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DEVELOPMENT OF A LIGHTWEIGHT NICKEL ELECTRODE

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SUMMARY

Nickel electrodes made using lightweight plastic plaque are about half the weight of electrodes made from state-of-the-art sintered nickel plaque. This weight reduction would result in a significant improvement in the energy density of batteries using nickel electrodes (nickel hydrogen, nickel cadmium and nickel zinc). These lightweight electrodes are suitably conductive and yield comparable capacities (as high as 0.25 AH/gm (0.048 AH/cm²)) after formation. These lightweight electrodes also show excellent discharge performance at high rates.

INTRODUCTION

The sealed sintered plate type nickel cadmium cell has been substantially improved over the past several years due to the transition from vacuum to the electrochemical methods of impregnation of active materials into a sintered nickel plaque. A significant increase in the capacity per unit weight of the nickel electrode from approximately 0.060 AH/g (0.025 AH/cm²) to about 0.086 AH/g (0.038 AH/cm²), was achieved by adopting this change (refs. 1 and 2). A further increase in capacity per unit weight can be accomplished by using other lightweight cell components.

This paper discusses a study to produce a lightweight nickel electrode for NiCd and NiH₂ batteries for use in space applications and for NiZn batteries for use in terrestrial applications by replacing the heavy sintered nickel plaque with lightweight plated plastic. Previous work in this area has been published (refs. 3 and 4). Different commercial and experimental materials were investigated as substrates. The material selected for study was a porous polyvinyl chloride, Amerace A-40 (Amerace Corp., Butler, NJ), with an expanded nickel grid (Delker Corp., Branford, CT) embedded in it to serve as the current collector. This porous plastic is very lightweight and has a porosity, pore size, and surface area comparable to a typical commercial nickel plaque.

The porous plaque for the standard sintered nickel plate is fabricated by depositing nickel powder on a nickel grid current collector and heating to a high temperature (1000° C) in a reducing atmosphere to sinter the nickel particles together. The nickel coating on the lightweight plastic substrate was deposited by an electroless plating process. Resembling electroplating, this controlled autocatalytic reduction of nickel onto a catalytic surface results in the production of a uniform, adherent coating of the metal. The electroless plating has some distinct advantages over the electrodeposition process: coatings can be deposited onto plastic, which cannot be done with electrodeposition. The coatings have a uniform thickness; no buildup of coating on points or edges; and no need for electrodeposition equipment. The plastic plaques made by the electroless plating process can be made to be as conductive and have greater capacity for active material than the heavier commercial sintered plaques (0.25 AH/g (0.048 AH/cm²) for the light-weight plastic plaque described here versus 0.086 AH/g (0.038 AH/cm²) for the commercial sintered plaque).

EXPERIMENTAL PROCEDURE

The general procedure for making a lightweight nickel electrode is shown in the following flow diagram:

Grid

Coating of grid with a mixture of plastic and silica particles

Extracting of silica

Substrate

Electroless plating of substrate

Plaque

Impregnating of plaque

Plate

Forming of plate

Washing, drying

Ni(OH)₂ Electrode ready for evaluation

Plaque Preparation

<u>Coating process</u>.- A 10-mil expanded nickel grid was coated with Amerace A-40 porous polyvinyl chloride by Amerace Corp. This contains silica and has a porosity of about 65 percent which can be increased by extraction of the silica.

<u>Extraction process</u>. - This procedure is used to remove the silica contained in the porous coated grid (called substrate) thus increasing its porosity to 85 percent or more. This step is necessary because the potassium hydroxide would dissolve the silica in the cell, thus breaking up the plated nickel layer if the silica is not removed before the plating process. The extracted substrate was cut to size, weighed, measured for thickness and soaked overnight at 80° C in 45 percent potassium hydroxide solution. The substrate was then rinsed thoroughly until free of potassium hydroxide and dried at 80° C for at least 4 hr. The substrate was then reweighed and remeasured for thickness.

