The shift from electric vehicle (EV) applications to hybrid EV (HEV) applications during the beginning of this decade has changed the focus of the Li-ion research away from a high-energy application toward a high-power-pulse application. The EV application leads toward high-power-pulse application. The EV application leads toward lithium polymer cells and high-capacity alloy anodes, both of which face serious challenges. The HEV application stresses the need for high transport via thin dimensions. Power performance can be significantly improved with modifications of the electrode design but has been done so at the expense of the inherently high-energy density of Li-ion cell chemistry. Both applications require advanced electrode engineering in order to meet stringent targets. Now, with the advent of the plug-in hybrid application (PHEVs) and, to some extent, rechargeable power tools, there will be a call for higher energy at moderate power levels. This new requirement of Li-ion cells will demand a comprehensive consideration of the design of electrodes. Beyond power and energy requirements, there is a 10–15 year lifetime requirement and a 3000–5000 cycle life requirement that will add additional burdens to proper electrode design. In order to achieve the lifetime requirement, it is understood that impurities that lead to side reactions must be minimized; moreover, the cycle life challenge will require both robust materials and robust electrode configurations. It is envisioned that the additional cost of removing impurities and modifying materials can be offset by the savings gained over the extended 10–15 battery life span.

Early papers centering on binder or conductive additive properties were typically in regard to performance in polymer or gel systems. Very few systematic studies have been done on polyvinylidene difluoride (PVDF) acting solely as a binder in commonly available organic electrolyte systems, such as LiPF$_6$ in carbonates, such as ethylene carbonate (EC) and diethyl carbonate (DEC). There is very limited reporting on power performance of lithium-ion cells with regard to systematic variations of binder and conductive additives in a broader range. Most of the early papers report the effects of electrode conductivity or capacity improvement with the changes in the level of inert materials of the ratio at a low to moderate current density (0.1–1 C). In most cases, the active materials are made in-house with lithium metal as the counter electrode. Some of the surface coating techniques on cathode materials involve the use of water-based systems. This is inherently problematic because most of the metal oxidizes react with water to form electroinactive surface layers, significantly increasing interfacial impedance. Minimizing the active material exposure to moisture is a key to designing electrodes with high-power performance. In our capacity, our electrode material mixing, coating, drying, and cell-fabrication facilities are under the most stringently controlled conditions of argon (Ar)-filled glove boxes. The active materials are purchased from high-volume manufacturers and subject to tight industrial quality-control processes. All of the electrodes discussed here can be discharged to the manufacturer’s specifications. The overall cell impedance in the cells we fabricate is comparable to that of industrially fabricated high-power 18650 cells. Coin cells can be cycled through hundreds of deep-discharge cycles at ambient conditions with minimal cell capacity fade. Our superb environmental controls and fabrication techniques allow us to study power performance issues with great confidence. We feel that an in-depth study of the contribution of the acetylene black (AB) nanoco nductive additive and PVDF polymer binder in a standard lithium-ion cell system is important and we are fully understanding the contributions of these components. We recently reported that the interfacial impedance dominates the lithium-ion cell impedance and the power performance is improved with certain inactive material concentrations. We demonstrated that increasing the PVDF content in a cathode electrode may lead to decreased cell impedance and that the cell impedance could be decoupled from the electronic conductivity of the cathode electrode. This current work is to demonstrate the cell performance changes as a function of the ratio of AB to PVDF, and to map out the performance spectra of AB/PVDF to active material concentration.

Although many different types of conductive additives have been developed for lithium-ion cells, including carbon fibers and large graphites, there is no report of the AB being replaced as conductive additive in commercial cells. Although cost may be the main reason why manufacturers have shied away from these other materials, there is an inherent advantage of AB nanoparticles that is different from other types of nanoparticles, because AB forms extended network structures to provide both conductivity and superb mechanical properties at low loadings. With regard to the polymer, there are several choices for binder materials, from fluorinated polymers to water soluble latex binders. As far as we know, PVDF is still a premium choice for lithium-ion cathode in the commercial cells. The current study therefore focuses on the AB and PVDF system to
demonstrate the functions that AB and PVDF play in the vast majority lithium-ion cells and the possible direction to take to optimize these electrodes for PHEV applications.

