Polymeric composite bipolar plates for vehicle applications

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Abstract

At present membrane electrode assembly performance levels and stack operating conditions of PEM fuel cells, a plate area specific resistance of less than approximately $20\,m\Omega\,cm^2$ and a plate thickness of less than 2 mm are required to meet the vehicular volumetric power density target ($>2\,kW\,l^{-1}$). It is, however, difficult to meet these aggressive requirements, and simultaneously obtain good mechanical properties when using polymeric plate materials. Polymers become brittle and break frequently at the high conductive filler loadings (e.g., >50 v/o graphite) required for high conductivity. This study investigates a potential approach for obtaining high plate conductivity at low conductive filler loadings, thus enabling high volumes of thin and ductile plates to be manufactured at low scrap rates.
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1. Introduction

In an electrochemical reaction, the direct conversion of chemical energy into electrical energy is not limited by Carnot-cycle constraints; thus, at standard operating temperatures, the thermodynamic efficiency limit of a fuel cell engine is approximately twice that of an internal combustion (heat) engine. However, in practice, voltage losses or overpotentials exist during the operation of PEM fuel cells and reduce this efficiency advantage. Three major types of losses are observed in low-temperature PEM fuel cells: (1) activation losses—slow reaction kinetics in the cathode catalyst layer; (2) mass-transport (gas and protons) losses—failure to transport sufficient reactant to the electrodes; (3) ohmic losses—low protonic and electronic conduction through the thickness of the polymer electrolyte and the bipolar plate, respectively [1].

A stack volumetric power density greater than $2\,kW\,l^{-1}$ is required for PEM fuel cells to be viable in vehicle applications. Although large cathodic activation overpotentials (300–400 mV at $1\,A\,cm^{-2}$) currently exist and are contribut-

ing most to the total losses, not much improvement has been made over the past 40 years in reducing these losses. High temperature membrane studies have only recently been initiated in an attempt to reduce these losses. Likewise, an increased focus on new polymer electrolyte materials has only recently begun in an attempt to increase the membrane protonic conductivity at reduced relative humidity. Instead, to achieve $2\,kW\,l^{-1}$ in the near future, many fuel cell manufacturers and plate suppliers have focused on reducing the losses attributed to the bipolar plate, namely reducing the mass-transport losses via an optimization in the flow field geometry and reducing the ohmic losses via a reduction in the plate thickness and resistivity.

At present membrane electrode assembly (MEA) performance levels and stack operating conditions, a plate area specific resistance of less than approximately $20\,m\Omega\,cm^2$ and a plate thickness of less than 2 mm are required to achieve the power density target. Suppliers are attempting to meet these resistance and thickness requirements using both metal and polymer plates.

Metal plates such as titanium and stainless steel exhibit good mechanical and low gas permeation characteristics, enabling thin plates to be manufactured at high dimensional tolerances and at low scrap rates. These metals also exhibit

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excellent corrosion resistance in a PEM fuel cell environment because a stable and protective metal oxide film (i.e., a passive film) forms on their surfaces [2]. To make these plates conductive, the electrically insulating oxide film must be removed or its thickness reduced, and a conductive coating must then be applied to prevent growth of the oxide film. Coating imperfections such as pin holes, which expose the metal surface to the fuel cell environment, are not problematic because these passivated pin-hole areas are electrochemically stable and represent only a small fraction of the total area for electron transport. Fuel cell manufacturers have developed such protective and conductive inorganic [3] and organic [4] coatings. Metal plates protected with these coatings have accumulated over approximately 1000 h of fuel-cell stack testing without any evidence of an increase in plate resistance nor a metal ion contamination of the MEA (anode-side), although long-term durability (i.e., oxide growth protection and anode plate dissolution) is still of concern and continues to be studied for both stationary (40,000 h target) and mobile (6000 h target) applications.

In contrast, polymer plates, for the most part, exhibit good electrochemical stability in the aggressive PEM fuel-cell environment and long-term durability is not a major concern [5]. It is, however, much more difficult to meet the resistance and thickness targets, and simultaneously obtain good mechanical properties when using polymer plates. The intrinsically insulating polymer resin must be filled with high loadings (>50 v/o) of corrosion-resistant conductive particles such as carbon black and/or graphite in order to meet the resistance target. These loadings greatly exceed percolation threshold concentrations (5–20 v/o) and approach critical pigment volume concentrations (60–70 v/o) used to formulate "electrically conductive" plastics and coatings [6]. It is at these critical concentrations that the electrical resistance decreases by many orders of magnitude and the material goes through an insulator–conductor transition. Unfortunately, at or above these concentrations, the material becomes brittle [7].

