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Journal of Power Sources 108 (2002) 8–14

JOURNAL OF POWER SOURCES

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A comparison of the electrode/electrolyte reaction at elevated temperatures for various Li-ion battery cathodes

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Received 24 September 2001; accepted 6 November 2001

Abstract

Differential scanning calorimetry (DSC) was used to compare the thermal stability of charged cathodes in 1 M $LiPF_6$ EC/DEC electrolyte. Seven possible cathode materials for lithium-ion batteries ($LiCoO_2$, $LiNiO_2$, $LiNi_{0.8}Co_{0.2}O_2$, $Li_{1+x}Mn_{2-x}O_4$, $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$, $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$, and $LiFePO_4$) were tested under the same conditions. Welded stainless steel DSC sample tubes, that ensured no weight loss during analysis, were used for these measurements, making them reliable. A consideration of these DSC results and the known electrochemical properties of the cathodes may assist the selection of the most suitable lithium-ion cathode material for use in a particular application. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: DSC; Cathodes; Lithium-ion

1. Introduction

The active electrodes of lithium-ion batteries are known to be reactive in the presence of electrolyte at elevated temperatures [1–3]. Therefore, lithium-ion cells must pass a number of safety tests before they can be shipped and marketed [4,5]. These tests are performed on full-sized lithium-ion cells and generally represent a full-cell response to the abuse condition [6]. From such tests it is difficult to determine which of the electrodes is responsible for thermal runaway, if it occurs. It is also not possible to study the reaction kinetics of the individual electrodes.

Many researchers in academic and small industrial laboratories are engaged in the search for new electrode materials for Li-ion batteries. In such environments, the production of full sized lithium-ion cells for safety evaluation is difficult since the manufacturing equipment and materials are not available. Commonly in such situations other thermal analysis techniques, that probe the individual electrode reactions, have been used. A number of researchers have performed experiments on individual electrodes in electrolyte to propose possible reaction mechanisms for the instability of lithium-ion cells at elevated temperatures [2,3,7,8]. These researchers have used differential scanning calorimetry (DSC) [2,3,7], thermal gravimetric analysis (TGA) [1] and accelerating rate calorimetry (ARC) [9,10] to analyse the stability of various components of a lithium-ion cell.

Recent work on modelling the response of oven-exposure tests on lithium-ion batteries has shown the importance of the cathode/electrolyte reaction [11]. Although the anode/electrolyte reaction is initiated first, the rapid reaction kinetics of the cathode/electrolyte reaction dominates the outcome of the oven-exposure test in cases where the specific surface area of the anode is small. If the temperature of the oven is high enough to initiate the cathode reaction substantially, it is very difficult to prevent a thermal runaway situation. Therefore, it is important to find the most thermally stable cathode available that also has the required electrochemical performance.

Currently, there are a number of reports that analyse the thermal stability of lithium-ion battery electrodes [2,3,8]. Many of these reports concentrate on one or two cathode types and there is often little consistency between reports from different research groups on the same materials. Here, we will present DSC results for seven possible cathode materials for lithium-ion batteries ($LiCoO_2$, $LiNiO_2$, $LiNi_{0.8}Co_{0.2}O_2$, $Li_{1+x}Mn_{2-x}O_4$, $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$, $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$, and $LiFePO_4$). The DSC analyses were performed using newly described preparative techniques that ensure no weight loss during analysis, which can be a concern in

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PII: S0378-7753(01)01013-8

judging the reliability of previous reports. It is the goal of this paper to show the thermal response of the cathodes analyzed, such that a manufacturer can select a cathode with appropriate thermal stability for a certain application.

2. Experimental

A list of the cathode samples analyzed, their suppliers and their respective surface areas is given in Table 1. $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$ and $LiFePO_4$ were synthesized in-house, while the other samples were donated by manufacturers.

$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$ was made by a co-precipitation process. $LiOH{\cdot}H_2O$(98%+, Aldrich), $Ni(NO_3)_2{\cdot}6H_2O$ (98%+, Fluka), $Co(NO_3)_2{\cdot}6H_2O$ (98%+, Fluka) and $Mn(NO_3)_2{\cdot}6H_2O$ (97%+, Fluka) were used as the starting materials. A 50 ml aqueous solution of the transition metal nitrates was slowly dripped (1–2 h) into 400 ml of a stirred solution of LiOH using a buret. This causes the precipitation of $M(OH)_2$ (M = Mn, Ni, Co) with a homogeneous cation distribution. The buret was washed three times to make sure that all the transition metal nitrates were added to the LiOH solution. The precipitate was filtered out and washed twice with additional distilled water to remove the residual Li salts (LiOH and the formed $LiNO_3$). The precipitate was dried in air at 180 °C overnight. The dried precipitate was mixed with the stoichiometric amount of $Li(OH){\cdot}H_2O$ and ground in an automatic grinder. Pellets about 5 mm thick were then pressed. The pellets were heated in air at 480 °C for 3 h. Tongs were used to remove the pellets from the oven and sandwich them between two copper plates in order to quench the pellets to room temperature. The pellets were ground and new pellets made. The new pellets were heated in air at 900 °C for another 3 h and quenched to room temperature in the same way. The sample discussed here is one of the samples reported in reference [12].