**Experimental**

**Materials.**— Battery-grade AB with an average particle size of 40 nm, a specific surface area of 60.4 m²/g, and a material density of 1.95 g/cm³ was acquired from Denka Singapore Private Ltd. PVDF KF1100 binder with a material density of 1.78 g/cm³ was supplied by Kureha, Japan. Anhydrous N-methylpyrrolidone (NMP) with <50 ppm of water content was purchased from Aldrich Chemical Co. Active cathode material LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂, with a particle size of 10 µm, a specific surface area of 0.78 m²/g, and lattice density of 4.73 g/cm³, was a gift from Toda, Japan. The manufacturer-suggested specific capacity is 173 mAh/g when cycled between 3 and 4.1 V. The active material was shipped under dry conditions from the manufacturer and stored in an Ar-filled glove box with oxygen content of <0.1 ppm and dew point below −80°C. AB and PVDF powders were dried at 120°C under 10⁻² Torr dynamic vacuum for 12–24 h. The dried AB and PVDF powder were also stored in an Ar-filled glove box. AB/PVDF mixtures were made by dissolving 5 g of PVDF in 95 g of anhydrous NMP. A amount of AB was dispersed in the PVDF polymer solution to meet the desired AB/PVDF ratios. To ensure the thorough mixing of the AB nanoparticles into the polymer solution, sonification was used. A Branson 450 sonicator equipped with a solid horn was used. The sonication power was set at 70%. A continuous sequence of 10 s pulses followed by 30 s rests was used. The sonic dispersion process took ca. 30 min. The slurry properties for all AB/PVDF in NMP were constant after 20 min of sonification. Slurries with active cathode material were made by adding the targeted amount of LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂ active material to the freshly premixed AB/PVDF/NMP slurry. The cathode mixture was homogenized using a Polytron PT-350 to a homogenizer at 3000 rpm for 15 min until a viscous slurry was acquired. All of the mixing processes were performed in Ar-filled glove boxes.

**Film and electrode casting.**— In order to understand the conductive behavior of the AB/PVDF composite system, specific amounts of AB were dispersed in PVDF-NMP solutions and cast into thin films. The film compositions extend from 0.1:1 to 1:1 AB/PVDF by weight. AB/PVDF films for four-point probe dc conductivity measurements were cast onto glass surfaces. Among the AB/PVDF ratios, 0.2:1, 0.4:1, 0.6:1, and 0.8:1 AB/PVDF ratios were chosen to mix with LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂ active material in the fabrication of electrodes. At 0.2:1 AB/PVDF, slurries made of 1:2, 2.4, 4.8, 9.6, and 24% (AB + PVDF) with the rest being active material were cast into electrodes. At 0.4:1 AB/PVDF, slurries of 2.8, 11.2, and 21% (AB + PVDF) were cast into electrodes. At 0.6:1 AB/PVDF, slurries of 3.2, 12.8, and 24% AB + PVDF were cast into electrodes. At 0.8:1 AB/PVDF, slurries of 3.6, 9, 18, and 27% (AB + PVDF) were cast into electrodes. All electrode laminates were cast on to a 30 µm thick battery-grade Al sheet using a Mitutoyo doctor blade and a Yoshimitsu Seiki vacuum drawdown coater to roughly the same loading per unit area of active material. The films and laminates were first dried under infrared lamps for 1 h until most of the NMP was evaporated and they appeared dried. The films and laminates were further dried at 120°C under 10⁻² Torr dynamic vacuum for 24 h. The film and laminate thicknesses were measured with a Mitutoyo micrometer with an accuracy of ± 1 µm. The typical thickness of an AB/PVDF film is ca. 20 µm with an average density of 1.2 g/cm³. The thickness of the typical AB/PVDF LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂ film is ca. 50 µm with an initial porosity of ca. 50% after drying. The electrodes were compressed to 35% porosity before coin cell assembly using a calender machine from International Rolling Mill equipped with a continuously adjustable gap.