<u>Pretreatment process</u>. - The plating apparatus as shown in figure 1 consists of two PVC pipes (o.d. = 8.8 cm, i.d. = 7.8 cm) with 2 cm thick PVC flanges at one end of each pipe. A 7.5 cm diameter, 1/2 cm thick porous carbon disc (Union Carbide Corp.) which was set into the top of the bottom pipe was used to support the substrate. This porous carbon was used since it was not itself plated by the plating solution and thus did not get clogged in the plating process. The two pipes were bolted together and the solution poured into the top pipe allowing it to flow through the substrate. It was necessary to have the solution flow through the plastic (at the rate of about 2 cc/min/cm²) in order to provide uniform plating in the interior of the plaque.

The substrate was cleaned by rinsing it in a hot solution of 5 percent KOH and a Triton X-100 wetting agent for 4 min in the flow-through plating apparatus.

The nonconductive plastic substrate must be catalyzed in order to initiate nickel deposition. The pretreatment stage which is a method of applying a catalytic metal to the plastic substrate consists of two steps:

1. Sensitizing - This process consists of adsorbing onto the plastic substrate a reducing agent. The oxidation of the sensitizer serves to deposit a catalytic film on the surface of the plastic substrate. The sensitizer used was an acid solution of stannous chloride. This solution is used at 32° C, with a flow-through time of 3 min. The substrate must then be thoroughly rinsed after sensitizing to remove excess sensitizer before the next step.

2. Activation - In this process the plastic substrate is made catalytic so that the plating is confined just to the substrate. The activating solution is applied by exposing the sensitized substrate to an acid solution containing palladium chloride (PdCl₂). The PdCl₂ salt is reduced to metallic Pd nuclei by the stannous chloride adsorbed in the previous step. These catalytic nuclei act to localize plating at the desired places. This solution is used at 52° C with a 3 min flow-through time. Thoroughly rinsing the substrate must follow this step.

<u>Electroless nickel plating</u>. - The electroless plating process is a method of chemical plating which provides a continuous build-up of metal coating on the substrate by a simple immersion in the plating bath. A reducing agent supplies the electrons in the solution for converting metal ions to the elemental form:

 $M^{2+} + 2e^{-} \frac{Catalytic}{Surface} M$

The reducing agents explored for the work reported here included hypophosphite and borohydride. The borohydride bath was chosen over the hypophosphite bath because the coatings from the former have much lower impurities and higher conductivity than from the latter bath.

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The electroless nickel plating process using sodium borohydride as reducing agent was designed to deposit a high purity nickel alloy containing a minimum of 99.5 percent nickel and a maximum of 0.5 percent boron onto the conducting surfaces. Borohydride is subject to rapid decomposition by hydrolysis in all but strongly alkaline solutions, thus the pH of the bath are usually maintained at 9 or higher. In order to prevent rapid decrease in the bath pH, and thus in deposition rate, it is advisable to include a buffering agent to the bath. To prevent precipitation of the nickel hydroxide from the bath, a complexing agent should also be present. A stabilizer is also required to reduce the rapid decomposition of the bath.

Plaque Characterization

The plated substrate was subjected to several characterization tests.

<u>Thickness and porosity</u>. - The thickness was determined at 5 locations on the plaque using a thickness gauge. The plaque porosity was calculated from these values taking into consideration the weight and volume of the substrate (grid and plastic coating).

$$P = \frac{v}{V_T} \times 100$$

$$V_{v} = V_{T} - (V_{N1} + V_{PVC})$$

 V_{T} = (thickness x area) plaque

$$V_{Ni} = (W_g + W_{Ni}) \frac{1}{\rho_{Ni}}$$
$$V_{PVC} = (W_{PVC}) \frac{1}{\rho_{PVC}}$$
$$V_v = V_T - \frac{1}{\rho_{Ni}} (W_g + W_{Ni}) - \frac{1}{\rho_{PVC}} (W_{PVC})$$
$$V_T = V_v + \frac{1}{\rho_{Ni}} (W_g + W_{Ni}) + \frac{1}{\rho_{PVC}} (W_{PVC})$$

where

P porosity in percent

 V_T total volume of plaque in cc/cm²

 V_v void volume in cc/cm²

 V_{N1} volume of Ni plate in cc/cm²

V_{PVC} volume of PVC plastic in cc/cm²

 W_{a} weight of grid in g/cm²

 W_{N1} weight of Ni plate in g/cm²

 W_{PVC} weight of PVC plastic in g/cm²

PNi density of Ni, 8.9 g/cc

PPVC density of PVC, 1.39 g/cc

<u>Electrical resistance</u>. - Resistivity was measured by the probe shown in figure 2(b). The plaque of known thickness was set in between the two copper rods and the probe was connected to a power supply as shown in the schematic in figure 2(a). The potential difference through a given thickness of the plaque was then measured. Knowing the current and the cross-sectional area of the copper rod, the resistivity, in ohm-cm, was calculated.