$LiFePO_4$ was prepared as follows. First, a stoichiometric mixture of $Fe(OOCCH_3)_2$ (Aldrich), $(NH_4)_2HPO_4$ (Aldrich) and 0.5 Li_2CO_3 (Aldrich), was ground together. The resulting mixture was then heated at 320 °C under argon for 12 h. The product was reground and pellets were pressed. The pressed pellets were heated to 550 °C under argon for 24 h. This synthesis temperature was selected based on the work of Yamada et al. [13]. X-ray diffraction showed pure

Table 1
Stock cathode materials used during the course of this paper

Stock electrode	Surface area (m^2/g)	Supplier
$LiCoO_2$	0.1	Manufacturer A
$Li_{1+x}Mn_{2-x}O_4$	0.6	Chemetals
$LiNiO_2$	0.7	FMC
$LiNi_{0.7}Co_{0.2}Mg_{0.05}Ti_{0.05}O_2$	0.8	FMC
$LiNi_{0.8}Co_{0.2}O_2$	0.3	FMC
$LiFePO_4$	15.2	Dahn
$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$	5.9	Dahn

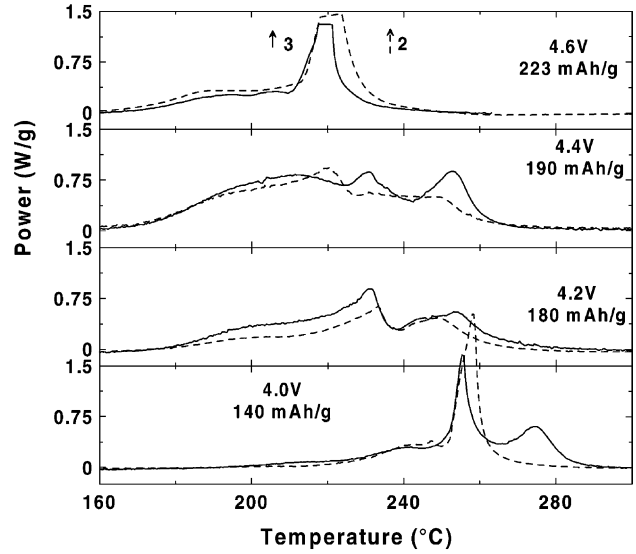

Fig. 1. DSC results for $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$ electrodes charged to the indicated potentials. Results for electrodes made using the "Bellcore" method (dashed line) and for electrodes coated on Al with PVDF binder (solid line) are shown.

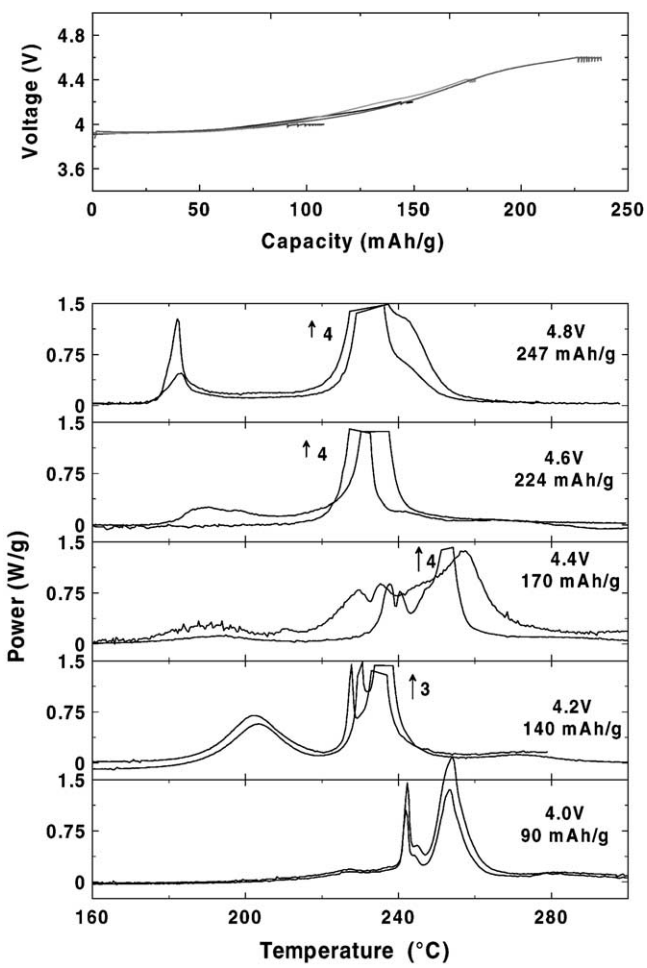

Fig. 2. DSC profiles of $LiCoO_2$ charged to indicated voltages (bottom panels). Duplicate scans are from a nominally identical sample. Top panel: charge characteristics of $LiCoO_2$.

