Characterization of microporous separators for lithium-ion batteries

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Abstract

Several properties including porosity, pore-size distribution, thickness value, electrochemical stability and mechanical properties have to be optimized before a membrane can qualify as a separator for a lithium-ion battery. In this paper we present results of characterization studies carried out on some commercially available lithium-ion battery separators. The relevance of these results to battery performance and safety are also discussed. Porosity values were measured using a simple liquid absorption test and gas permeabilities were measured using a novel pressure drop technique that is similar in principle to the Gurley test. For separators from one particular manufacturer, the trend observed in the pressure drop times was found to be in agreement with the Gurley numbers reported by the separator manufacturer. Shutdown characteristics of the separators were studied by measuring the impedance of batteries containing the separators as a function of temperature. Overcharge tests were also performed to confirm that separator shutdown is indeed a useful mechanism for preventing thermal runaway situations. Polyethylene containing separators, in particular trilayer laminates of polypropylene, polyethylene and polypropylene, appear to have the most attractive properties for preventing thermal runaway in lithium ion cells. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Lithium-ion; Batteries; Microporous; Separators; Membranes

1. Introduction

Lithium-ion batteries are rapidly becoming the preferred choice for powering portable electronic products, particularly laptop computers and cellular phones [1,2]. This choice is primarily driven by the demand for batteries having higher energy densities than that offered by nickel cadmium (NiCd) and nickel metal hydride (NiMH) systems. The energy density for a lithium-ion battery is anywhere between 30–60% higher than that demonstrated by the nickel based systems. In addition, lithium-ion cells can also provide longer cycle life and higher voltages. Lithium-ion batteries operate on the same principle as lithium metal batteries but do not have many of the problems that are associated with the latter, particularly the unstable lithium metal interface. This is because lithium-ion technology utilizes lithium-intercalated or lithium-inserted compounds of graphite, amorphous carbon or low voltage metal oxides as the negative electrode instead of the highly reactive metallic lithium. The cathodes are usually higher

voltage metal oxides, chosen from a list that includes lithium cobalt oxide ($LiCoO_2$), lithium nickel oxide ($LiNiO_2$) and lithium manganese oxide ($LiMn_2O_4$). Interposed between the anode and the cathode is a porous sheet or film commonly referred to as the *separator*. The pores in the separator are filled with an ionically conductive liquid electrolyte—a solution of lithium salt in non-aqueous solvents. Electrode and separator combinations are wound into tight rolls to be fitted into rigid cylindrical or prismatic (rectangular) metal cans. The main functions of a separator are: (I) to isolate the anode and the cathode so that no electrons can flow between them within the cell and (II) to allow the ions that are contained in the liquid electrolyte unrestricted passage between anode and cathode.

The next generation of the lithium-ion batteries, commonly referred to as *lithium-ion polymer* [1], utilizes gel electrolytes—gels of liquid electrolytes with a gellable polymer such as poly(vinylidene fluoride) (PVDF). Lithium-ion polymer batteries are expected to provide battery makers with the flexibility for designing cells with unique shapes and sizes. Furthermore, plastic lithium-ion batteries may not require the rigid metal cans that are

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required for conventional lithium ions to ensure stack pressure between electrodes and separator. This allows one to pack the cell in flexible foil packaging material and achieve a significant improvement in gravimetric energy density of the battery.

In one version of the lithium-ion polymer, or *plastic lithium-ion*, battery that is being developed by Bellcore, the anode and the cathode are laminated onto either side of a gellable membrane [3]. Lamination is carried out in the presence of a plasticizer, which is subsequently extracted to give the required porous structure in the battery. Good adhesion between the electrodes and the membranes is possible because all three sheets contain significant amounts of a poly(vinylidenefluoride-co-hexafluoropropylene) copolymer that can be melted and bonded during the lamination step. Due to the presence of the plasticizer, the lamination can be done at relatively low temperatures.

