the potential use of elastomers as bipolar plate materials.

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Table 1 Elastomer selected properties

Property	Silicone [13]	Santoprene [15]
Density (g/cm^3)	1.37	0.95
Electrical resistivity (Ω cm)	5.7×10^{14}	—
Thermal conductivity (W/m K)	0.31	0.12
Hardness, Shore A	62	60
Tensile strength (MPa)	3.24	5.9
Tear strength (kN/m)	3.33	32
Operating temperature range (°C)	−60–204	−60–135

Table 3 Graphite DKD (vol %) in matrix material

Formulation No.	Graphite DKD (vol %)
1	0
2	25
3	30
4	35
5	40

2 Experimental Study

2.1 Material

2.1.1 Elastomer Selection. Two matrix materials have been selected: a two component liquid silicone elastomer (GE Silicones, RTV 627A and B) and a grade of polyolefin thermoplastic elastomers (Santoprene).

The silicone elastomer is composed of vinyl polydimethyl siloxane and contains dimethylhydrogen siloxane as curative. The two component polymer can be cured either at room temperature or higher temperature for faster curing time. The polymer has exceptional thermal stability, a wide range of usage temperature, and shows very little degradation under exposure to fuel cell reactant gases [12]. Mechanical, electrical, and thermal properties of the polymer are shown in Table 1 [13].

The dynamically vulcanized thermoplastic elastomer (Santoprene TPV 8201-60) is a two phase solid polymer consisting of a lightly vulcanized ethylene-propylene rubber phase dispersed in a thermoplastic polypropylene matrix. The polymer has good thermal, UV, oxidative, and water resistance, providing ideal conditions for use in PEM fuel cells. As a thermoplastic elastomer, the material can be processed using conventional plastic techniques [14], including extrusion and injection molding. Mechanical and thermal properties of the polymer are also shown in Table 1 [15].

2.1.2 Conductive Filler Selection. The two main conductive additives investigated thus far are the thermal graphite fibers (Cytec DKD and CKD). The properties of these fibers along with other conductive additives used in this investigation are shown in Table 2. The two grades are pitch-based high thermal conductivity fibers developed for thermal management applications. Both grades are also highly electrically conductive [16]. The electrical resistivity is less than 3 $\mu\Omega$ m. Tensile strength of the fibers is greater than 2060 MPa. The only difference between the grades is the fiber length; namely, 10^6 μm for CKD grade and 200 μm for DKD.

Other conductive additives investigated include a grade of synthetic graphite flake (Asbury 4012) and high surface area conductive carbon black nanoparticles (Cabot Black Pearls 2000, Degussa Printex XE 2, and Cabot Vulcan XC-72). The diameters of those particles fall between 12 nm and 29 nm, and their surface area is 180–950 m^2/g, as seen in Table 2. They are all classified and used as highly conductive additives for elastomers and other materials [17–19].

2.2 Processing. The main investigation reported in this paper includes compositions containing an elastomer matrix and a conductive filler Cytec DKD. Compositions containing 25–40% by volume conductive filler were prepared, as shown in Table 3. Two series of these compositions were prepared using silicone rubber in one series and thermoplastic elastomer for the other. The methods of mixing and molding the compositions were different for the two elastomeric matrices.

In the case of the liquid silicone matrix GE RTV 627A and B, the two polymer components were first mixed either by hand or in some cases using an electric blender. The conductive additives were then incorporated and mixed with the elastomer matrix to form a slurry. A weighed amount of the slurry was spread over one surface of a $10 \times 10 \times 0.3$ cm^3 compression mold. The mold was closed and placed in a heated press to cure the thermoset polymer. Molding was conducted for 20 min at a temperature of 160°C and a plate pressure of 35 MPa.