<u>Pore size distribution</u>. - The pore size distribution measurements were made by mercury intrusion porosimetry (Micromeritics, Norcross, Georgia).

<u>Scanning electron micrographs (SEM)</u>. - Scanning electron micrographs were taken on a JSM-2 (JEOL, Japan).

Electrochemical Impregnation

The active material for positive plates is nickel hydroxide. Several methods can be used to incorporate nickel hydroxides into the nickel plaque, including the electrochemical impregnation method used in this study. The Ni(OH)₂ active material was electrochemically deposited in the pores of the plaque by passing a cathodic current while the plaque is immersed in nitrate solution.

Table I lists the conditions used for the electrochemical impregnation. The pH and temperature of the solution are varied depending on the desired weight increase of the plaque. The alcohol used was denatured alcohol Type IV, containing methyl alcohol (Continental Chemical Corp., Terre Haute, Indiana 47808). The electrical circuit schematic diagram for impregnation is shown in figure 3 and the cathodic impregnation apparatus is shown in figure 4. The Ni plaque was mounted vertically in a glass beaker centrally located between two standard sintered Ni counterelectrodes. The preheated electrolyte was poured into the beaker, electrical connections were made wherein the plaque was connected to the negative terminal of the power supply and the counterelectrodes connected to the positive terminal. The power supply was then adjusted to the desired constant current density. An impregnation run typically requires about 300 min to complete. The bath pH was adjusted to approximately 2.0 prior to the start of the run and was monitored during the course of the process not allowing it to exceed 4.0. This was done by the addition of dilute nitric acid.

Following impregnation, the plaques were rinsed with water and dried under vacuum for at least 6 hr at room temperature. The plaques are not dried by

heating to avoid distortion of the underlying porous plastic. The plaques were then weighed and measured for thickness.

Formation

After impregnation, the plaques were formed by cycling several times in 31 percent solution of potassium hydroxide electrolyte. The first objective of formation is the removal of impurities (chiefly nitrates) carried over from the impregnation bath. The second objective is to give the active material (nickel hydroxide) electrochemical "exercise" by a repeated oxidation-reduction process which brings it to full capacity.

The impregnated plaque was mounted between two standard sintered nickel counterelectrodes. A series of 30 charge and discharge cycles was applied to the electrodes using a 31 percent solution of potassium hydroxide. An amalgamated zinc electrode was used as reference electrode. This formation method was based on Pickett's process (ref. 2).

RESULTS AND DISCUSSIONS

Extraction

Table II shows extraction data for the plastic substrate after soaking in a potassium hydroxide solution overnight at 80° C. The extracted plastic lost an average of 40 percent of its weight and decreased 4 to 13 percent in thickness. Further extraction using the same conditions as the initial extraction gave an additional loss of 2 to 5 percent of its weight while the thickness remained the same. Overnight soaking at 80° C seemed to be sufficient for a complete extraction of the silica since no further loss of weight was measured.

Electroless Plating

Plaques of porosities ranging from 69 to 90 percent were prepared. The resultant data are given in table III. An average weight gain of 0.04 g/cm² was obtained without a sizeable increase in thickness for the thinner plaques while the thicker plaques experienced more surface plating with an average weight gain of 0.09 g/cm². Temperatures below and above the recommended temperature (table I) were used to plate some substrates. Thicker substrates plated at a lower temperature for a short period of time experienced a large increase in thickness. An increase in plating weight reduces porosity of the final plaque as expected. A plaque with porosity of about 80 percent is comparable to state-of-the-art nickel electrodes. There is a good deal of scatter in the data. The chief reason for the scatter is believed to be variations in porosity and thickness of the experimental plastic. The incorporation of the grid into the commercial plastic was done on an experimental basis and has not been optimized.

A plating time and temperature of 30 min and 60° C are sufficient to yield more uniform and conductive plaques.