Many studies have been conducted with the intent of reducing the percolation threshold so that high material toughness and high conductivity can be achieved. Factors such as binder type, filler particle size/distribution [8], degree of mix [6], polymer blends [9], polymer crystallinity [10], and particulate polymer microstructure [11], and their affect on percolation threshold were investigated. Although these studies were successful in reducing the percolation thresholds and in developing materials with resistivities sufficient for antistatic ($10^{12}\,\Omega\,cm$), electrostatic painting ($10^6\,\Omega\,cm$), and electromagnetic interference shielding ($10^1\,\Omega\,cm$) applications, these materials are not conductive enough for plate applications ($10^{-2}\,\Omega\,cm$). Consequently, separator plates (2.5–3 mm thickness) are presently formulated well beyond these critical filler concentrations and, as a result, are inherently brittle. Not much can be done to toughen the materials at such low polymer concentrations. Excessive scrap rates are observed during plate demolding, adhesive bonding, and

stack assembly operations, and higher scrap rates are anticipated using these brittle materials in thinner, targeted plate geometries.

A more recent study investigated the use of low loadings of high aspect-ratio conductive particles (e.g., carbon/graphite fibers, flakes, carbon-black aggregates, etc.), an alignment process [12], and a conductive-tie layer [13] to meet plate conductivity, thickness, and toughness targets [14]. The long and more conductive axis of discrete conductive fillers was aligned during mold filling in the current-flow or through-thickness plate direction using an optimized mold (plate) geometry containing excess land heights. As a result, a significant reduction in resistivity was realized in that same direction at reduced filler loadings, which, in turn, increased the material toughness. A conductive-tie layer was then applied to the plate surface to reduce the resulting high contact resistances at the plate-to-diffusion medium (DM) interfaces, which are attributed to the low surface concentration of conductive particles. Unfortunately, this approach requires an expensive machining operation to remove the excess land heights needed for alignment, and although a significant reduction in contact and bulk resistance was observed, the reduction was not enough. The fibers were found to align predominantly in the in-plane plate direction and high filler concentrations were still required to meet the conductivity target. The materials were too brittle at these high filler loadings.

In this study, we investigate the use of porous and compressible expanded graphite (EG) particles as a conductive filler material for PEM bipolar plates. The goal is to achieve high plate conductivity at low EG loadings. At high polymer loadings and with the selection of the appropriate resin system, plate cost and plate properties (density, ductility, etc.) can be controlled or tailored more easily, enabling high volume manufacturing of thin and inexpensive automotive bipolar plates at low scrap rates.

2. Resistance measurements

The resistance of the plate materials should be measured at "stack conditions", namely the sample should contain flow field channels and should be sandwiched and compressed at stack pressures between two pieces of diffusion media (DM), as shown in Fig. 1. The plate resistance is the sum of the bulk material resistance and the pressure-dependent contact resistances located at the two DM/plate interfaces. The resistance is measured using a four-point method and calculated from measured voltage drops and from known applied currents and sample dimensions. The voltage drop is measured across either the two diffusion media (total resistance) or two points on the plate surface (bulk resistance). For the former, thick DM (1.0 mm, Toray TGP-H-1.0 T) is used so that needle-like voltage probes can make contact with the DM easily. The contact resistance is the total resistance less the bulk resistance.
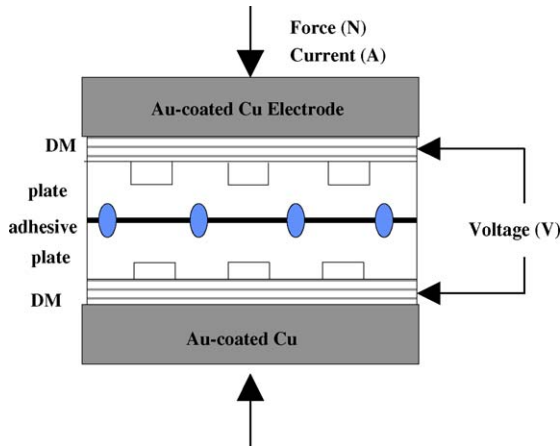
Fig. 1. A four-point method for measuring the area specific resistance of composite plates (cross-sectional view).