The thermoplastic elastomer (Santoprene) compositions were blended using a 42 mm diameter counter-rotating twin screw extruder (C.W. Brabender Type D-51). The materials were extruded into continuous stands using a 3 mm rod die. Extrusion was conducted at 30 rpm and extruder temperatures of 120°C at the first zone closest to the resin feeder, followed by temperatures of 169°C and 160°C in the extruder barrel zone and 156°C at the die. After cooling down to around room temperature, the strands were pelletized using a Honeywell machine. Pellets were then compression molded at a temperature of 190°C for 5 min at a pressure of 35 MPa, using the same mold used for preparing the silicone composite plates.

In addition to the above compositions, conductive elastomers were also prepared using combinations of carbon/graphite fibers and nanoparticle carbon black materials. The exact compositions of those materials will be shown in Sec. 3.

Table 2 Conductive filler selected properties

	Thermal graphite fibers		Carbon black nanoparticles			
Property	CKD	DKD	Printex XE 2	Black Pearls 2000	Vulcan XC-72	Graphite flake Asbury 4012
Density (g/cm^3)	2.20	2.20	1.8	1.8	1.8	2.23
Electrical resistivity (Ω cm)	<0.0003	<0.0003	—	—	—	0.03
Thermal conductivity (W/m K)	400–700	400–700	—	—	—	—
Additive shape	Fiber	Fiber	Particulate	Particulate	Particulate	Flake
Additive dimensions						
Diameter (μm)	10	10	0.025	0.012	0.029	50–800
Length (μm)	1×10^6	200	—	—	—	—
Surface area (m^2/g)	0.4	0.4	950	1450	180	1.5

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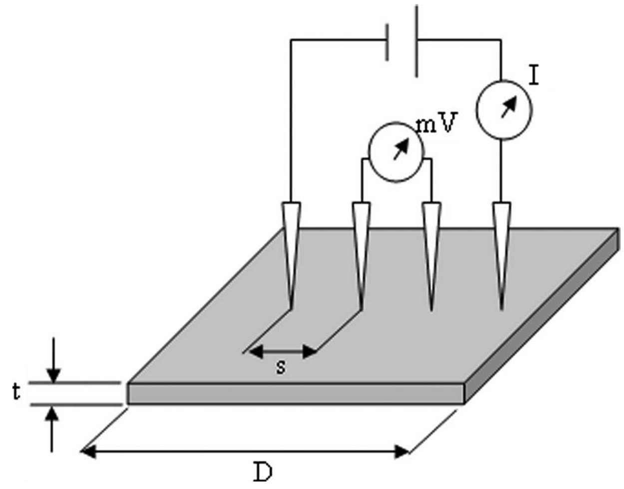

Fig. 1 Experimental schematic of the four-point probe resistivity setup [20]

2.3 Material Characterization

2.3.1 Processability Evaluation. It is well known that the addition of fine high surface area fibers or particles can increase the viscosity of polymer-filler composites to high values, resulting in materials that are difficult to flow and process. A melt flow index apparatus is used (MP 993 extrusion plastometer, Tinius Olsen) to determine the effects of the conductive fillers on processability of the composites. The capillary die diameter of the apparatus was 2.1 mm, and the length of the die was 8.0 mm. The measurements were all conducted at 190°C with a load of 10.66 kg.

2.3.2 Conductivity Measurements. A four point resistivity probe (Signatone Model S-301-4) connected to a Keithley digital multimeter was used to measure the electrical resistance of composite plates. The four point probe consists of two center current carrying probes and two outer voltage measuring probes shown in Fig. 1 [20]. Sheet resistance, in ohms, is measured by this technique.

The composite resistivity can be calculated from sheet resistance using Eq. (1) [21]:

$$\text{sheet resistance}(R_s) = \rho/t = (V/I) \times \text{CF}_1 \times \text{CF}_2 \tag{1}$$

where ρ is the resistivity, t is the sample thickness, CF_1 is the correction factor 1 (D/s) ratio, and CF_2 is the correction factor 2 (t/s) ratio.