Recently, there have been a number of reports of lithium-ion polymer batteries in which the gel electrolyte layer contains an inert microporous separator. These microporous separators are similar to the separators that are used in a conventional lithium-ion battery [4–7]. Gel-covered and/or gel-filled separators have some characteristics that may be harder to achieve in the separator-free gel electrolytes. For example, they can offer much better protection against internal shorts when compared to gel electrolytes and can therefore help in reducing the overall thickness of the electrolyte layer. In addition, the ability of some separators to shutdown at a particular temperature allows safe deactivation of the cell under overcharge conditions [8,9]. Until the appearance of truly robust liquid-free polymer electrolytes, inert microporous separators may be an important component of lithium-ion polymer batteries as well.

2. Experimental

2.1. Materials

Samples of separators were obtained from four different suppliers. Due to the confidential nature of Motorola's relationships with these suppliers their names are not disclosed. Instead, samples from the four suppliers will be referred to as A, B, C and D. Seven samples (A1–A7) were received from supplier A, two (B1 and B2) from supplier B, two (C1 and C2) from supplier C and one (D1) from supplier D. Table 1 gives a list of the separator samples that were studied, along with the composition and thickness values.

Mineral oil [CAS # 8020-83-5] was used for membrane porosity determination. It was purchased from Aldrich Chemical (Milwaukee, WI) and has a density of 0.86 g/ml. Porewick® [CAS # 86508-42-1], a non-volatile per-fluorinated liquid was used for pore size measure-

Table 1
Composition and thickness values of separators studied

Separator type	Composition	Thickness (μm)
A1	PP//PE//PP	25
A2	PP	25
A3	PP//PP	50
A4	PP	25
A5	PP//PP	50
A6	PE	25
A7	Surfactant coated PP	25
B1	PE	25
B2	PE	30
C1	PP	25
C2	PE	25
D1	PP/PE/PP	25

ments. Porewick® has a surface tension of 16 dynes/cm and was purchased from Porous Materials (PMI).

Prismatic Li-ion cells containing a $LiCoO_2$ cathode, a graphite anode and a PVDF coated polyolefin separator were used for studying the shutdown characteristics of different separators. Apart from variations in the separators, the cells were identical in all respects and are hence expected to have identical thermal transport properties. Further details about the cell assembly can be obtained from Ref. [7].

2.2. Methods

Pore dimensions were measured using a Model MFP1500A capillary flow porometer (CFP) manufactured by PMI. This instrument is designed to handle pressures of up to 500 psi. Most experiments described in this manuscript however were carried out at pressure values under 250 psi. In a typical CFP experiment, a porous membrane is filled with Porewick® and subjected to gas pressure. The pressure on the membrane is increased in a controlled fashion while the flow rate of the gas through the membranes is simultaneously monitored. Pore size information can be obtained from these two values as described later.

Membrane porosity was determined by a simple liquid absorption test. The liquid chosen for this test was mineral oil. Permeability studies were carried out using the *pressure drop test*, which was also carried out by utilizing the CFP. In the pressure-drop test, the separator is subjected to compressed gas pressure of around 100 psi in a closed sample chamber. Permeability information can be obtained by monitoring the rate at which the gas escapes through the pores of the separator.

A Rheometrics Scientific STA625 differential scanning calorimeter (DSC) was used to determine the melting points of the separators. Impedance measurements were made at a frequency of 1 kHz using a Tegam Model 252 Impedance meter. Mechanical measurements were carried out with a Rheometrics Scientific Minimat (MMT2000)

mechanical tester working in the tensile mode. The Minimat was equipped with a 200 N load cell.

3. Results and discussion

Commercially available Li-ion battery separators are made out of polyolefins like polyethylene and polypropylene. These materials have been found to be compatible with the cell chemistry and can be cycled for several hundred cycles without significant degradation in chemical or physical properties. Polyolefin separators have existed long before the advent of lithium-ion technology, either in the form of microporous membranes or as non-woven materials [10]. They have been and continue to be used in batteries with other chemistries, including lithium primaries and lead-acid. With the recent surge of activity in the lithium-ion polymer area, poly(vinylidenefluoride) homopolymers and copolymers have also emerged as suitable candidates for separator material [3]. Unlike the polyolefin separators, however, the PVDF ones are used in a partially or completely gelled state as discussed earlier in this manuscript.