$LiFePO_4$. The $LiFePO_4$ was then coated with carbon using the method reported by researchers at Hydro-Quebec [14]. A sucrose-water solution was prepared. $LiFePO_4$ was added to the solution and the water evaporated while grinding. Then the sucrose/$LiFePO_4$ mixture was heated to 550 °C under argon for 4 h to carbonize the sucrose. The amount of sucrose initially added was selected based on the target of approximately 3% by mass of carbon in the final product. Trials showed that 1 g of sucrose results in 0.23 g of carbon under these conditions. The carbon-coated $LiFePO_4$ still retained the literature X-ray diffraction pattern [15].

The electrodes (except for $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$) were prepared by combining 7% by mass, each of Super S Carbon Black (MMM Carbon, Belgium) and polyvinylidene difluoride (PVDF, 10% in *N*-methyl pyrolidinone (NMP), NRC) with the electrode powders. To the mixture an extra portion of NMP was added to form a slurry, which was then mixed for 10 min. The slurry was then coated on a piece of thin aluminum foil (16 μm thick). The electrode was then dried overnight in a 110 °C oven. The next day 13 mm diameter disks were punched.

For $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$ and $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$, ''Bellcore-type'' electrodes were prepared for electrochemical testing. About 500 mg of the sample was mixed with 10% (by weight) super S carbon black and 10% Kynar 2801 (VdF-HFP)(Elf-Atochem). The mixture was mixed with 2 g of acetone and 200 mg of dibutyl phthalate (DBP, Aldrich) to dissolve the polymer. After several hours of stirring and shaking, the slurry was then spread over a glass plate using a notch bar spreader to obtain an even thickness of 0.66 mm. The dry films were peeled off the plate and punched into circular disks with a diameter of about 13 mm. The punched electrode was washed several times in anhydrous diethyl ether to remove the DBP. The washed electrode was dried at 90 °C overnight before use.

The electrochemical cells were prepared in standard 2325 coin-cell hardware with a single lithium metal foil used as both the counter and reference electrode. Cells were assembled in an argon-filled glovebox, following previously described procedures [12]. The electrolyte used for analysis was 1 M $LiPF_6$ in EC/DEC (33/67).

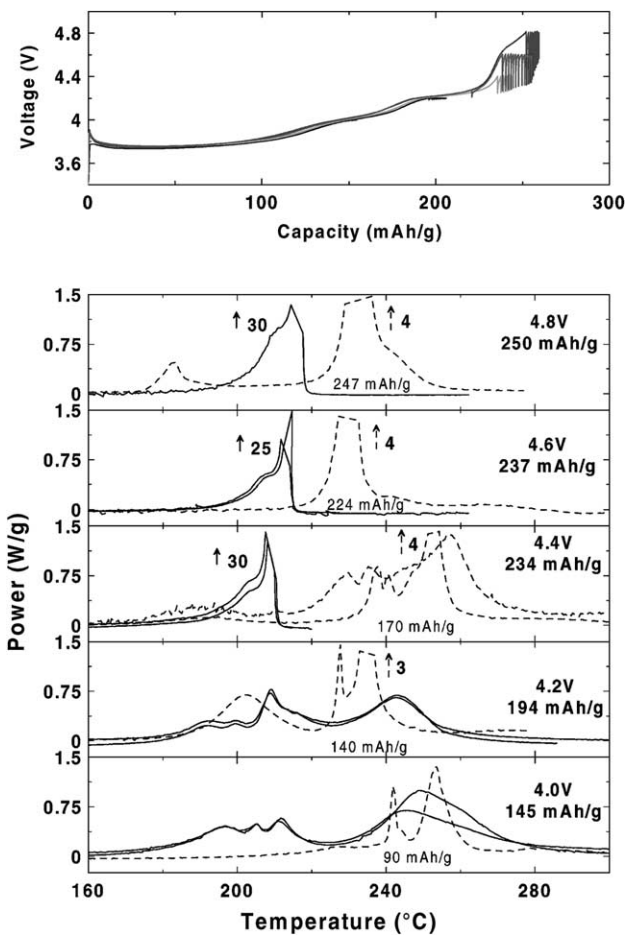

Fig. 3. DSC profiles of $LiNiO_2$ charged to indicated voltages (solid lines, bottom panels). Duplicate scans are from a nominally identical sample, while dashed lines are from the $LiCoO_2$ sample at the indicated voltage. Top panel: charge characteristics of $LiNiO_2$.