2.3.3 Determination of Compression Modulus. The elastic behaviors of the pure polymers and of their conductive composites were evaluated by measuring the compression modulus under stress loading and unloading conditions. A low compression modulus and a low compression set after release of the stress indicate high elastic behavior, low deformation resistance, and high sealing capability. The compression moduli were determined on button samples 21.0 mm in diameter and about 8.2 mm in thickness. A universal testing machine (MTS Q Test/50 LP) was used for the measurements. Samples were compressed 25% of their original thickness at a strain rate (crosshead speed) of 0.508 mm/min and returned to zero stress at the same strain rate. Stress-strain data were collected during stress loading and unloading.

3 Results and Discussions

3.1 Electrical Resistivity. Resistivity measurements of composites made of silicone elastomer containing various concentrations of graphite (DKD) fiber are shown in Figure 2. The resistivity values were calculated using the sheet resistance values measured by the four point probe apparatus and applying the correction factors (Eq. (1)) [21] to account for sample diameter and thickness. As seen from the data, the addition of as little as 25% by volume graphite fiber reduces the resistivity to 0.327 Ω cm. Resistivity continues to decrease with additional graphite fiber concentration, as shown in Fig. 2. A resistivity of 0.063 Ω cm is reached at 35% conductive filler concentrations. This resistivity compares favorably to that of graphite plate Grade FU 4369, which is measured at 0.028 Ω cm (manufacture value is 0.05 Ω cm [22]). The resistivity reduction in this system is more pronounced than comparable systems using thermoplastic polymer matrices filled with particulate and/or fibrous conductive additives [2,5,23].

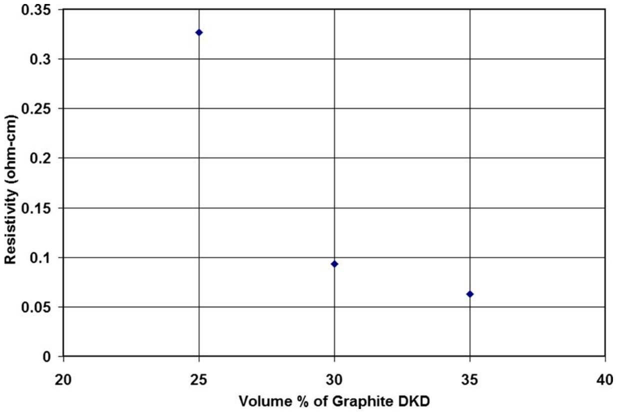

Fig. 2 Silicone resistivity as a function of vol % of graphite DKD

A similar study using a polyolefin based thermoplastic elastomer (Santoprene) showed that electrical resistivity of this elastomer is not as responsive to the concentration of conductive graphite fiber as was the silicone matrix. Resistivity results for compositions containing graphite volume concentrations between 25% and 40% are shown in Fig. 3. The resistivity remains very high, 4036 kΩ cm, even at a high concentration of graphite filler of 35% by volume. The resistivity drops down to 2.91 kΩ cm at 40% graphite fiber concentration. Further addition of graphite was not possible because of great difficulty in melt extrusion mixing of such compositions.

Further investigations are needed to explain the different behaviors of the two elastomeric matrices with respect to the filler content. The differences could be attributed to the chemical composition of the polymers, namely, the silicone elastomer has a silicon backbone structure and, therefore, is probably more compatible