Most separator manufacturing is done by some combination of extrusion and stretching/drawing [11]. This process allows one to achieve relatively low (≤ 75 μm) film thickness values. Furthermore, in some cases the stretching operation also creates the necessary porous structure in the membrane. For example, the pores in the Hoechst Celgard® line of separators are produced by a process that involves high melt-stress extrusion and elevated temperature annealing [11]. If multiple layers are desired in the separator element, co-extrusion and/or lamination processes may also become involved [12]. The stretching process gives the separators an anisotropy in mechanical properties. Fig. 1 shows the results from tensile tests that
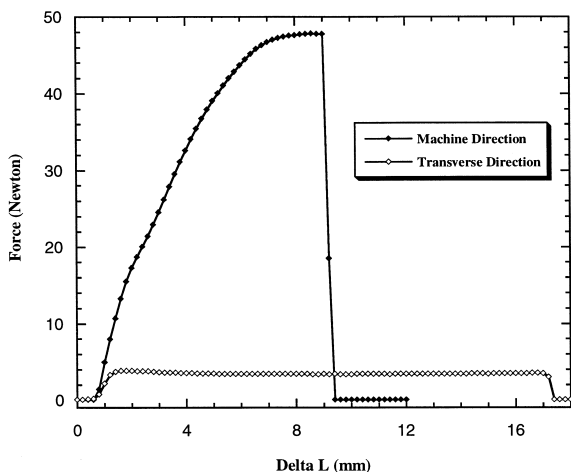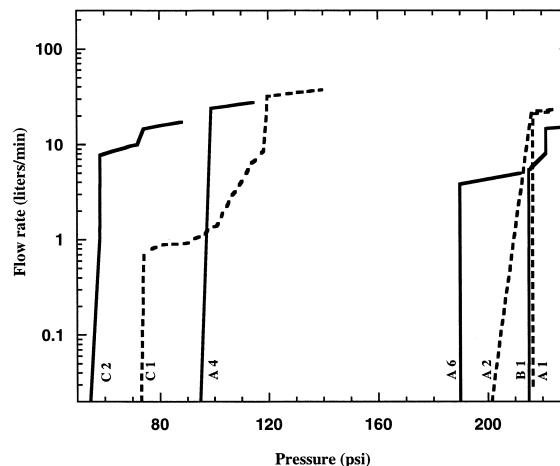

Fig. 2. Flow rate vs. pressure curves for commercial Li-ion battery separators.

were carried out on sample A2. As is clear from the results shown, the tensile strength for break is much higher for the sample in the machine (stretching) direction (~ 50 N) when compared to the transverse direction (~ 5 N).

Some separator manufacturers utilize plasticizers during the extrusion process. After extrusion the plasticizer is removed through an extraction process, to generate micropores in the film [13,14]. Plasticizers are particularly useful for making filler containing separators because they reduce the viscosity of the filler containing melt. Fillers are used to give the separator a skeletal structure, which prevents the pores and the film from shrinking or collapsing during plasticizer extraction. In addition, fillers could improve the mechanical strength of the separators, making them more effective for preventing electrical shorts. Filled separators are used mainly in lead acid batteries [13] and are also being considered for the lithium-ion polymer batteries [14,15]. Lithium-ion polymer battery separators can be processed either by extrusion or by solvent casting [14].

3.1. Pore dimensions

Fig. 2 shows the pressure vs. flow rate plot for liquid filled separators from three different suppliers. The curves in this plot can be used to collect information about the dimensions of the pores in the separator. The pressure, p, required to displace the liquid from a circular pore of diameter d is given by the equation:

$$p = C\frac{\tau}{d} \tag{1}$$

where τ is the surface tension of the liquid and C is a capillary constant [16]. As suggested by Eq. (1), liquid from the larger pores in the separator is expelled before the liquid from the smaller pores. The pressure required to expel liquid from the largest pore in the separator is usually referred to as the *bubble point pressure*, P_{bp}. The

Fig. 1. Tensile force at break for separator A2 in the machine direction and the transverse direction.