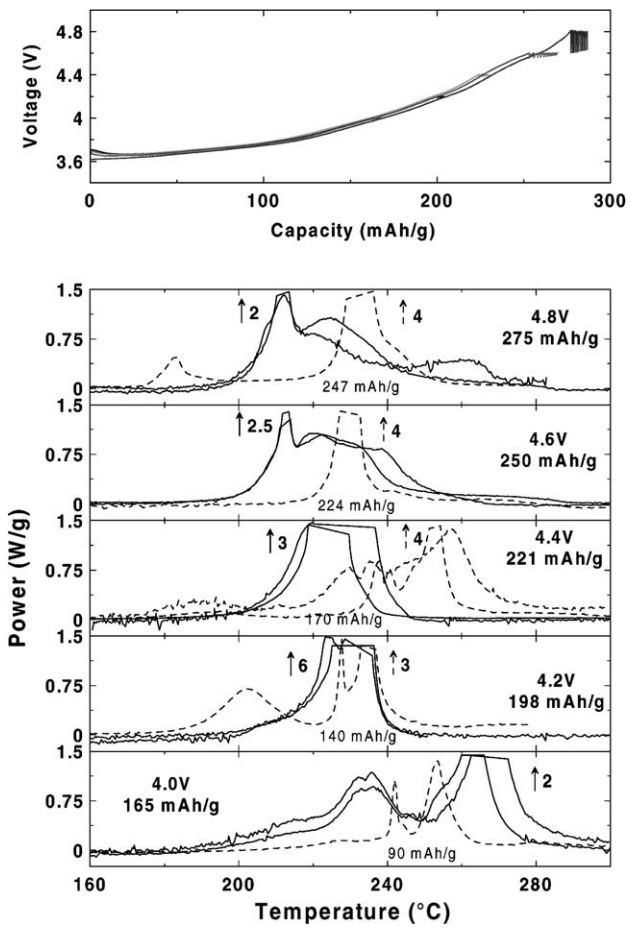

Fig. 4. DSC profiles of $LiNi_{0.8}Co_{0.2}O_2$ charged to indicated voltages (solid lines, bottom panels). Duplicate scans are from a nominally identical sample, while dashed lines are from the $LiCoO_2$ sample at the indicated voltage. Top panel: charge characteristics of $LiNi_{0.8}Co_{0.2}O_2$.

The cells were then removed from the glovebox and placed on the charging system (E-One/MoLi Energy). The cells were charged with a specific current of 14 mA/g. When the cell reached the desired voltage, the current was stopped for 30 min. Then the current was restarted, at 80% of the previous current, until the desired voltage was reached again. This cycling regime was repeated 12 times so that the cell stabilized near the desired voltage. After the charging cycle, the cell was removed and DSC sample cells were prepared in welded stainless steel tubes, as described previously [16]. Upon cell disassembly in the glovebox, the sample was removed from the aluminium current collector and transferred to the sample tube. The tube was sealed with no additional electrolyte or solvent added. The samples were analyzed in the DSC using a temperature scan rate of 2 °C/min.

3. Results and discussion

As described in the experimental section, there were two different electrode preparation procedures used. The "Bellcore" technique incorporates more binder in the electrode and this may affect the thermal response of the heated electrode. Fig. 1 shows DSC results for $LiNi_{0.7}Co_{0.2}Mg_{0.05}Ti_{0.05}O_2$ electrodes prepared by both the "Bellcore" (dashed line) and PVDF coating methods (solid line). There is no significant difference between DSC response as a function of binder type for the $Li_xNi_{0.7}Co_{0.2}Mg_{0.05}Ti_{0.05}O_2$ samples.

Fig. 2 shows the DSC results of $LiCoO_2$ charged to various voltages. The top panel of Fig. 2 shows the charge curves of the electrochemical cells used to prepare the electrodes for the DSC experiments. The specific capacity of the cell, from the top panel, is listed in the DSC panels under the corresponding voltage of the cell. The two curves in each of the DSC panels indicate duplicate samples from two different cells charged to the indicated voltage. Fig. 2 serves as the benchmark for further comparisons, since $LiCoO_2$ is the most widely used cathode material in commercial lithium-ion cells. The *y*-axis of all the DSC plots has been limited to 1.5 W/g. If the power of a sample exceeds 1.5 W/g dramatically, its profile has been clipped and the peak power has been indicated with an arrow near the peak.