**Coin cell fabrication and testing.**— Coin cell assembly was prepared in standard 2325 coin cell hardware with LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂ films as the active material. The laminates were prepared with either 0.2:1, 0.4:1, 0.6:1, or 0.8:1 AB/PVDF ratios with different active material loadings. A 1.26 cm disk was punched out from the cathode laminate for use in the coin cell assembly. Detailed information regarding the different laminates and the cell configuration are listed in Table I. The MCBM10-28 graphite-based active material was used in making the counter electrode. The MCBM10-28 negative electrodes have 10% excess capacity per unit area compared to the corresponding cathodes of interest, and were compressed to 35% porosity. The counter electrodes were cut to 1.47 cm diam disks, which is physically larger than the cathode to prevent lithium deposition at the electrode edges at high charge rates. The cathode electrode was placed in the center of the outer shell of the coin cell assembly and two drops (at ca. 0.02 mL/drop) of 1 M LiPF₆ in EC:DEC (1:1 weight ratio) electrolyte purchased from Ferro Inc. were added to wet the electrode. A 2 cm diam of Celgard 2400 porous polyethylene separator was placed on top of the cathode electrode. Three more drops of the electrolyte were added to the separator. The counter electrode was placed on the top of the separator. Special care was taken to align the counter electrode symmetrically above the cathode. A stainless steel spacer and

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<th>Act. mater. loading (mg/cm²)</th>
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¹ Total means weight of the electrode including AB, PVDF and LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂.

**Film imaging.**— The surface morphologies of 0.2:1, 0.5:1, and 0.8:1 AB:PVDF ratio films were imaged by field-emission scanning electron microscopy (FESEM) with a JEOL FESEM set at 5 kV. The internal morphology was imaged by transmission electron microscopy (TEM) with a JEOL 200CX microscope operating at 200 kV. A RMC Boekeler PR XL ultramicrotome was used to prepare 50 nm thin sections of the films. Samples were embedded in low-viscosity Spurr’s epoxy for added stability when sectioning. Films were cryomicrotomed at −100°C using a cryogenic attachment and a glass knife. After the samples were sectioned, they were then carbon coated to help dissipate charge.

**Four-point probe dc testing.**— The conductivities of both AB/PVDF and AB/PVDF/active material films were measured using a four-point probe. The dc conductivity measurement was performed using a Jandel equal-distance linear four-point probe apparatus with a Solartron 1286 Electrochemical Interface and a CorrWare software package. A direct current was applied between the two outer probes; the voltage was registered between the two inner probes.

### Table I. Compositions and configurations of the cathode electrodes used to fabricate the lithium-ion coin cells.

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¹ Total means weight of the electrode including AB, PVDF and LiNi₀.₈Co₀.₁₅Al₀.₀₅O₂.
Figure 1. (Color online) DC conductivities of the AB and PVDF composites films (100% legend represents the bulk film; 10, 3, and 1% represent the uniformed reduction of conductivity to these values).