diameter, d_1, for the largest pore in the separator can hence be given by the relationship:

$$d_1 = C \frac{\tau}{P_{bp}} \qquad (2)$$

Pores in lithium-ion battery separators were detected in the pressure range 50–230 psi (see Fig. 2). In general, lower pressures are preferred for analysis since they are less likely to deform the pores. Porewick® was chosen as the wetting liquid for this reason. It has a lower surface tension relative to other possible candidates such as mineral oil or the alcohols and allows pore size detection at lower pressures (see Eq. (1)). Separators A1, B1, A2 and A6 have pore sizes in the range 0.03–0.05 μm. Separators A4, C1 and C2 have pore sizes in the range 0.09 to 0.12 μm. For any curve in Fig. 2, the height of the flow rate jump can be taken to be a measure of the number of pores detected at that pressure range. In most of the separators shown in Fig. 2, the largest pore diameter was found to be within 0.01 μm from the average pore diameter, suggesting relatively narrow distributions of pore sizes. Separator C1 showed the broadest distribution of pore sizes. The multi-step nature of the C1 curve suggests a bimodal pore size distribution.

Sub-micron pore dimensions are critical for preventing internal 'shorts' between the anode and the cathode of the Li-ion battery, particularly since these separators tend to be as thin as 25 μm. This feature will be increasingly important as separator manufacturers continue to make the membranes thinner. Thinner membranes are desired for increasing the energy density and decreasing the impedance of batteries, particularly those designed for portable electronic products. A more quantitative investigation of average pore diameters and pore size distributions is underway in our laboratory. These studies will be done by comparing the flow rate vs. pressure curve for a liquid-filled membrane with that of a liquid-free membrane. A wider range of liquids will be used for these studies and the results will be published as a separate manuscript.

3.2. Porosity

Fig. 3 shows the porosities for a number of polyolefin separators. The porosities were calculated by measuring the mass of mineral oil absorbed by the separator. A density value of 0.90 g/cm³ was used for polypropylene materials and 0.95 g/cm³ was used as the density value for the polyethylene separators. Trilayer separators were also assumed to have a materials density of 0.90 g/cm³.

Most of the separators that were studied had porosity values between 40 and 50%. Only separators C1 and C2 have higher porosities, around 55%. Significantly low porosity values were observed in the A7 separators. This may be either because the pores in these surfactant-coated separators are partially filled with the surfactant or because
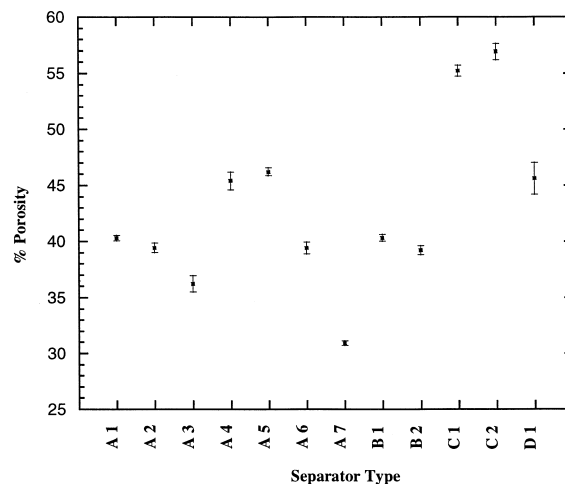

Fig. 3. Percent porosity for Li-ion battery separators as measured by liquid absorption.

the mineral oil was inefficient at wetting these modified materials.

High rate capability of a battery depends on, among other factors, the ionic conductivity of the liquid electrolyte filled separator. Typical non-aqueous liquid electrolyte compositions, of the type used in Li-ion batteries, demonstrate ionic conductivity values in the range, 10^{-2} to 10^{-3} S/cm. Although separators are effective in preventing electrical shorts between anode and cathode, their presence in between the two electrodes decreases the effective conductivity of the electrolyte, raising cell impedance. Some separators will reduce the ionic conductivity values by as much as one order of magnitude. However, separators enable the battery manufacturer to minimize the distance between the anode and cathode thereby reducing the impedance of the cell. For any given electrolyte, lower impedance values are observed when separators have higher porosities and/or lower thickness values. It is possible to commercially manufacture separators with porosities significantly greater than 50%. However, such materials may not be desirable since their puncture resistance and other mechanical characteristics tend to be poorer than those of the separators with lower porosities. In some cases separators with similar porosity and thickness values may contribute differently to the overall impedance of the battery. This would probably be expected if there is a significant difference in the tortuousity of the pores.