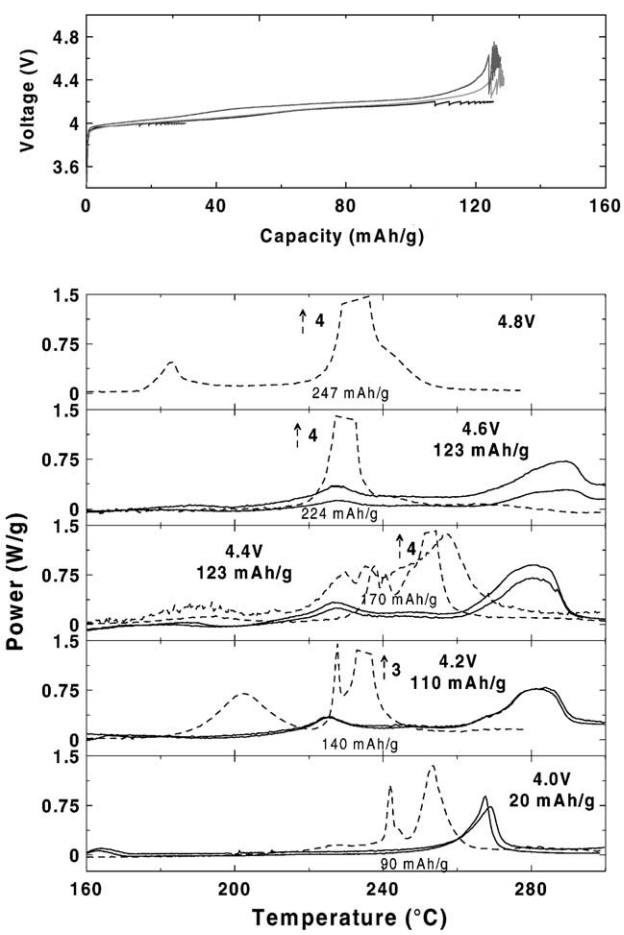

Fig. 5. DSC profiles of $LiMn_2O_4$ charged to indicated voltages (solid lines, bottom panels). Duplicate scans are from a nominally identical sample, while dashed lines are from the $LiCoO_2$ sample at the indicated voltage. Top panel: charge characteristics of $LiMn_2O_4$.

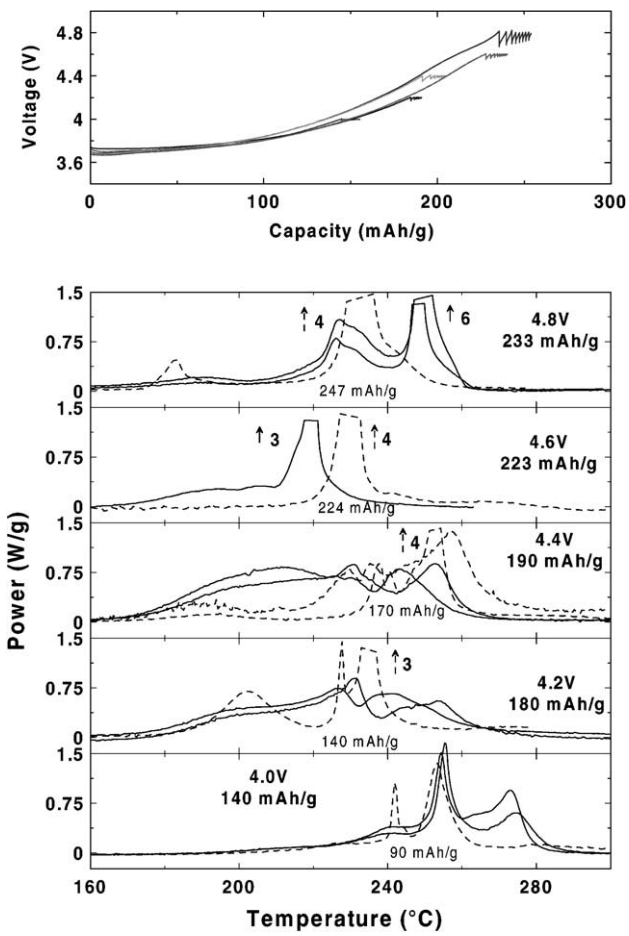

Fig. 6. DSC profiles of $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$ charged to indicated voltages (solid lines, bottom panels). Duplicate scans are from a nominally identical sample, while dashed lines are from the $LiCoO_2$ sample at the indicated voltage. Top panel: charge characteristics of $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$.

Fig. 2 shows that as $LiCoO_2$ is charged to higher voltages, the electrode becomes more thermally unstable. It is the goal of all manufacturers to optimize electrochemical properties (cycling characteristics, potential range, rate capability, capacity), thermal stability and cost and thus this thermal stability comparison will be useful. The surface area of the $LiCoO_2$ tested here is very low (0.1 m^2/g) so these results are probably close to the "best case" for Li_xCoO_2 in the selected electrolyte. Samples of $LiCoO_2$ with larger surface area are less stable in our experiments.

Electrodes prepared from $LiNiO_2$ have long been known to be thermally unstable [1], but the favourable electrochemical performance of $LiNiO_2$ and its potentially low cost compared to $LiCoO_2$ have ensured its continued study and possible use. Fig. 3 shows the DSC profiles of $LiNiO_2$, charged to various voltages in 1 M $LiPF_6$ EC/DEC (33/67). The results for $LiCoO_2$ are shown as the dashed lines for comparison and the $LiCoO_2$ capacity is shown near its respective profile. $LiNiO_2$ demonstrates much more thermal instability than $LiCoO_2$, since the Li_xNiO_2 samples liberated more heat and the onset of thermal instability usually occurred at a lower temperature than Li_xCoO_2. The surface area of the $LiNiO_2$ tested was 0.7 m^2/g. When $LiNiO_2$ was charged to a voltage of 4.4 V or higher the sample exhibited a very exothermic DSC peak. Although $LiNiO_2$ gives an increase in capacity compared to $LiCoO_2$, the severe compromise in the thermal stability of the electrode hinders the possibility of using $LiNiO_2$ in commercial applications.