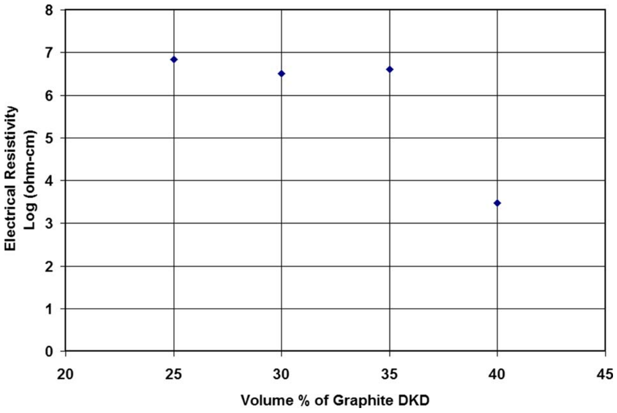

Fig. 3 Santoprene resistivity as a function of vol % of graphite DKD

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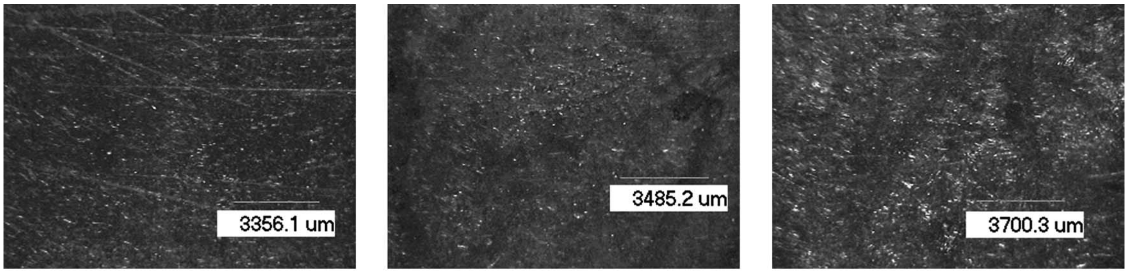


(*a*) (*b*) (*c*)

Fig. 4 Comparison of vol % silicone/graphite DKD filler composition microscopy images. (*a*) 75/25, (*b*) 70/30, and (*c*) 65/35.

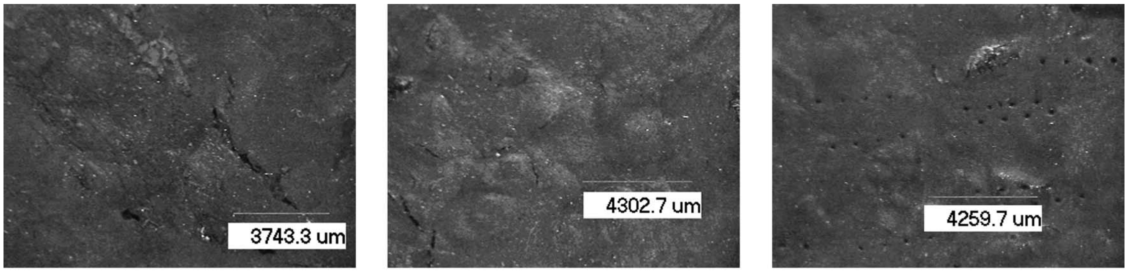
with the graphite fiber than the all hydrocarbon structure of the thermoplastic elastomer. Different polymer matrices have been found to have varying responses to conductive filler content, as was shown in the case of polyphenylene sulfide and polypropylene composites [5]. Reduction in resistivity in the case of polyphenylene sulfide was much more responsive to filler concentration than in the case of polypropylene. The other cause for the resistivity behavior could be the differences in sample mixing. The liquid silicone elastomer is blended in a liquid slurry form using hand mixing or blender mixing. For the thermoplastic elastomer melt mixing, a twin screw extruder was used. This extensive mixing might have resulted in breakdown of the fiber length, thus reducing its effectiveness as conductive filler. Light microscopy of the surfaces of molded slabs of the two polymer compositions supports this hypothesis. Images of the slab surfaces of silicone composites and Santoprene composites were taken using MOTIC IMAGE 2000 software and are shown in Figs. 4 and 5. The lighter parts of the images are the graphite fibers, where the gray areas are polymer matrices. It can be observed that at comparable magnification graphite fibers are easily detected in the case of the silicone, but are hardly noticeable for the Santoprene samples, probably due to pulverizing the graphite during extruder melt mixing.