3.3. Permeability

A qualitative understanding of both porosity and tortuosity of the separator can be achieved by measuring the gas permeability of the separators. As described earlier, the permeability of the separators to gas was measured by using a novel pressure drop technique. In the pressure drop technique, a sample chamber containing the separator is pressurized to a predetermined 'upper pressure'. The
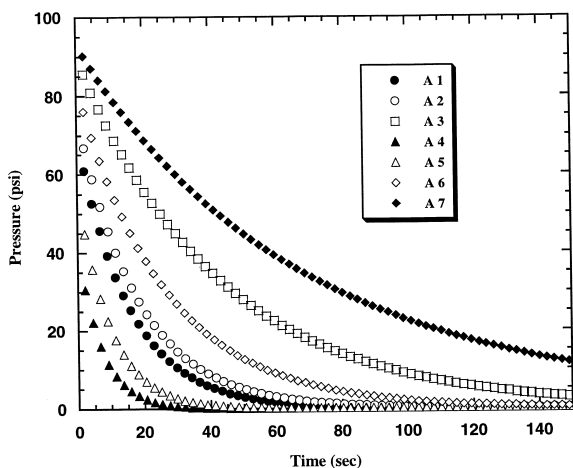
Fig. 4. Pressure drop curves for separators from supplier A.

chamber is then isolated from the gas source. As the gas in the chamber escapes through pores of the membrane, the chamber pressure drops. This pressure drop across the membrane is monitored as a function of time. Membranes with higher permeability demonstrate a more rapid drop in pressure than membranes with lower permeability. Fig. 4 shows the pressure vs. time curves for a number of separators manufactured by supplier A. The long pressure drop time for sample A7 suggests that this sample does indeed have a relatively low porosity value. Samples A4 and A5 have the shortest pressure drop times because of the higher porosity values in these separators. The bilayer separators A3 and A5 have slightly lower permeability values when compared to their single layer counterparts A2 and A4, respectively. Some separators with similar porosity numbers, like A1 and A2, have pressure drop curves that look fairly similar. However, other separators that have similar porosities, like A2 and A6, have significantly different pressure drop profiles. The pressure drop in separator A6 is much slower than the pressure drop in separator A2.

This suggests that the pores in separator A6 are significantly more tortuous than the pores in separator A2.

From the data in Fig. 4 it is possible to estimate the time required for the pressure across each of these separators to drop from 100 psi to an arbitrary lower value (20 psi). These times, henceforth referred to as the *pressure drop times*, are given in Table 2 for all the separators from supplier A. In summary, an increase in porosity and/or a decrease in tortuosity leads to higher permeabilities and lower pressure drop times.

The most commonly used test method for gauging permeability is the Gurley method [17]. This test is similar in principle to the pressure drop technique that is described above, except that it measures the time taken for a fixed volume of gas to be forced through a membrane at constant pressure. This time is frequently referred to as the *Gurley number*. The third column in Table 2 shows the Gurley numbers for separators from supplier A. These numbers are average values of the Gurley number range that was reported by the supplier in the product datasheets. No numbers were available for sample A7. For samples A1 to A6, the trend observed in the pressure drop times for the separators is identical to the trend in the Gurley numbers reported by the supplier in the product data sheets.

Fig. 5 shows the pressure vs. time curves for separators from suppliers B, C and D. The pressure drop times for these samples are also included in Table 2. Gurley numbers for these separators are not included however, since every supplier does not use the same conditions to do the test. Samples C1 and C2 have relatively low pressure drop times because of their high porosities. Samples B1 and B2 have higher pressure drop times when compared to samples A1 and A2, in spite of the fact that all four of these separators have porosity values of around 40%. Again, this suggests that the pores in samples B1 and B2 have higher tortuosity than the pores in samples A1 and A2.
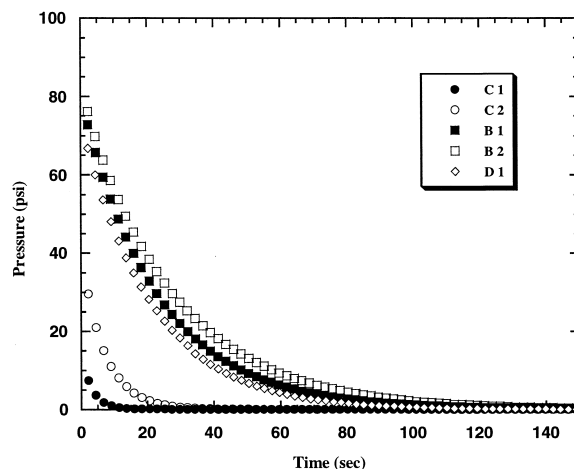
Table 2
Comparison of pressure drop times and Gurley numbers for microporous separators