To improve the thermal stability of $LiNiO_2$, commercial suppliers have replaced a small portion of the Ni by Co, to give $LiNi_{0.8}Co_{0.2}O_2$ [17]. This material shows good electrochemical characteristics but the thermal stability, as shown in Fig. 4, is not appealing. The maximum evolved power and onset temperature for $LiNi_{0.8}Co_{0.2}O_2$ are lower and higher, respectively, than $LiNiO_2$, but the reactivity is still enhanced over $LiCoO_2$. The surface area of the tested $LiNi_{0.8}Co_{0.2}O_2$ (0.3 m^2/g) was very close to that of the $LiCoO_2$.

Currently, $Li_{1+x}Mn_{2-x}O_4$ has been commercialized as a cathode for lithium-ion batteries due to its improved thermal stability and potentially lower cost compared to $LiCoO_2$. Fig. 5 shows the DSC profiles of $Li_{1+x}Mn_{2-x}O_4$ at various voltages. Clearly, $LiMn_2O_4$ demonstrates improved thermal stability over $LiCoO_2$, although its surface area is somewhat larger (0.6 m^2/g). The DSC profiles of $Li_{1+x}Mn_{2-x}O_4$ charged to 4.2, 4.4, and 4.6 V are very similar. This is due to the fact that very little lithium remains in the structure at these voltages and thus the electrodes are almost identical. This is clearly shown in the capacity graph in the top panel of Fig. 5. Although $Li_{1+x}Mn_{2-x}O_4$ at 4.2, 4.4, and 4.6 V has an initial thermal instability near 220 °C, it releases little heat.

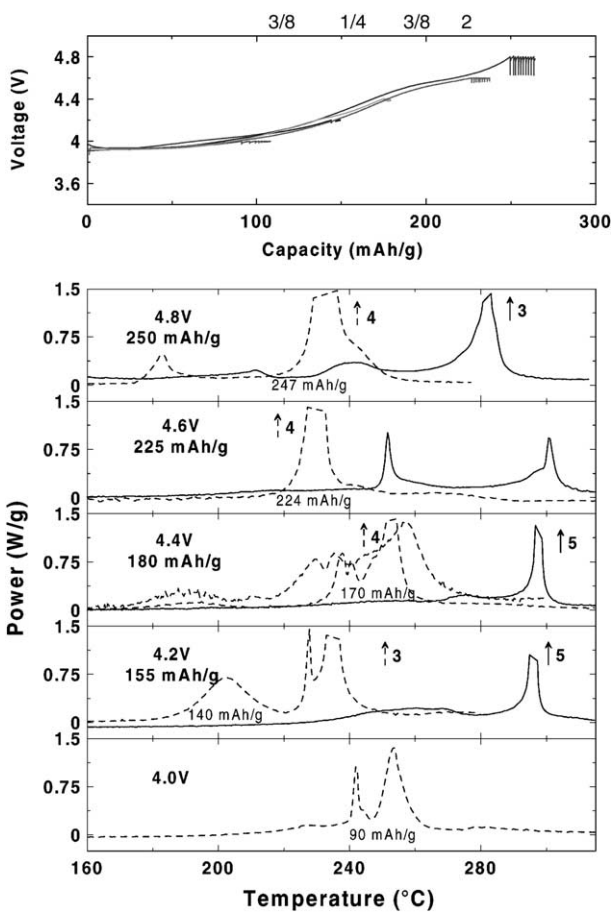

Fig. 7. DSC profiles of $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$ charged to indicated voltages (solid lines, bottom panels). Duplicate scans are from a nominally identical sample, while dashed lines are from the $LiCoO_2$ sample at the indicated voltage. Top panel: charge characteristics of $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$.

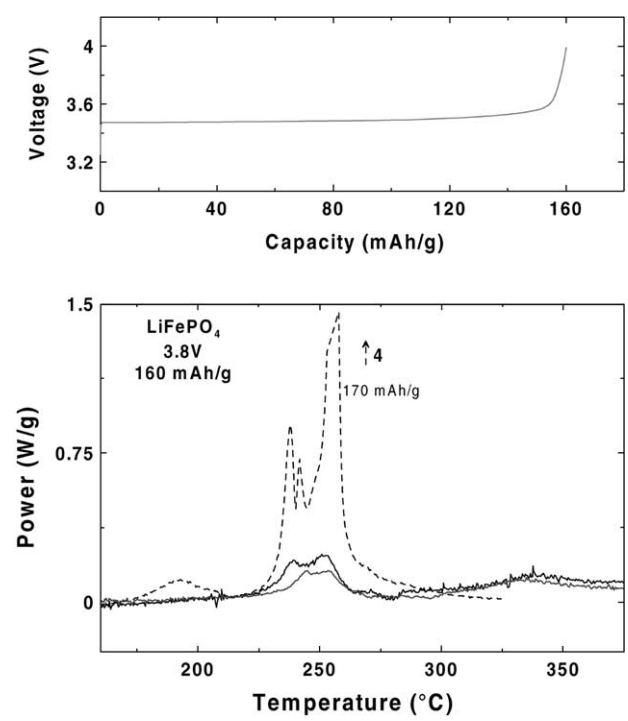

Fig. 8. DSC profiles of $LiFePO_4$ charged to 3.8 V (solid line). Duplicate scans are from a nominally identical sample, while dashed lines are from the $LiCoO_2$ sample at the indicated capacity. Top panel: charge characteristics of $LiFePO_4$.