The contact resistance (CR) at the DM/plate interface is significant and may contribute more to the total plate resistance than the bulk resistance, particularly at low compression pressures. CR is not clearly understood because it depends on so many factors, such as bulk conductivity, sample flatness and roughness, pressure, and surface composition. It is clear, however, that the CR must be reduced in order to meet the resistance target at reduced filler loadings and at low stack pressures.

The CR at the DM/plate interface can be reduced significantly with the use of a conductive-tie layer. Fig. 2 is a schematic illustrating the conductive-tie layer electron-transport mechanism. High CR at this interface is attributed to few physical contact points between the carbon fibers in the DM and the carbon particles at the plate surface. In the uncompressed state, the DM is only 20–25 v/o fiber, the remainder air. The surface of a composite plate may contain anywhere from 30 to 90 v/o carbon. Hence, at low compressions, the probability of having few fiber-to-carbon contact points and, in turn, a large CR is high. If the compression pressure is increased, the DM density and the number of contact points increases, thereby reducing the CR. Increasing the compression pressure is, however, a poor solution. It is more advantageous to compress stacks at low pressures to reduce
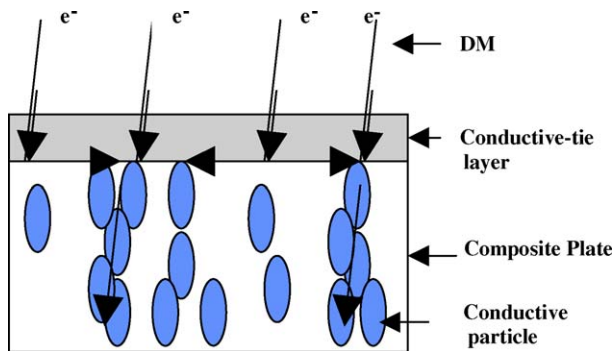
the mass-transport overpotentials and to reduce the mass and size of the bulky end and side plates, which are used to hold the stack under compression. A better alternative is to use a more electrically conducting layer, i.e., a conductive-tie layer, at the DM/plate interface that "short circuits" the flow of current. The use of a conductive-tie layer becomes increasingly more important as we attempt to reduce the filler loadings for improved mechanical properties.

3. Proposed conductivity mechanism

Ideally, to minimize the plate's conductive filler concentration, i.e., maximize its polymer content for enhanced mechanical properties while maintaining high conductivity, the filler should extend continuously through the thickness of the plate. Fig. 3 shows graphite fibers extending from the anode-side of the plate, where electrons are produced in the H_2 oxidation reaction, to the cathode-side of the plate, where electrons are consumed in the O_2 reduction reaction. The fibers provide a low resistance path for anode-to-cathode electron transport. The plate's maximum through-thickness resistivity ρ_{max} should not exceed roughly 0.05 Ω cm, assuming a negligible contact resistance at the plate/DM interface, an area specific resistance of less than 20 mΩ cm^2, a plate thickness t of less than 2 mm, and a land-to-channel width ratio of one (i.e., maximum current density i of 2 A cm^{-2}):

$$\rho_{max} \equiv \frac{V}{it} \equiv \frac{0.020\,\text{V}}{(2\,\text{A cm}^{-2})(0.2\,\text{cm})} \equiv 0.050\,\Omega\,\text{cm}. \tag{1}$$

Because continuous fibers extend through the plate thickness, the "rule of mixtures" for conductivity can be applied to determine the volume fraction β of graphite fiber required to meet the ρ_{max} requirement:

$$\sigma_T \equiv \beta\sigma_F + (1 - \beta)\sigma_P \cong \beta\sigma_F, \tag{2}$$

where σ_T, σ_F, and σ_P are the total composite, fiber, and polymer conductivities, respectively, and $\sigma_F \gg \sigma_P$. Only 0.5 v/o of pitch-based graphite fiber (e.g., BP Amoco P100) is required for conductivity:

$$\beta \equiv \frac{\rho_F}{\rho_{max}} \equiv \frac{2.5\text{E}^{-4}\,\Omega\,\text{cm}}{5.0\text{E}^{-2}\,\Omega\,\text{cm}} \equiv 0.005. \tag{3}$$