Separator type	Pressure drop time (s)[a]	Gurley Number (s)[b]
A1	20	28
A2	24	33
A3	64	65
A4	5	10
A5	10	20
A6	37	37
A7	108	n/a
B1	31	–
B2	38	–
C1	5	–
C2	< 5	–
D1	28	–

[a]For pressure to drop from 100 to 20 psi (sample area = 1.25 in.^{2}).
[b]Average value of range from supplier A datasheets (sample area = 1 in.^{2}).

Fig. 5. Pressure drop curves for separators from suppliers B, C and D.

3.4. Thermal properties and shutdown mechanism

Like most batteries, Li-ion will generate heat if over-charged [18]. Above a threshold temperature the battery starts to 'self-heat' because of exothermic reactions within the components of the cell [18]. Accidental overcharging may occur during any one of the following situations: a malfunctioning charger, malfunctioning safety circuit, electrical misuse/abuse of battery pack. Separator 'shutdown' is a useful safety feature for preventing thermal runaway reactions in Li-ion batteries [8,9]. Separator shutdown is also a useful mechanism for limiting temperature and preventing venting in short-circuited cells [19,20]. Shutdown usually takes place close to the melting temperature of the polymer when the pores collapse, turning the porous ionically conductive polymer film into a non-porous insulating layer between the electrodes. At this temperature there is a significant increase in cell impedance and passage of current through the cell is restricted. This prevents further electrochemical activity in the cell thereby shutting the cell down before an explosion can occur. In principle most polyolefinic separators are shutdown separators since they melt at reasonable ($< 200°C$) temperatures. Fig. 6 shows the DSC thermograms for separators made out of (a) polypropylene, PP (b) polyethylene, PE and (c) a trilayer combination of the PP/PE/PP. Polypropylene separators melt at around 170°C and may be effective for shutting down cells that have relatively high thermal runaway temperatures. However, most commercial lithium-ion cells use separators with a lower shutdown temperature. For example polyethylene (PE) separators have shutdown temperatures between 130 and 140°C (see Fig. 6). In some cases, even after shutdown the cell temperature may continue to increase before actually beginning to cool. This is a likely scenario for a cell in a battery pack where heat dissipation is expected to be slow. Robust mechanical properties are therefore expected of the separator even above the shutdown temperature. If the separator undergoes a *meltdown*, i.e., if the mechanical properties of the
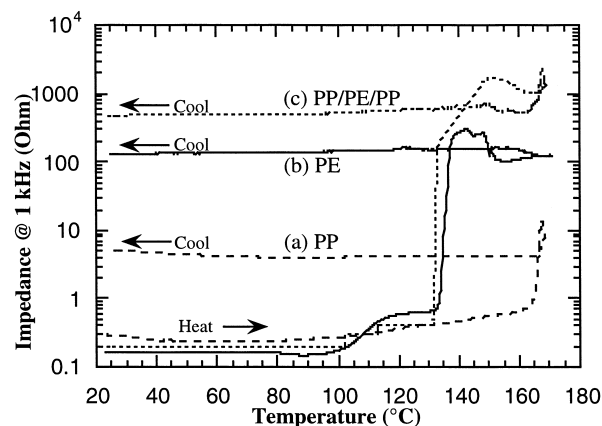
Fig. 7. Impedance vs. temperature curves for cells containing polyolefin separators: (a) polypropylene, (b) polyethylene and (c) polypropylene/polyethylene/polypropylene multiple layer.

separator deteriorate greatly after shutdown, the cell may internally short creating a safety hazard. More recently, some separator manufacturers have started offering products which have the porous PE layer sandwiched between two porous PP layers (see Fig. 6 for DSC of PP/PE/PP). The PE layer offers lower shutdown temperature while the PP provides the mechanical stability at and above the shutdown temperature. These multi-ply separators are expected to provide a wider shutdown window and may be safer than the single layer separators for some applications.