3.1.1 Electrical Resistivity of Composites Containing Mixed Conductive Fillers. A preliminary investigation was made of the two polymer matrices containing a mixture of conductive additives. Table 4 shows such composition based on silicone elastomer as the matrix. The results are also plotted in Fig. 6. The resistivity decreases with the total conductive filler concentration in a predictable manner. At the same filler concentration, the composite plate with Cytec CKD graphite filler exhibits a lower electrical resistivity compared to those with Cytec DKD graphite filler due to a long fiber length of 1×10^{6} μm for Cytec CKD opposed to a short fiber length of 200 μm for Cytec DKD. However, without introducing different conductive fillers in the matrix, the Cytec CKD thermal graphite fibers will not be able to be used at that level of conductive filler in a bipolar plate due to the sample's surface finishes.

An attempt to incorporate mixtures of graphite fiber (Cytec DKD) and nanoparticle carbon black particulate (Black Pearls 2000) was not successful because of degradation in processability of the composites, making it very difficult to prepare acceptable samples. Composites containing volume percent of 21.47 graphite fiber with 8.86 nanoparticle carbon black and 18.33 graphite fiber with 7.47 nanoparticle carbon black both failed to process well.

Mixed conductive filler systems were also tried using the thermoplastic olefin as the matrix. In one case a mixture of graphite flake (Asbury 4012), nanoparticle carbon black (Cabot Vulcan XC-72), and another nanoparticle carbon black (Degussa Printex XE-2) were melt mixed with the thermoplastic olefin matrix at volume percent ratios of 20.80, 2.03, and 10.41, respectively. The resistivity of the composite was measured at 40 Ω cm. The total concentration of the conductive filler was 33.24% by volume. The resistivity is much lower than that of a comparable composite containing graphite fiber DKD (3200 kΩ cm, see Fig. 3). In a second composition, a mixture of 32.35 of graphite fiber DKD and 7.7 vol% of nanoparticle black (Vulcan XC-72) were blended with the thermoplastic olefin. The conductivity of this mixed conductive filler composite (76 Ω cm) is again much lower than a comparative graphite only composite (2.91 kΩ cm). Apparent synergistic interactions in a blend of mixed conductive fillers are a possible explanation for the high unexpected reduction in resistivity. This phenomenon needs to be explored further.

3.3 Melt Flow Index. Processability of composites made of a polymeric matrix and conductive fiber or particulate additives has been always a concern. A desire for low resistivity to meet bipolar plate requirements necessitates addition of high concentrations of the conductive filler. Such high concentrations of high surface area additives degrade the processability as mentioned earlier in our report. One way to obtain quantitative data on processability is to conduct melt flow index measurements. The apparatus is described in Sec. 2. Measurements for thermoplastic elastomer (Santoprene) composites with graphite fiber (Cytec DKD) were



(*a*) (*b*) (*c*)

Fig. 5 Comparison of vol % santoprene/graphite DKD filler composition microscopy images. (*a*) 75/25, (*b*) 70/30, and (*c*) 65/35.

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Table 4 Composition and electrical resistivity of polymer-mixed conductive filler composites

Formulation No.	Conductive filler (vol %)			Electrical resistivity (Ω cm)
	Cytec thermal graphite fiber		Total conductive filler	
	DKD	CKD		
1	6.76	6.76	13.52	14.848
2	10.40	10.4	20.80	0.438
3	11.87	12.11	23.98	0.169
4	29.41	0	29.41	0.111
5	0	29.41	29.41	0.044
6	22.28	9.04	31.32	0.077
7	38.46	0	38.46	0.074
8	0	38.46	38.46	0.069

conducted for 5 min at 190°C and under a load of 10.66 kg.