Results and Discussion

Conductivity log plot of AB/PVDF composites.— The interaction of the inactive materials AB and PVDF plays a critical role in providing functionality to an electrode. AB, although only present at a level of a few weight percent in the electrode composite, has significant surface area and therefore plays a dominant role in the interaction with the polymer binder. A semilog plot of the conductivity vs AB content given in Fig. 1 points to two conductivity regions. The first region is the low-AB content region between 0.1 and 0.2:1 AB:PVDF, where the conductivity is quite low. This region corresponds to the gradual formation of an AB three-dimensional network structure within the PVDF phase. The conductivity improves rapidly because this network is fully formed at ca. 0.2:1 AB:PVDF. The second region is the conductivity plateau region, where the conductivity changes more slowly with increase of AB content. This region corresponds to the fully formed AB network. The conductivity is controlled by the conductivity of the junctions between the AB aggregates. Addition of AB plays a competing role to increase the conducting material but to weaken the conductive network. The AB/PVDF composite forms a conductivity plateau from 0.2:1 ratio and peaks at 0.8:1 when wetted by electrolyte solvent, then drops with further increase of AB content due to insufficient binder to maintain the AB network structure. Any formulation variations or process inhomogeneities will cause significant conductivity fluctuations in the electrode in the first conductivity region. The plateau region has a relatively high, stable conductivity in a range of compositions and is the focus of this work. There are two limit compositions in this plateau region: 0.2:1 AB:PVD ratio, which gives the highest electronic conductivity in the AB/PVDF composites. Further increase in the concentration of the AB leads to a decline in the electronic conductivity as well as major reductions of the mechanical strength and is therefore not of interest. In this paper, we also evaluated two intermediate compositions: 0.4:1 and 0.6:1 AB:PVDF.

The diamond points in Fig. 1 correspond to the measured conductivity of the AB/PVDF films at different AB:PVDF ratios, and the solid line connecting the diamond points is for visual aid. In a working electrode, the active material is mixed into the conductivity AB/PVDF composite. Because of the semiconductive nature of the oxide active materials, the active material particles mostly take up space and create tortuosity but do not significantly contribute to the electronic conductivity of the composite. Therefore, the electronic conductivity of the film will decline with the addition of active material. Assuming the AB/PVDF distribution is uniform, the dotted lines account for the conductivity proportionally to the (AB + PVDF) volume fraction raised to a factor that accounts for tortuosity. The electrolyte resistance dominates the cell high-frequency cutoff impedance also known as bulk impedance when the separator between the positive and negative electrode is 25 μm thick.

SEM and TEM images of the AB/PVDF composites.— The morphology of the AB/PVDF composites at 0.2:1, 0.5:1 and 0.8:1 AB:PVDF ratios is studied in detail by SEM and TEM imaging techniques (Fig. 2 and 3). Large domains of PVDF phases exist at the 0.2:1 AB:PVDF ratio as can be visualized by SEM and TEM images; although, as discussed, this concentration still allows for a fully formed electronic network throughout the film. Increases in the AB concentration tends to reduce the PVDF islands to ever smaller domains. At the 0.8:1 ratio, the individual domains of PVDF polymer are very difficult to visualize. This is a result of the strong association between the AB surface and the PVDF polymer.
were carefully designed to have similar capacities per unit area of an AB/PVDF composite to the cell performance. The electrodes tend to have higher capacity; therefore, a separate anode electrode. However, low loading of the MCMB10-28 graphite to minimize the variation from the counter electrode. At each AB:PVDF ratio, different fractions of gate the contribution of changing the AB/PVDF loading on electrode performance. At each AB:PVDF ratio, the polymer phase is closely associated with the AB particles with little excess polymer visible and a maximum in conductivity. Because of the strong interaction of the PVDF with AB, there is limited PVDF polymer available to provide continuous adhesion through the film, thus increases in AB:PVDF of >0.8:1 leads to oversaturation of the polymer, and hence loss of mechanical integrity and a reduction in conductivity.

Cell performance.—Slurries of AB/PVDF at ratios of 0.2:1, 0.4:1, 0.6:1, and 0.8:1 were used in the fabrication of electrodes with different \( \text{LiNi}_{0.80}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2 \) active material loadings to investigate the contribution of changing the AB/PVDF loading on electrode performance. At each AB:PVDF ratio, different fractions of active material were added to investigate the integrated contribution of a AB/PVDF composite to the cell performance. The electrodes were carefully designed to have similar capacities per unit area (Table I). Counter electrodes were punched from the same laminate of MCMB10-28 graphite to minimize the variation from the counter electrode. However, low loading of the (AB + PVDF) content electrodes tend to have higher capacity; therefore, a separate anode laminate is used for all low loading of the (AB + PVDF) laminates. The 0.2:1 and 0.8:1 AB-PVDF-based films are the outer limits of the ratios we tested; the electrochemical performances of the two compositions follow different trends as the active material content changes.