At this very low fiber concentration, high strength and ductile plate materials can easily be formulated. Note,

Fig. 2. A schematic illustrating the electron transport mechanism using a conductive-tie layer.
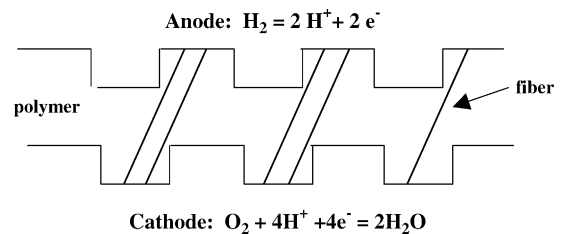

Fig. 3. Idealized plate with continuous graphite fibers extending through the plate thickness.

however, that the CR is not negligible at low fiber concentrations and a conductive-tie layer would have to be applied to reduce the CR. Some work has begun recently using this approach, although to manufacture plates with continuous graphite fibers directed in the thickness direction is extremely challenging [15].

A simpler approach is to use a *continuous* conductive filler, such as expanded graphite. Particles or "chunks" of EG (as opposed to exfoliated EG "worms") are desired and generated by grinding sheets of EG foils to a size ranging from 10 to 2000 μm (e.g., SGL's Conductograph® EG-15 and GFG-500). Because the EG chunks are both porous and compressible, they tend to occupy more space and percolate electrons at lower loadings than do non-porous and incompressible graphite particles, which conventionally are used in conductive plastics. These EG chunks act as "graphite sponges". The sponge walls provide an efficient and interconnected path for electron transport, the pores enable resin impregnation for enhanced strength and gas impermeability, and its compressibility facilitates extension through the plate thickness [16]. In addition, low through-thickness resistance and a lower degree of material anisotropy is realized because some of the chunks are aligned after compression molding with their conductive axes in the thickness direction of the plate.

Fig. 4 illustrates schematically two means by which low loadings of EG can produce high conductivities. In Fig. 4a, the EG chunks (>2000 μm) are initially larger than the final composite plate thickness. In an SMC-like process, the large EG chunks are sprinkled into a wet resin film; the resin is partially cured (B-stage resin); the composite material is cut into sheets and placed into a compression mold; and flow-field channels are molded into the plate. High conductivity and strength is achieved at low loadings because the large EG chunks extend through the plate thickness and resin is allowed to penetrate into the EG porous structure. In addition, because the EG chunks are compressible, they easily conform to the mold surface during molding and remain near the plate surface for low contact resistance. In Fig. 4b, the EG chunks are smaller (200–1000 μm) than the final plate thickness and larger than typical graphite particles used in conductive plastics (10–50 μm). The particles are mixed into a thermosetting or thermoplastic resin and then compression molded. A higher EG loading is required here than that used
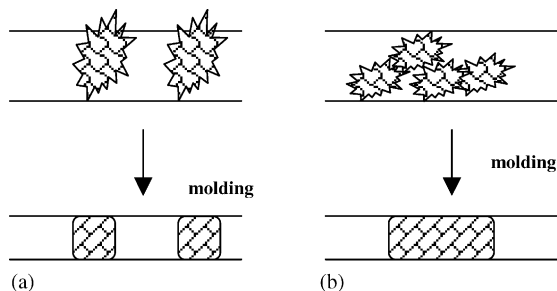
in Fig. 4a, but the mechanism of obtaining high conductivity at low loadings is essentially the same.

4. Results

4.1. Resistance

A study was performed investigating the effect of graphite loading on the total resistance (CR plus bulk) of two composite materials. UCAR GP195X graphite powder (<50 μm) was "dry" mixed with Kynar® 721 PVDF powder in a ceramic milling jar for 1 h at RT. The mix was then compression molded at 4 t to a 0.65 mm thickness using a Parma pellet press (13 mm diameter). High tonnage was used to ensure high densification and, in turn, high conductivity. The mix was also "wet" mixed in a Brabender at 200 °C followed by compression molding at 195 °C to a 0.65 mm thickness. The EG material was prepared by hand mixing EG chunks (200–1000 μm) into liquid epoxy resin (100 parts Dow 383 epoxy, 80 parts Lonza MTHPA-EG anhydride, 2 parts BDMA) and compression molding the mix to a 0.65 mm thickness at a cure temperature of 155 °C for 20 min. The samples were tested at a compression pressure of 1.4 MPa using the four-point resistance method described above. The EG samples were tested before and after sanding of their surfaces to determine whether a polymer-rich surface layer exists, which may increase the CR. All of the UCAR samples were sanded.