Melt flow index is the number of grams that passes through a fixed diameter die in a specified amount of time. Since all samples were collected for the same amount of time, the melt index becomes a measure of shear rate. At constant stress used, the melt index is then inversely proportional to melt viscosity. Hence a plot of the ratio of melt index of unfilled elastomer matrix to that of graphite fiber filled composites $(\mathrm{MFI})_m/(\mathrm{MFI})_f$ versus content of filler (Φ_f) will be equivalent to a plot of the ratio of viscosity of the graphite composite to the viscosity of the matrix μ_f/μ_m. The plot of $(\mathrm{MFI})_m/(\mathrm{MFI})_f$ versus % (Φ_f) is shown in Fig. 7. Moderate increase in viscosity is observed at low filler concentration (up to 25%) followed by a sharper increase at higher filler content (25–35%), and very sharp increase (beyond 35%) indicating poor flow and processability. The same trend of dependence of viscosity on filler content is observed for Newtonian fluids [24,25] filled with spherical glass beads. The behavior can be explained in terms of filler particle interactions within the polymer matrix. At low concentration a moderate increase in viscosity with increased filler content is predicted by the suspension theory and indicates no filler particle interactions [26] within the matrix. At higher concentrations filler aggregates start to form and collide under shear, causing the viscosity to rise rapidly. At very high filler concentrations filler network structures are formed within the matrix, which causes the viscosity to increase vertically. At that point the material becomes very difficult to flow exhibiting solid behavior.

3.4 Elastic Property Evaluation. Maintaining elastic behavior of the elastomer containing conductive filler is a requirement for a bipolar plate intended to eliminate added seals when making a proton exchange membrane cell assembly. Sealing between the bipolar plate and active membrane is required to contain reactant gases and reaction by-products. To evaluate whether or not the two elastomer systems maintain good elasticity after the addition of fillers, cyclic compression testing was conducted to evaluate changes in the compression modulus and compression set as a function of filler contents.

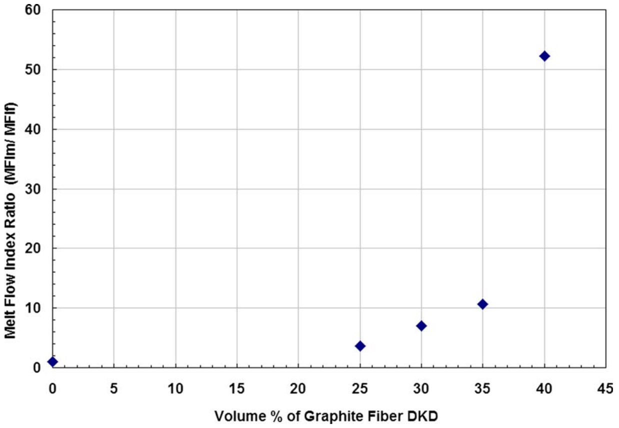

Fig. 7 Melt index of thermoplastic elastomer (Santoprene) composites as a function of vol % of graphite DKD collected for 5 min at 190°C and at a load of 10.66 kg

The stress-strain curves of a 100% silicone rubber matrix and a composite containing 60% silicone rubber and 40% by volume graphite fiber (Cytec DKD) are shown in Figs. 8 and 9, respectively. The curves show that compressing the samples through 25% of their original thickness and then unloading the stress results in almost complete recovery of the sample to its original thickness. This shows good elastic behavior for both pure rubber matrices and composites containing high graphite fiber content (40%).

The stress-strain curves were used to calculate compression modulus for samples with and without conductive fillers. The results are shown in Table 5. The table also shows compression set values for all materials calculated using the thickness of samples before and after compression testing. All values are averages of triplicate runs. Low modulus values and low compression set values for the pure matrix and the composites indicate that the materials maintain good elastic behavior and should provide good sealing at low pressure. Scatter in the data for the silicone composites might be the result of variation in the degree of curing of the elastomer matrix, caused either by interference of the conductive filler or improper curing conditions. The results for the ther-

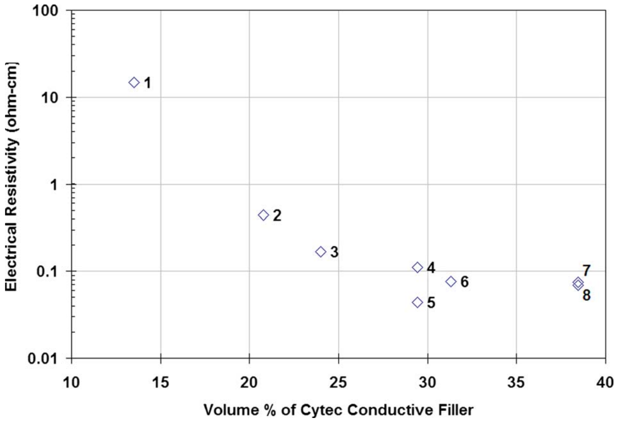

Fig. 6 Electrical resistivity versus conductive filler concentration for silicone mixed filler composites shown in Table 4