We first discuss the impact of the binder-poor composition of 0.8:1 AB:PVDF on the cell performance provided in Fig. 4. The minimum required to provide enough adhesion between particles is 3.6% of the (AB + PVDF). The integrity of the electrode is adversely affected with less (AB + PVDF). The EIS tests were done at 40% of DOD (at cell voltage of 3.82 V) from \( 10^{-2} \) to \( 10^{5} \) Hz frequency range (Fig. 4a). Depressed semicircle reflects the charge transfer impedance of the cell comes from the charge transfer impedance, while the bulk resistance is fairly constant with change of the (AB + PVDF) loading. Because the anode remains the same for all compositions, the change of the charge transfer impedance is due to the changing composition of the cathode. The apex frequencies of the semicircles are presented in Fig. 4a for both semicircles at 27% (AB + PVDF) loading. When the charge transfer impedance grows, the two semicircles combine into a single, depressed semicircle, making it difficult to distinguish the apex of the charge transfer impedance phenomenon of either electrode. The positions of these two frequencies were also labeled on the semicircle of other loadings of this series. The charge transfer impedance increases in the cathode with decreases of the amount of (AB + PVDF) composite. The increased charge transfer impedance directly correlates to the decreasing binding force and aggregation of the AB particles in the electrode. The binder is also adsorbed to the active material surface leaving lesser binder available for adsorption to the AB conductive additive. The deficit of binder in the electrode composite contributes to the uneven distribution of the AB additive leading to the impedance rise at high active material loadings. The full-cell HPPC results are in very good agreement with the EIS results, showing a high area-specific impedance (ASI) at low (AB + PVDF) loading and a low ASI at high loading.

We now discuss the impact of the binder-rich composition at 0.2:1 AB:PVDF ratio on the laminate properties and cell performance (Fig. 5). In this composite, the carbon agglomerates into strands of carbon surrounded by PVDF and there are regions of just PVDF as shown in the TEM (Fig. 3a). Laminates of 1.2% (AB + PVDF) with the rest of the electrode as \( \text{LiNi}_{0.80}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2 \) active material can be made with adequate mechanical integrity. Impedance tests were performed at the similar conditions as those performed of laminates of 0.8:1 AB:PVDF. This time, however, the impedance systematically increases as the amount of (AB + PVDF) increases. The apex frequencies of the semicircles are presented in Fig. 5a. Only one depressed semicircle can be seen in this series of laminates. The trend of the impedance change of 0.2:1 AB:PVDF is just opposite to the 0.8:1 AB:PVDF series. The difference in the impedance trend is due to a different mechanism as to where the impedance originates. The more (AB + PVDF) at 0.2:1 AB:PVDF, the harder the coating of PVDF on each of the active material particles. At high binder to AB ratios, the excess binder is

Figure 3. TEM images of the AB/PVDF composites: (A) AB/PVDF = 0.2:1, (B) AB/PVDF = 0.5:1, and (C) AB/PVDF = 0.8:1 (Scale bars: 200 nm).

Figure 4. (Color online) Impedance and ASI of \( \text{LiNi}_{0.80}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2 \) graphite cells at AB:PVDF = 0.8:1: (A) EIS test results and (B) HPPC test results.

Figure 5. (Color online) ASI impedances of \( \text{LiNi}_{0.80}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2 \) graphite cells at AB:PVDF = 0.2:1: (A) EIS test results and (B) HPPC test results.
attracted to the active material, leading to encapsulation of individual particles. This results in an increase of impedance with increase in (AB + PVDF) due to the lithium-ion blocking effect of the PVDF polymers. Because 0.2:1 AB:PVDs has excess PVDF to provide adhesion, electrodes at very low (AB + PVDF) loadings (e.g., 1.2%) can be made with good mechanical strength. AB loading as low as 0.2 and 98.8% active material composite in the electrode is feasible with acceptable rate performance as seen in full-cell HPPC performance shown in Fig. 5b. This may open a new direction in the formulation of the power-based cells where energy density is equally important.