Fig. 5 clearly illustrates the advantage of using EG chunks over standard-sized graphite particles used in conventional high graphite plate formulations. Only 15 v/o of EG compared with 65 v/o of UCAR graphite is required to achieve less than 20 mΩ cm^2 resistance. The resistance of the EG samples was observed to remain nearly constant with increasing EG loading at approximately 20 v/o EG, suggesting that 20 v/o EG should be used to maximize the polymer content and, in turn, the mechanical strength. Although not shown in the figure, the through-plane resistances of the 20 v/o EG/epoxy composite (13.5 mΩ cm^2) and 100% EG foil samples (11 mΩ cm^2) are similar, suggesting that the

Fig. 4. Proposed conductivity mechanism using EG: (a) "large" EG chunks and (b) "small" EG chunks.
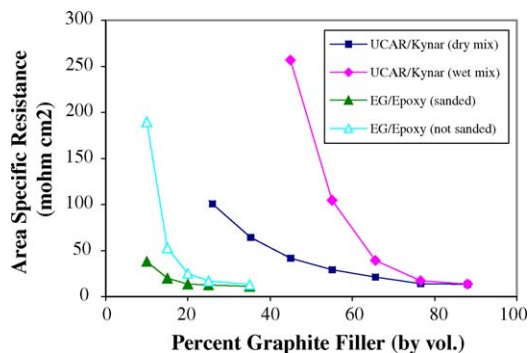

Fig. 5. Effect of graphite loading and sanding on resistance (0.65 mm, 1.4 MPa).
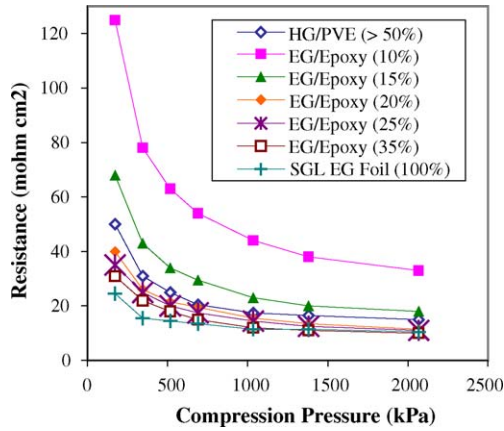
Fig. 6. Effect of graphite loading and compression pressure on resistance (0.65 mm thickness; volume percent).

bulk resistance for the 20 v/o EG sample is low and that the 20 v/o EG sample is less anisotropic than the 100% EG foil sample.

Two other important conclusions can be drawn from the data presented in Fig. 5. First, the "dry" UCAR/Kynar mix is more conductive than the "wet" mix, particularly at low carbon loadings, as expected. During "wet" mixing the PVDF coats the individual graphite particles, insulating them from each other. Second, sanding significantly reduces the CR for the EG/epoxy samples.

The data presented in Fig. 6 also suggest that a 20 v/o EG loading should be used. All samples were 0.65 mm thick and sanded, except the 100% EG foil was not sanded. At approximately 20 v/o EG, the EG/epoxy and HG/PVE samples exhibited similar resistances throughout the compression pressure range. HG/PVE (>50 v/o graphite in a poly vinyl ester resin) is considered a high graphite formulation and exhibits strengths and resistances similar to other high graphite plate formulations. Again, at high pressures, the 20 v/o EG/epoxy and 100% EG sample resistances were similar.