Its main reactivity is near 280 °C, some 30–40 °C higher than $LiCoO_2$.

Recently, researchers at FMC Corporation have introduced a new cathode material which they describe as offering improved capacity and safety characteristics. The active electrode is similar to $LiNi_{0.8}Co_{0.2}O_2$, but with addition of Ti and Mg in small amounts to give $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$ [18]. Fig. 6 shows the comparison of DSC profiles of this material with $LiCoO_2$ at various voltages. Although the FMC material offers more capacity than $LiCoO_2$, the heat released at the various voltages is larger and the onset temperatures are generally lower. Its surface area is larger (0.8 m^2/g) than the comparison $LiCoO_2$. There is a gain in the capacity of the electrode but no significant increase in the safety characteristics of the electrode was seen.

The materials studied thus far show no clear safety advantage over $LiCoO_2$, except for $Li_{1+x}Mn_{2-x}O_4$ whose low capacity has limited its widespread introduction to the commercial market. Although the fundamental factors that govern the reaction kinetics of a delithiated cathode and electrolyte are not fully understood, we contend that there must be safer electrodes. Recently, in our laboratory, we discovered the layered solid solution series $Li[Ni_xCo_{1-2x}Mn_x]O_2$ that shows excellent capacity and safety characteristics [12,19,20]. The cycling regime and DSC profiles of one member of this series, $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$, is shown in Fig. 7, which shows that the main exothermic reaction for

Table 2
Analysis of DSC profiles from Figs. 2–8

Voltage	Cap. (mAh/g)	T_o[a] (°C)	T_p[b] (°C)	P_p[c] (W/g)	H_{tot}[d] (J/g)	Surface area (m^2/g)
4.0 V						
$LiCoO_2$	247	175	232	3.5	1700	0.1
$LiNiO_2$	250	187	216	31.2	1200	0.7
$LiNi_{0.8}Co_{0.2}O_2$	275	192	212	2.0	1200	0.3
$LiMn_2O_4$	–	–	–	–	–	0.6
FMC	233	173	250	5.8	1600	0.8
$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$	246	230	282	2.8	660	5.9
4.2 V						
$LiCoO_2$	224	180	231	4.0	760	
$LiNiO_2$	237	184	214	23.9	1600	
$LiNi_{0.8}Co_{0.2}O_2$	250	193	213	2.5	1200	
$LiMn_2O_4$	123	207	289	0.7	990	
FMC	223	169	231	3.1	1200	
$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$	219	252 (1)		1.0	400	
		300 (2)		1.0		
4.4 V						
$LiCoO_2$	170	181	256	3.8	1100	
$LiNiO_2$	234	182	209	30.5	1300	
$LiNi_{0.8}Co_{0.2}O_2$	221	197	228	3.1	1600	
$LiMn_2O_4$	123	209	280	0.8	860	
FMC	190	175	220	0.9	1600	
$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$	185	270	297	4.3	290	
$LiFePO_4$	160 (3.8 V)	221	252	0.3	520	15.2
4.6 V						
$LiCoO_2$	140	190	236	3.4	990	
$LiNiO_2$	194	181	209	0.8	1200	
$LiNi_{0.8}Co_{0.2}O_2$	198	194	234	5.7	1300	
$LiMn_2O_4$	110	216	281	0.8	890	
FMC	180	180	231	0.9	1200	
$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$	158	245	295	5.2	310	
4.8 V						
$LiCoO_2$	90	219	254	1.8	760	
$LiNiO_2$	145	174	247	0.8	1300	
$LiNi_{0.8}Co_{0.2}O_2$	165	193	267	2.5	1400	
$LiMn_2O_4$	20	254	267	0.8	310	
FMC	140	236	255	1.6	720	
$Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$						

[a] Onset temperature.
[b] Peak temperature.
[c] Peak power.
[d] Total evolved heat.

this new material is above 280 °C, some 40–60 °C higher than $LiCoO_2$, at potentials of 4.4 V and lower. At 4.6 V, the new material has two well-resolved DSC peaks, whose total power is about the same as Li_xCoO_2. However, the splitting of the power into two peaks may help mitigate serious consequences in safety tests. The electrode at 4.8 V has a similar reactivity as $LiCoO_2$. Clearly, there can be progress towards understanding the safety characteristics of electrode/electrolyte pairs by synthesizing and studying new materials in a systematic manner. As an example, notice that $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$ is more thermally stable than $LiCoO_2$ even though its specific surface area is much larger (5.9 m^2/g).