Fig. 7 shows the impedance vs. temperature curves for three cells each containing a different separator. All cells contained a $LiCoO_2$ cathode, a graphite anode and a PVDF coating on the separator. The cells were assembled and formed under identical conditions, as described in Ref. [7]. As indicated on the figure, impedance values for each cell were collected and plotted as the cells were heated up to 170°C and then cooled back down to ambient temperature. Fig. 7a corresponds to a cell containing a single layer polypropylene separator. The shutdown in this cell occurs at around 165°C, as is indicated by the sharp rise in impedance at this temperature. However, the impedance increase is approximately two orders of magnitude which may not be large enough for 'complete' shutdown i.e., cells may continue to overcharge albeit at lower rates and this will continue to be a safety hazard. As expected the shutdown temperature for the cell containing a polyethylene separator is at around 135°C (Fig. 7b). The impedance increases by about three orders of magnitude at this temperature. The lower shutdown temperature and the higher impedance of the cell after shutdown would make the polyethylene separator the preferred one for most lithium-ion cells. The shutdown behavior of the cell containing the PP/PE/PP multi-ply separator (Fig. 7c) is very similar to the one containing the PE separator. The impedance increase for this cell at the shutdown temperature is larger than that seen in Fig. 7b and may also be a result of the additional layers of PP that are present in the multi-ply separator.
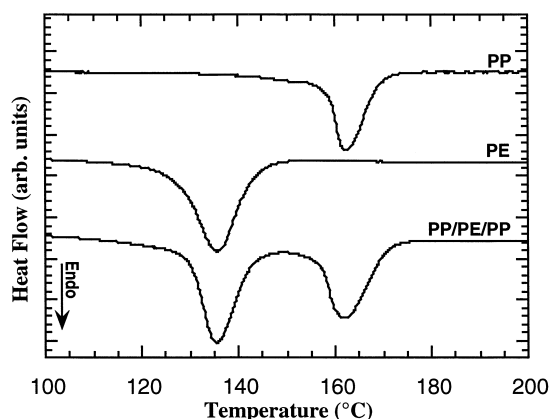

Fig. 6. DSC thermograms of representative polyolefin separators. PP represents polypropylene, PE represents polyethylene and PP/PE/PP represents a trilayer separator.

The voltage, current and skin temperature data of two prototype cells were collected during an overcharge test and plotted as a function of time in Fig. 8a and b. Both cells contained a LiCoO$_2$ cathode, a graphite anode and a PVDF coated polyolefin separator. The cells were assembled and formed under identical conditions as described in Ref. [7]. The overcharge test was performed by charging an optimally charged cell at a constant current value of 1.5 A. A 10 V (maximum) power supply was used to deliver the current. No additional voltage cut-off was used. The data shown in Fig. 8a is for a cell containing a trilayer PP/PE/PP separator. As is evident from the temperature profile in the figure, the cell starts to heat up because of the overcharge process. The heating rate increases significantly after 12 min. After about 19 min the applied current drops from 1.5 A to around 0.04 A. The current value stays at around 0.04 A even after the power supply voltage is increased to the 10 V limit. The low current value and high voltage value is an indication of high cell impedance arising from timely separator shutdown. After the drop in
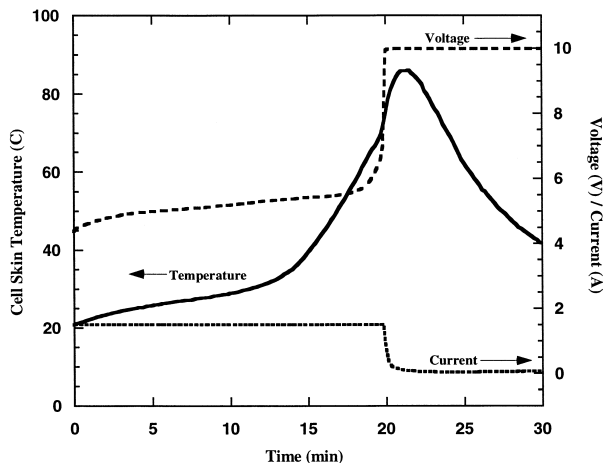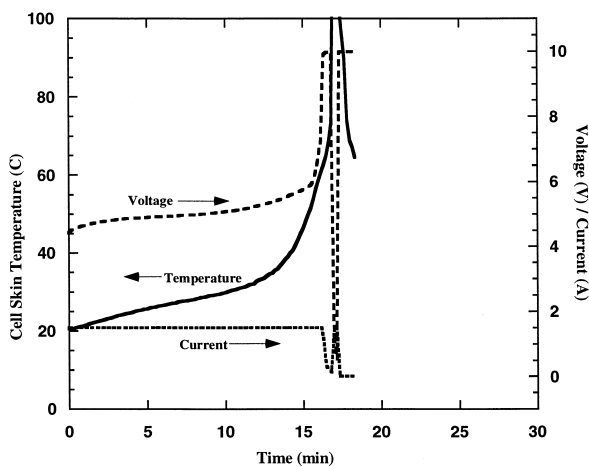
the current value, the cell skin temperature reaches a maximum and then begins to revert back to ambient. The maximum skin temperature is around 80°C. The internal temperature, however, is expected to be greater than 135°C. Typically, post-mortem analysis of a cell that demonstrated this kind of shutdown behavior revealed a deformed jelly roll in which the separator had been transformed into a translucent film. Both these observations indicate that separator shrinkage and pore closure is the most likely shutdown mechanism in these cells.

