COMSAT'S DESTRUCTIVE PHYSICAL ANALYSIS OF AEROSPACE NICKEL-CADMIUM CELLS FOR NASA/GODDARD SPACE FLIGHT CENTER

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Over the past 5 years, COMSAT has performed numerous destructive physical analyses (DPAs) on NASA-Goddard-supplied nickel-cadmium (Ni/Cd) cells. The samples included activated but uncycled cells, wet stored cells, cycled cells, and anomalous cells. The DPAs provided visual, morphological, and chemical analyses of the cell components. The DPA data for the analyzed cells are presented herein. For the cells investigated, the leading cause of poor performance, as determined by DPA, has been poor negative electrode utilization, which resulted in negative-electrodelimiting operation.

INTRODUCTION

Traditionally, NASA/Goddard has requested destructive physical analyses (DPAs) on Ni/Cd cells with anomalous performance. This technique has been used to understand poor performance and failure mechanisms in the cells. COMSAT recommends DPAs of cell components and cells at the beginning of life to establish a database, which can then be used in determining causes of cell anomalies and for predicting cell life.

Over the past 5 years, COMSAT has performed approximately 20 DPAs (Table 1) on NASA-Goddard-supplied NASA Standard Ni/Cd cells. The majority of these cells have been of the NASA Standard 50-Ah design built since the mid 1980s. These samples have included wet stored cells, activated cycled and uncycled cells, and anomalous cells. The characteristics of anomalous cells have included accelerated separator degradation, cell shorting, and loss of overcharge protection. Although various reasons exist for poor cell performance, one characteristic that has become evident from DPA data is a negative-electrode-limited condition. where cell capacity is limited by the negative electrode on discharge. DPA provided evidence of this condition, which is caused by poor utilization in the negative plate.

RESULTS AND DISCUSSION

Electrical Cycling Performance

When cells are received for DPA, various electrical tests are performed to evaluate and characterize the cell. One area of poor performance in some of the NASA cells has been a continual drop in capacity with successive measurements (Table 2). This behavior indicates a negative-limited cell. The effect is also observed in the charge profile, where voltage rollover occurs at an earlier time with each successive cycle for a negative-limited cell (Figures 1 - 4). Voltage rollover is associated with the

point where the cell goes into Earlier voltage rollover overcharge. indicates that charge input, and therefore capacity, is reduced in successive cycles. The negative-limited condition of the cell on discharge inhibits the positive electrode from completely discharged. being Consequently, the positive electrode, which is already in a partially charged state, will reach overcharge at an earlier point during the next charge period. In a positive-limited cell, the capacity of the rollover point remains fairly constant for a given charge rate.

At a C/10 charge rate at 10°C, rollover occurs where charge input approximately equals cell capacity. One positive-limited cell (UARS Lot 2 S/N 7), exhibited voltage rollover occurring much later than the point where charge input equaled cell capacity (Figure 5). This late rollover is atypical for a positive-limited cell.

The second evidence for a negative-limited cell can be found in the resistive discharge profile generated after a power discharge (Figure 6). The resistive discharge profile for a positive-limited cell exhibits a gradual drop in voltage to a plateau around 0.6 V. A sudden drop in voltage and a voltage plateau around 0.2 V indicate a negative-limited cell.

The third evidence from electrical testing for the negative-limited condition can be found in the voltage recovery stand, where the cell is discharged, shorted, and then open-circuited while the voltage is monitored (Figure 7). Negative-limited cells exhibit higher voltages throughout the 24-hr open circuit period. This higher voltage is likely a result of the higher state of charge of the positive electrode due to the negative-limited condition. Negative limited cells also exhibit fast voltage rise during the first hour of the voltage recovery stand. Positive-limited cells typically show more gradual initial voltage rise.

DPA Work

On completion of the electrical characterization, the cell is opened and visually examined. Comments are made on the physical condition of the cell components, electrolyte distribution, and overall cleanliness.

Chemical, electrical, and microscopic analyses are then performed on the cell components. The electrolyte is analyzed for potassium hydroxide (KOH) and potassium carbonate (K_2CO_3) concentrations. The separator is analyzed for cadmium content and tested for tensile strength. Positive and negative plates are chemically analyzed and electrically cycled in a flooded condition. Microscopic analysis is conducted on sample plates. Precharge and overcharge protection are then calculated for the cell.

The following text presents the results from DPA which confirm the negative limited condition. The source of this condition was determined to be poor performance of the negative electrode. The test results for the cell components (i. e., electrolyte, separator, and positive electrode) suggest that variations in results within these components have been due to natural degradation processes or are the result of the negative-limited condition of the cell.

Electrolyte

The K_2CO_3 and KOH concentrations were determined for the electrolyte (Table 3). As expected, carbonate concentrations increased with increased cycling due to separator degradation. In response to these changes, KOH concentrations also change. However. differences in hydroxide concentration could not be explained by the formation of carbonate alone. Some cells were found to contain excess water in the electrolyte. This excess was evidenced in lower KOH concentrations and increased electrolyte volume relative to quantities added during cell activation. As water is

consumed at the positive electrode during discharge, excess water in the electrolyte can be explained by the fact that the positive electrode is not fully discharged. This condition is consistent with cells that are negative-limited in discharge.