Fig. 7 presents preliminary results investigating the effect of the EG grinding process on resistance. Epoxy samples were compression molded to a 2 mm thickness using EG
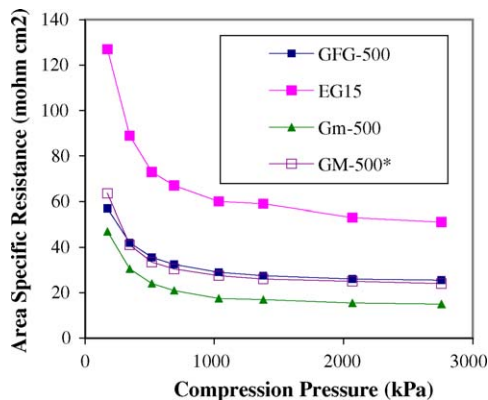

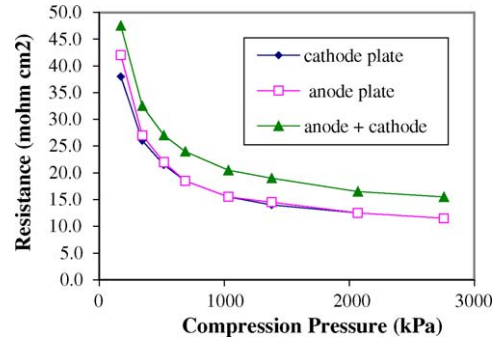Fig. 7. Effect of EG particle size and grinding method on resistance.

Fig. 8. Resistance of 20 v/o EG/epoxy bipolar plate containing flow fields and bondline (1.8 mm thickness).

chunks generated by different grinding methods. All samples contained 20 v/o EG and were sanded. The EG-15 material (<90 μm) was grounded at SGL using a proprietary process and exhibited the highest resistance. The GFG-500 sample (290–1020 μm), which was grounded at SGL from compacted foils (0.8–1.2 mm), gave higher resistances than the sample grounded internally (GM-500). These GM-500 chunks (227–950 μm) are about the same size as the GFG-500 material and were prepared by grinding thick (13 mm) pre-compacted EG foil (area weight = 2300 g m^{-2}). It appears that grinding from a pre-compacted, low-density foil produces a more porous EG particle, which percolates electrons more effectively across the plate thickness. To confirm this theory, a 0.6 mm compacted foil was grounded internally into large chunks (GM-500[*]), similar size as the GFG-500 material. The resistance of the sample containing these less porous GM-500[*] particles was higher than that obtained using the GM-500 particles. Additional work needs to be done to optimize the EG particle size and density for maximum plate conductivity.

At 20 v/o EG, it is possible to meet both the resistance and thickness plate targets. Fig. 8 presents the data for two 0.9 mm thick plates containing flow field channels. These plates were bonded together using a one-part conductive epoxy adhesive (Vantico Araldite CV 5749). An area specific resistance of 19 mΩ cm^2 was achieved at 1.4 MPa and 1.8 mm thickness. The bulk resistance contribution to the total resistance is approximately 5 mΩ cm^2, which is less than the CR contribution.

4.2. Flexural strength

Table 1 compares the flexural properties of HG/PVE (>50 v/o graphite) with those of EG/epoxy as a function of EG concentration. The ASTM D790 three-point flex test and a 0.5 mm sample thickness were used. The flexural strength and elongation at break increased with decreasing EG concentration, and the flexural modulus decreased with decreasing EG concentration. The HG/PVE sample was stiffer (greater modulus) and more brittle (lower extension at break) than the EG samples. Hence, if high elongation is more desirable than stiffness, the EG materials are preferable.

Table 1
Flexural properties (ASTM D790)

Sample	Max. load (N)	Strength (MPa)	Modulus (MPa)	Extension at break (μm)
>50% HG/PVE	4.6	54.9	15069	975
25% EG/epoxy	2.6	27.2	5654	1354
20% EG/epoxy	3.9	34.1	5761	1570
15% EG/epoxy	3.9	45.6	5602	2377
10% EG/epoxy (<1000 μm)	4.9	56.7	4944	3256
10% EG/epoxy (>2000 μm)	2.6	27.2	4199	1773
100% epoxy	9.8	106.7	4591	6640

Note that the 10 v/o EG/epoxy sample, which contains large EG particles (>2000 μm), broke prematurely. This was not expected and certainly not desired because at 10 v/o loading the large EG chunks generate higher conductivities than the small EG chunks (<1000 μm), as shown in Fig. 9. Additional work is required to determine the failure mechanism. If the sample fails adhesively due to poor EG-to-resin adhesion, then a wetting/coupling agent should be added. If the sample fails cohesively within the EG particle itself, then different particle densities and sizes should be investigated.