Plaque Characterization

The result of some electrical resistivity measurements made in the laboratory are given in table III. The electrical resistivity of the lightweight plaques are about 2 to 4 times lower than those measured on the Eagle-Picher heavy sintered plaques and about 2 to 4 times higher than those measured on the Tyco plaque. As anticipated, the resistivity of the more porous plaques were higher than the less porous plaques.

Figure 5 shows the pore size distribution curves of several plaques. Plaque E500 shows a major peak at about 7 μ while plaque C119 displays a sharp peak at about 11 μ . Plaques A404801 and A404805 show distributions which are similar. A greater distribution of large pores is found in the pore size distribution of plaque A404809 with peaks from 2 μ to 61 μ .

Figure 6 shows the SEM photomicrograph of plaque E500 which consists of relatively irregular pores.

Electrochemical Impregnation

The electrochemical impregnation data are presented in table IV. Electrodes 1-1 to 1-8 are data on segments taken from the same plaque, A404805, impregnated at different conditions. Electrodes 2-1 to 2-6 are data from plaque A404809, while Electrode 3-1 is the impregnated Cl19. It can be seen that loading levels of greater than 1.6 g Ni(OH)₂/cc void were readily attainable. Although there is considerable scatter a lower current density is favored over a higher current density in attaining satisfactory electrodes without a large increase in thickness.

Thickness increase showed a dependance on current density and impregnation time, varying from a minimum of 1.1 mil for an electrode loaded at 54 mA/cm² (0.35 A/in²) for 120 min to 22.8 mils for an electrode loaded at 78 mA/cm² (0.50 A/in²) for 360 min.

In this study, the impregnation condition which appeared most promising in obtaining a good lightweight porous electrode is to impregnate at a current of 54 mA/cm^2 (0.35 A/in²) with a pH of less than 4 at 80° C. Impregnation for 120 min gave a loading of 1.7 gm/cc void.

Formation and Performance

Formation data of Electrode 2-2 and Electrode 2-6 in 31 percent KOH solution are found in table V. The theoretical capacities of the electrodes based on the reaction

 $Ni^{2+} + 20H^{-} \rightarrow Ni(0H)_{2}$

with a theoretical conversion factor of 0.289 AH/gram of Ni(OH)₂ based on a one electron transfer were 0.038 AH/cm² for Electrode 2-2 and 0.044 AH/cm² for Electrode 2-6. The Pickett formation procedure (ref. 2) was followed where 30 cycles were carried out at the rate of c/2 of the theoretical capacity. The electrodes were charged according to the capacity of the previous cycle with

10 percent overcharge. The cut-off point for the discharge was 0.9 V versus an amalgamated Zn electrode. After the 20th cycle, the formation assembly was disassembled and the Ni plate was scrubbed to remove surface deposits, rinsed thoroughly, vacuum dried, weighed and remeasured for thickness. The plate thickness of Electrode 2-2 was reduced from 44.1 to 41.2 mils and from 49.6 to 45.0 mils for Electrode 2-6.

The formation assembly was reassembled and the electrodes formed for another 10 cycles. The utilization reached 114 percent and the final delivered capacity of 0.14 AH/g (0.043 AH/cm²) for Electrode 2-2. Electrode 2-6 reached a utilization of 110 percent and a final capacity of 0.25 AH/g (0.048 AH/cm²). Both electrodes showed no sign of blistering or electrode expansion. Figure 7 are representative SEM photomicrographs of the formed Electrode 2-2. Note that the surface of the electrode is completely covered with Ni(OH)₂ crystals.

The discharge curve for Electrode 2-6 as cycled against a cadmium electrode is shown in figure 8. A General Electric sintered NiCd cell (ref. 5) is also shown for comparison. Both cells were discharged at a c rate and both curves show the discharge voltage dropping to 1 volt at 100 percent capacity. The lightweight beaker (flooded) cell has a much higher discharge voltage than that of the G.E. sintered NiCd cell. The lightweight cell had a constant discharge voltage from 20 to almost 98 percent of its discharged capacity after which the voltage fell very rapidly. A battery voltage of 1 volt defines the end of discharge.