Recently, $LiFePO_4$ has been suggested as an electrode material for lithium-ion batteries [13,14,21]. Although its theoretical capacity is rather low (169 mAh/g), preparative methods have been developed such that nearly all the lithium (160 mAh/g) in the structure can be extracted and re-inserted without loss. Fig. 8 shows a comparison of DSC experiments made on Li_xFePO_4 charged to 3.8 V (no lithium remaining in structure) to experiments on Li_xCoO_2 with the same specific capacity of lithium removed (4.4 V). Li_xFePO_4 (3.8 V) shows a very small exotherm as compared to Li_xCoO_2, although the main exothermic activity occurs at the same temperature. This is even more impressive when one recalls that the specific surface area of the $LiFePO_4$ sample is 15.2 m^2/g while that of the $LiCoO_2$ sample is 0.1 m^2/g. In view of these results, $LiFePO_4$ may be well suited to large size lithium-ion cells where safety and cost are extremely important.

Table 2 presents a summary of the DSC results for the electrodes studied here. The table contains a column for each of specific capacity (mAh/g), onset temperature (°C), peak temperature (°C), peak power (W/g), and total evolved heat (J/g). Tables such as these are required in order to choose the optimum electrode for a specific application.

4. Conclusions

DSC was used to compare the thermal stability of seven different charged cathodes in 1 M $LiPF_6$ EC/DEC electrolyte. In our opinion, the cathode materials can be ranked from most safe to least safe in the following order $LiFePO_4$, $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$, $Li_{1+x}Mn_{2-x}O_4$, $LiCoO_2$, $LiNi_{0.7}Co_{0.2}Ti_{0.05}Mg_{0.05}O_2$, $LiNi_{0.8}Co_{0.2}O_2$, $LiNiO_2$. The safest materials from this group had the largest specific surface areas, which we believe is coincidental, and reflects their inherent stability. This suggests that if the synthesis of $LiFePO_4$ and $Li[Ni_{3/8}Co_{1/4}Mn_{3/8}]O_2$ can be optimized to give specific surface areas in the range of the other materials (i.e. <1 m^2/g), then extremely inert materials can be produced. $LiFePO_4$ appears to be the best candidate for large size Li-ion cells based on cost and safety.

References

[1] J.R. Dahn, E.W. Fuller, M. Obrovac, U. von Sacken, Solid State Ionics 69 (1994) 265.
[2] P. Biensan, B. Simon, J.P. Peres, A. deGuibert, M. Broussely, J.M. Bodet, F. Perton, J. Power Sources 81/82 (1999) 906.
[3] H. Maleki, G. Deng, A. Anani, J. Howard, J. Electrochem. Soc. 146 (1999) 3224.
[4] Underwriters Laboratory Inc., Standard for Lithium Batteries, document 1642, 3rd Edition, 1995, ISBN 1-55989-829-1.
[5] International Air Transport Association, Transport of Dangerous Goods—Lithium Batteries, UN document ST/SG/AC.10/11, 1998.
[6] S.-I. Tobishima, K. Takei, Y. Sakurai, J.-I. Yamaki, J. Power Sources 90 (2000) 188.
[7] A. Du Pasquier, F. Disma, T. Bowmer, A.S. Gozdz, G. Amatucci, J.-M. Tarascon, J. Electrochem. Soc. 145 (1998) 472.
[8] Z. Zhang, D. Fouchard, J.R. Rea, J. Power Sources 70 (1998) 16.
[9] D.D. MacNeil, L. Christensen, J. Landucci, J.M. Paulsen, J.R. Dahn, J. Electrochem. Soc. 147 (2000) 970.
[10] M.N. Richard, J.R. Dahn, J. Electrochem. Soc. 146 (1999) 2078.
[11] T.D. Hatchard, D.D. MacNeil, A. Basu, J.R. Dahn, J. Electrochem. Soc. 148 (2001) A755.
[12] Z. Lu, D.D. MacNeil, J.R. Dahn, Elect. Solid-State Letts. 4 (2001) A200.
[13] A. Yamada, S.C. Chung, K. Hinokuma, J. Electrochem. Soc. 148 (2001) A224.
[14] N. Ravet, Y. Chouinard, J.F. Magnan, S. Besner, M. Gauthier, M. Armand, J. Power Sources 97/98 (2001) 503.
[15] International Center for Diffraction Data, Newtown Square, PA, 19703 USA, JCPDS file #40-1499.
[16] D.D. MacNeil, H. Fortier, J.R. Dahn, Thermochimica Acta, in press.
[17] C. Delmas, I. Saadoune, A. Rougier, J. Power Sources 43/44 (1993) 595.
[18] Y. Gao, M.V. Yakovleva, ECS Meeting Abstracts, MA 98-2 (1998) 118.;
Y. Gao, M.V. Yakovleva, W.B. Ebner, Elect. Solid-State Letts. 1 (1998) 117.
[19] Z. Lu, D.D. MacNeil, J.R. Dahn, Elect. Solid-State Letts. 4 (2001) A191.
[20] Z. Lu, J.R. Dahn, J. Electrochem. Soc., submitted for publication.
[21] A.K. Padhi, K.S. Nanjundaswamy, J.B. Goodenough, J. Electrochem. Soc. 144 (1997) 1188.