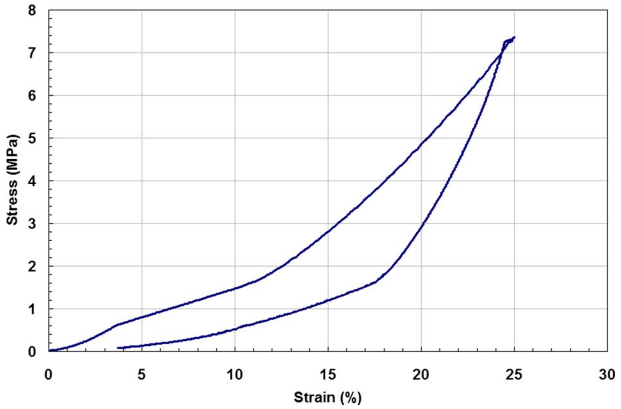

Fig. 8 Stress/strain behavior of silicone rubber matrix during stress loading/unloading cycle

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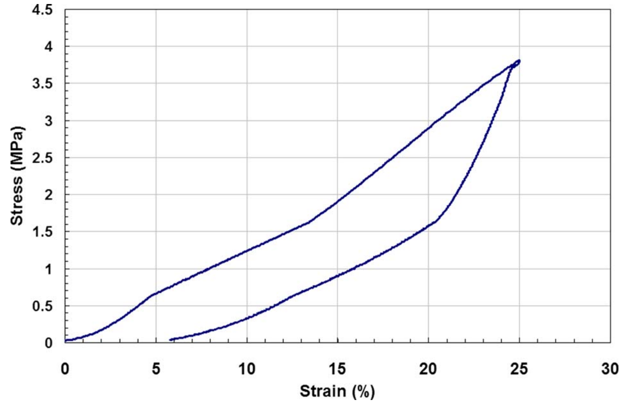

Fig. 9 Stress/strain cycle of a composite containing 60/40 (by volume) silicone rubber matrix/graphite fiber composite during stress loading and unloading cycle

moplastic elastomer, which do not undergo curing, are much more consistent.

4 Conclusions

Electrically conductive elastomeric composites were made based on a two component liquid silicone rubber or polyolefin thermoplastic elastomers (Santoprene) as the matrices. The conductive fillers used were thermally and electrically conductive graphite fiber (Cytec DKD) and blends of the graphite fiber with other conductive additives, including nanoparticle carbon black grades and graphite flake. Using silicone elastomer as the matrix, electrical resistivity measurements showed low resistivity comparable to that of a commercial graphite bipolar plate material. Such low resistivity was not achievable using the thermoplastic elastomer as the matrix material. In both cases, however, good elastic behavior was maintained even at high additive concentration. On the other hand, processability characteristics degraded at higher filler concentration.

Future work will be directed at the development of highly conductive elastomeric composites that maintain good processability. The use of polymer blends as matrices and mixed conductive filler systems will be investigated.

Table 5 Properties of elastomeric compositions in compression

Polymer/conductive filler volume ratio	Compression set (%)	Compression modulus (MPa)
Thermoplastic elastomer/graphite fiber		
100/0	2.1	6.34
75/25	3.1	8.09
70/30	2.8	8.18
65/35	3.7	9.35
0/40	3.6	9.80
Silicone/graphite fiber		
100/0	1.6	23.35
75/25	2.0	19.07
70/30	3.9	30.47
65/35	3.6	25.56
62/38	6.3	23.79
60/40	4.4	12.92

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