The 0.2:1 and 0.8:1 AB:PVDs ratios represent either too much binder or too little binder when the active material content varies. Both of them represent extreme conditions that the AB/PVDF functions in a lithium-ion cell environment. The 0.4:1 and 0.6:1 AB:PVDs ratios are in between the two extremes that may yield cell behavior that is of interest for more broad-based formulation requirements. The impedance trend seen in the 0.4:1 AB:PVDs ratio lithium-ion cell series is similar to the one in 0.2:1 series as shown in Fig. 6. Based on the electron microscopy information, the PVDF is still dominant in the 0.5:1 AB:PVDs composite. An ion-blocking effect exerted by PVDF phase is still dominant with an increase in the amount of (AB + PVDF) in the composite. However, the impedance increase is less prominent with increase of (AB + PVDF) compared to the 0.2:1 AB:PVDs series. A further increase of the AB:PVDs ratio to 0.6:1 appears to further stabilize the interfacial impedance regardless of the (AB + PVDF) content, as shown in Fig. 7. Very small changes of the interfacial impedance and full-cell ASI come with the change of (AB + PVDF) content in the 0.6:1 AB:PVDs series.

Conclusions

A complete picture of AB/PVDF contribution to the lithium-ion cell power performance was mapped out. Because the bulk impedance is very similar for the cells due to controlled designs, all of the charge transfer impedances are plotted in Fig. 8 to demonstrate the performance with changing composition. A few general conclusions can be drawn from this chart. At higher PVDF contents, such as 0.2:1 AB:PVDs, the active material particles are encapsulated by the AB/PVDF composite leading to an ion-blocking effect that dominates the cell resistant. This effect can be reduced by decreasing the total amount of (AB + PVDF) in the electrode. At high-AB content such as 0.8:1 AB:PVDs ratio, AB aggregation is the cause of cell-resistant growth when (AB + PVDF) loading decreases. An increase of the overall amount of (AB + PVDF) will significantly reduce the impedance. At 0.6:1 AB:PVDs ratio, the cell resistance is stabilized regardless of the (AB + PVDF) content. This means that neither the ion-blocking effect by the PVDF observed at 0.2:1 AB:PVDs ratio nor the AB aggregation at 0.8:1 ratio is dominant in 0.6:1 AB:PVDs combinations.

There is a trade-off in the electrode design to improve energy and power performance for a given active material. High loading of (AB + PVDF) at high AB:PVDs ratios may lead to electrodes with good power performance. Both AB and PVDF have low density compared to the metal oxide active materials. Therefore an increased (AB + PVDF) content leads to decreased volumetric energy density. An alternative choice for power performance is to exploit the low AB:PVDs ratio composition, where impedance decreases at decrease of (AB + PVDF) content. This direction of optimization may yield electrode that has high loading of active material with low impedance. This may satisfy applications that both energy and power are important such as in the plug-in HEVs.

Acknowledgments

This research was funded by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies of the U.S. Department of Energy under contract no. DE-AC02-05CH11231. Electron microscopy was performed at the National Center for Electron Microscopy, Lawrence Berkeley Lab, which is supported by the U.S. Department of Energy under contract no. DE-AC02-05CH11231.

Lawrence Berkeley National Laboratory assisted in meeting the publication costs of this article.

Figure 6. (Color online) Impedance and ASI of LiNi0.80Co0.15Al0.05O2/graphite cells at AB:PVDF = 0.4:1: (A) EIS test results and (B) HPPC test results.

Figure 7. (Color online) Impedance and ASI of LiNi0.80Co0.15Al0.05O2/graphite cells at AB:PVDF = 0.6:1: (A) EIS test results and (B) HPPC test results.

Figure 8. (Color online) ASI of the charge transfer impedance based on EIS data vs inactive materials weight (AB + PVDF) for lithium-ion cells.
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