The weight of components in a typical state-of-the-art 20 AH nickel cadmium (NiCd) cell is shown in the left-hand side of figure 9. A major weight reduction of from 0.88 kg with an energy density of 33 WH/kg to 0.53 kg with an energy density of 55 WH/kg can be accomplished by the improvement of material utilization, the reduction of the case weight and the reduction of the plaque weight.

CONCLUSION

A commercial porous polyvinyl chloride plastic has been found to be a satisfactory substrate for making lightweight nickel electrodes. Pore size, resistivity, plaque loading, and performance of electrodes made from this plated plastic are comparable to standard sintered electrodes. The electroless plating process using sodium borohydride as a reducing agent yields a uniform, defect-free, strong and easily loaded plaque.

The electrochemical impregnation of plated nickel plaque from a high temperature, acidic bath of nickel nitrate-cobalt nitrate dissolved in a mixture of alcohol and water yields loading levels comparable to commercial Ni electrodes. These high loadings are effectively utilized in the flooded formation cells.

When impregnation is carried out at a low rate of 0.054 A/cm^2 (0.35 A/in²) plate swelling is minimal.

The lightweight electrodes have been cycled up to 40 cycles with excellent performance retention.

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TABLE I. - CONDITIONS FOR ELECTROCHEMICAL IMPREGNATION OF NICKEL

$Ni(NO_3)_2$, molar
Ethyl alcohol, percent by volume
pH \ldots
Current density, mA/cm^2 (A/in ²)
Current duration, min \ldots

Substrate	Unextract	ed substrate	Weight	Decrease in thickness		
	Weight, gm/cm ²	Thickness, mils	Weight, gm/cm ²	Thickness, mils	percent	percent
C119	0.067	60.0	0.039	48.5	42	19
A40135	.064	49.4	.037	49.4	42	0
A404801	.058	36.5	.036	35.0	38	4
A404805	.059 -	38.4	.036	36.9	39	4
A404809	.058	38.8	.036	36.9	38	5
A40117	.065	23.5	.033	20.5	49	13
A4017A	.067	47.0	.040	45.3	40.	4

TABLE II. - EXAMPLES OF EXTRACTION DATA

TABLE III. - PREPARATION OF PLASTIC PLAQUES

Plaque	Plating conditions		Plaque porosity.	Weight increase.	Thicknes	s, mils	Measured volume	
	Time, min	Temperature, C	percent	gm/cm ²	Final	Change	resistivity, ohm-cm	
A40127 A4017 A40147 C119 E201 E300 A404801 A40121 A404805 A404809 A404807	15 15 15 15 22 30 30 30 30 30 40 40	60 43 43 43 60 60 60 60 60 60 80 60	81 72 90 90 70 69 72 82 73 75 85	0.057 .073 .037 .028 .120 .190 .035 .055 .050 .056 .048	26.3 49.3 51.8 58.0 53.6 61.5 37.8 26.8 41.9 41.6 37.3	1.2 4.0 9.1 9.5 8.6 9.2 2.8 3.9 5.0 	$ \begin{array}{c} \hline \\ \hline \\$	
E500 A40108 A4048011 Eagle Picher ^a Tyco ^b	43 50 60 	60 60 60 	85 87 88 ^c 80.5 ^d 62.5	.053 .021 .025 	32.9 27.0 30.7 	9.4 3.2 2.9 	4.4×10 ⁻⁴ 	

^aEagle-Picher plaque, Joplin, Missouri.
^bTyco plaque, Waltham, Mass.
^c, ^dMeasured by Mercury Porosimeter.
Average plaque area is 60 cm² except A4017, A40147, C119, E201, and E300 with an average plaque area of 8 cm².