The data in Fig. 10 demonstrate the *opportunity* to significantly enhance the mechanical properties of high polymer-content plate materials such as EG/epoxy. As mentioned previously, the HG/PVE (>50 v/o graphite) material is stronger, stiffer, and more brittle than the EG/epoxy materials. However, for example, if 4 v/o of the

EG is replaced with a reinforcing carbon fiber (unsized PAN 33; 3 mm length) while maintaining the total carbon concentration at 25 v/o, then the EG/epoxy material becomes stronger and tougher than the HG/PVE material, without sacrificing conductivity. The addition of 4 v/o of PAN 33 to the HG/PVE formulation would also increase its strength and toughness, but to a lesser extent because of its initial high graphite content. The sample containing MF 30 PAN fibers (150 μm length) exhibited higher ductility than the 25% EG/epoxy sample. The sample thickness was 1.0 mm.

4.3. H_2 permeation

Low H_2 permeation rates were measured through the thickness of the 20 v/o EG/epoxy plate material. For a plate thickness of 0.5 mm, the rate of permeation was 1.2×10^{-6} cm^3 cm^{-2} s^{-1} (0.01 mA cm^{-2} equivalent at 25 psig and 80 °C), fives times less than the target. This suggests that the low-viscosity epoxy successfully impregnated the porous EG particles and that thin plates can be used effectively.

4.4. Fuel cell performance

Flow field channels were machined into the 25v/o EG/epoxy plate material to create 100 mm × 100 mm anode and cathode plates. These plates were tested in a fuel cell test stand at 25 psig, 100% RH, and 80 °C. As the data indicate in Fig. 11, the plates performed well. Cell resistance results (65–80 mΩ cm^2) are similar to those observed with 100% graphite plates (60–75 mΩ cm^2; not shown).
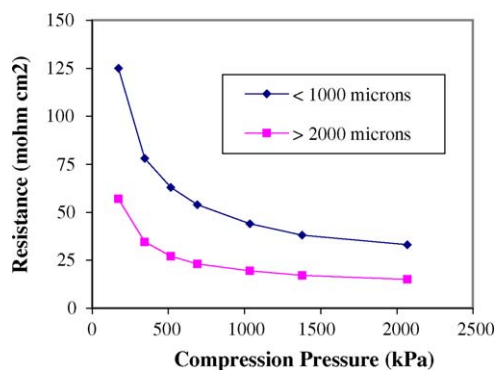

Fig. 9. Effect of EG particle size on resistance (0.65 mm thickness, 10 v/o EG/epoxy).
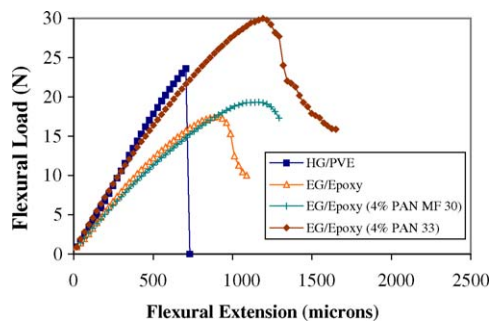

Fig. 10. Flexural property enhancement with the addition of reinforcing PAN graphite fibers (25 v/o total graphite concentration, 1.0 mm thick).
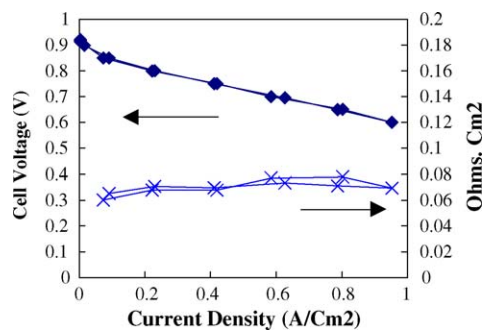

Fig. 11. Fuel cell performance of machined 25 v/o EG/epoxy anode and cathode plates (80 °C, 25 psig, 100% RH).

5. Summary

The use of expanded graphite particles as a conductive filler for composite bipolar plates shows great promise. Automotive fuel-cell plate resistance targets can be met at low graphite loadings (e.g., 20 v/o EG), which, in turn, help maintain low plate costs (EG is more expensive than graphite powders typically used) and provide the opportunity to enhance plate strength and ductility with the addition of other reinforcing fillers. Moreover, the thickness of the EG-filled plate material is not limited by H_2 permeation rates. Plates as thin as 0.5 mm can be used.

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