Fig. 8b shows the voltage, current and temperature vs. time profiles for a cell containing a single layer PP separator. As is evident from the temperature profile in this figure, the cell temperature reaches the thermal runaway point before the cell can actually shutdown. [1] This thermal runaway condition resulted in violent venting and ignition of the cell. This illustrates the potential safety advantage of separators with lower shutdown temperatures like the PE-based material. It is essential to point out, however, that not all PE containing separators are capable of demonstrating the same shutdown behavior. The ability of the PE-based separator to shutdown the battery is determined by its molecular weight, percent crystallinity (density) and process history. Materials properties and processing methods might need to be tailored so that the shutdown response is fast and complete. This optimization needs to be done without effecting the mechanical properties of the material in the temperature range of interest and is easier to do with the trilayer separators since one material is utilized for the shutdown response and another for the mechanical properties.

4. Conclusion

Porosity, pore-size, gas permeability, thermal properties and shutdown characteristics of commercial Li-ion battery separators were studied. Most lithium-ion battery separators were found to have porosity values between 40 and 50%. Average pore sizes were typically less than 0.1 μm. The relatively low porosity and pore size values allow these separators to be efficient at preventing internal shorts between the electrodes even when their thickness values are as low as 25 μm. The permeability of these separators to nitrogen gas was measured using a novel pressure drop method that is similar in principle to the Gurley method. As was expected, the pressure drop times were strongly influenced by the porosity and the size of the pores in the separators. Comparison of pressure drop times on separators with similar porosities gave qualitative information about the tortuosity of the pores in those separators. For

Fig. 8. (a) Overcharge test results for a cell containing a polypropylene/polyethylene/polypropylene trilayer separator. (b) Overcharge test results for a cell containing a polypropylene separator.

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[1] The applied voltage and the current for this cell after thermal runaway are 10 V and 0 A, respectively. This happens because the power supply is now connected to an open circuit, which was formed during cell disassembly.

one particular series of separators, the trend observed in the pressure drop times was in agreement with the trend in the Gurley numbers reported by the supplier. Separator shutdown properties were studied by measuring the impedance of a Li-ion cell containing the separator as a function of temperature. Overcharge tests were also performed to check the efficiency of the shutdown behavior. Polyethylene containing separators, in particular trilayer separators, appear to be efficient at timely prevention of thermal runaway in lithium ion cells. This is made possible by the lower melting/shutdown temperature of the polyethylene layer, and also the improved mechanical stability rendered to the trilayer construction by the polypropylene layer at the higher temperatures. The ability of separators to provide shutdown capability coupled with their ability to prevent shorts while maintaining thickness values of less than 30 μm also make these materials very attractive for use in the gel-electrolyte layer of lithium-ion polymer batteries.

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