Electrode	Opera	Operating conditions				Thickness, mils		Weight increase.	Loading level.	Theoretical capacity.
	Current density, mA/cm ²	Temper- ature, °C	Time, min	рН	Final	Change	percent	gm/cm ²	gm/cc void	AH/cm ²
1-1	54	85	180	2	47.6	3.5	43	0.083	1.59	0.024
1-2	54	85	120	4	44.2	3.4	43	.082	1.69	.024
1-3	54	90	180	4	49.7	5.6	43	.090	1.66	.026
1-4	54	90	210	4	39.5	1.1	40	.072	1.79	.021
1-5	78	80	150	2	51.5	11.5	43	.096	1.71	.028
1-6	78	80	120	4	52.2	13.0	43	.097	1.70	.028
1-7	78	87	120	4	48.7	7.6	42	.106	2.04	.031
1-8	109	85	300	2	44.6	4.8	42	.104	2.18	.030
2-1	67	85	360	4	44.8	.3	43	.095	1.94	.027
2-2	70	80	420	2	44.1	3.1	41	.130	2.82	.038
2-3	78	85	360	4	63.6	22.8	50	.230	2.84	.066
2-4	109	80	240	2	35.3	7.3	44	.074	1.87	.021
2–5	109	85	300	3	50.3	8.7	43	.112	2.03	.032
2-6	78	85	300	4	49.6	13.5	51	.153	2.30	.044
3-1	78	80	88	4	50.9	5.3	51	.148	2.24	.043

TABLE IV. - ELECTROCHEMICAL IMPREGNATION DATA

Average electrode area is 2.0 cm². Electrode 1-1 to 1-8, plaque 404805. Electrode 2-1 to 2-6, plaque 404809. Electrode 3-1, plaque C119.

Cycle	Charge time, min		Discharge time, min		Capacity	, AH/cm ²	Utilization ^a ,			
	G(2-2)	G(2-6)	G(2-2)	G(2-6)	G(2-2)	G(2-2) $G(2-6)$				
					-()		G(2-2)	G(2-6)		
1	147	133	32	34	0.029	0.033	76	75		
5	60	55	43	36	.032	.038	86	86		
10	58	55	46	39	.034	.041	90	93		
15	58	57	55	40	.036	.043	95	98		
20	73	60	48	44	.039	.046	104	105		
25	76	64	54	44	.040	.047	106	107		
30	93	67	42	47	.043	.048	114	110		

TABLE V. – NI	CKEL OXIDE	FORMATION	DATA FOR	ELECTRODES	2-2	AND	2-6
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^aPercent utilization = $\frac{Ampere-hour out}{Theoretical capacity} \times 100$

Loading levels: $(2-2) - 1.16 \text{ g/cc void } (0.13 \text{ g/cm}^2)$ $(2-6) - 1.57 \text{ g/cc void } (0.153 \text{ g/cm}^2)$

TABLE VI. - COMPARISON OF PLATED PLASTIC ELECTRODES WITH A TYPICAL SINTERED ELECTRODE (ref. 6)

	Typical sintered electrode	Electrode 2-6
Plaque thickness, mils (cm) Plaque porosity, percent Plaque weight, g/cm ² g/cc Electrode thickness, mils (cm) Electrode porosity, percent Loading, g/cm ² g/cc g/cc void Electrode weight, g/cm ² g/cc Theoretical capacity, AH/g of electrode weight Theoretical electrode weight for 20 AH cell, g	$\begin{array}{c} 29.5 & (0.075) \\ & 80.0 \\ & 0.106 \\ & 1.69 \\ 30 & (0.076) \\ & 46 \\ & 0.110 \\ & 1.45 \\ & 1.6 \\ & 0.30 \\ & 3.82 \\ & 0.118 \\ & 169 \end{array}$	$\begin{array}{c} 41.6 & (0.106) \\ & 83 \\ & 0.033 \\ & 0.32 \\ 49.6 & (0.126) \\ & 51 \\ & 0.153 \\ & 1.21 \\ & 2.30 \\ & 0.25 \\ & 1.89 \\ & 0.228 \\ & 88 \end{array}$











Figure 3. - Electrical circuit schematic for electrochemical impregnation.











Figure 6. - SEM photomicrograph of plaque E500, 300X.



(a) 250X.



(b) 1200X.

Figure 7. - SEM photomicrographs of for med electrode made from plaque A404809.



(C-rate).





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reduction would result i	n a significant i	morovement in th	e energy dens	ity of bat-			
teries using nickel elec	trodes (nickel hv	drogen, nickel c	admium and ni	ckel zinc).			
These lightweight electr	odes are suitably	conductive an y	ield comparab	le capaci-			
ties (as high as 0.25 AH	/gm (0.048 AH/cm ²)) after formati	on. These li	ghtweight			
electrodes also show exc	ellent discharge	performance at h	igh rates.				
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