The calculated electrolyte quantity per Ampere-hour of theoretical positive capacity was obtained by converting the total potassium weight to 30-weight percent KOH and dividing this by the theoretical cell capacity, which is based on positive plate active material loading. This value has typically been around 2 cm^3/Ah for the NASA 50-Ah Standard Cells. Differences in these values have been caused by variations in positive plate loading. The exceptions within the data reported here-in have been the IUE cells that were manufactured with more electrolyte.

Separators

The separators were characterized for their cadmium content (Table 4). Pellon 2505 was used in the cells analyzed. Cadmium migration into the separators was measured both by the amount per cell and the amount in the heaviest migrated area. As expected, increased cycling leads to increased migration. However, negative-limited cells have shown lower-than-expected cadmium migration levels, due to inactive cadmium in the negative electrode.

Positive Electrode

The positive plates were chemically and electrochemically analyzed. Positive electrode weight differences between cells have been due to loading differences between cell lots. Active nickel loading has typically been greater than 1.9 g/cm³ of void volume Cobalt levels have been (Table 5). consistent among lots and account for approximately 5 weight percent of the total active material. The total cadmium in the plate comes from two sources: the cadmium added during manufacturing as an "antipolar mass," and that which has migrated from the negative to the

positive electrode. Analysis of the cadmium in the positive plates has shown not only decreased cadmium migration in the negative-limited cells relative to the positive-limited cells, but also migration patterns within a plate where positivelimited cells contain more cadmium in the bottom of the positive plates (Figure 8).

Theoretical plate capacity was calculated assuming a one-electron transfer of the active Ni(OH)₂ during discharge. Cell utilization based on the theoretical positive cell capacity is typically around 85 to 90 percent for the positive electrode in a new cell. Because of the negative-limited condition and capacity fading in negative-limited cells, cell utilization has been as low as 70 percent. When in a flooded state, all positive plate performed well, with utilization of 85 percent or greater.

Negative Electrode

Chemically, there are only slight variations in active material loading between cell lots (Table 6). Loading also changes with cycle life due to cadmium migration. The major differences in negative plate characteristics between the subject cells are in the electrochemical performance of the negative plate. Negative electrodes from negative-limited cells have shown approximately 60 percent negative plate utilization, whereas negative electrodes from positive-limited cells have achieved 75 percent negative plate utilization.

Electron microscopic examination has been performed on the cross section of the plate (Figures 9 and 10) to qualitatively judge the pore and active material distributions in the negative plates. Backscattered electrons were used to generate the images shown, and X-ray maps were made to distinguish particle composition on plate cross sections. In the cross-sectional images, the brighter areas were determined to be cadmium rich, while the gray areas are Due to a lack of gray level sinter. contrast, charged and discharged cadmium could not be separated. Voids in the plate appear black. Plates from negative-limited cells were found to have cadmium agglomerating in the center of the plates. This condition would cause charged cadmium in the center of an agglomeration to become isolated and thus electrochemically unusable. This is believed to be responsible for the measured low utilization in these electrodes.

Surface cadmium crystals were also examined by scanning electron microscopy (Figures 11 and 12). The crystal sizes on the negative plates from positive- and negative-limited cells were different. The majority of crystals in a positive-limited cell were 1 μ m in size, and occasionally a crystal as large as 20 μ m was found. Conversely, negativelimited cells contain many larger crystals.

Precharge and Overcharge Protection

From the data on both the chemical and electrochemical analyses, precharge and overcharge protection (OCP) values were calculated for each cell The values for these (Table 7). parameters have varied from cell to cell and lot to lot. Generally, with increased cycling, there has been increased precharge capacity and loss of OCP due to separator degradation and loss in negative electrode utilization. Cells that were diagnosed as being negative-limited have shown a slight increase in precharge levels.

CONCLUSION

The electrical characterization and subsequent DPA data on NASA Standard Aerospace Ni/Cd cells have been collected. For the cells investigated, the leading cause of poor performance was poor negative plate utilization, which resulted in a negative-limited condition. This condition has been found in several cells manufactured since the mid 1980s.



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ANALYSIS

FOR NASA/GODDARD SPACE FLIGHT CENTER **OF AEROSPACE NICKEL-CADMIUM CELLS**

COMSAT'S DESTRUCTIVE PHYSICAI

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CELL	HISTORY	TEST TYPE	Nameplate Capacity (Ah)	Plate Manufacturing Date	Cell Activation Date	Negativ Limite Conditio
IUE 3-010	ATP	RT storage	12	mid 1974	early 197	5
IUE 3-011	ATP	RT storage	12	mid 1974	early 197	5
TDRSS 15-78	ATP	cold storage	40	late 1981	mid 1983	
EUVE 16-053	ATP	cold storage	50	early 1985	early 198	ω
EUVE 4-005 back-up	ATP	cold storage	50	early 1990	late 1990) bordelin
EUVE 4-068 back-up	Battery ATP		50	early 1990	late 1990) yes
GOES 5-110	I AND T		12	late 1986	late 1986	
TDRSS L8-69	60 cycles	test battery	40	mid 1980	early 198	0
GRO 17-073	576 cycles	40 % DOD	50	early 1985	late 1986	
EUVE 16-030	2480 cycles	40 % DOD	50	early 1985	early 198	3 yes
EUVE 16-079	3900 cycles	15 % DOD	50	early 1985	early 198	.0
UARS 2-073	5010 cycles	15 % DOD	50	late 1988/1989	late 1989	yes
EUVE 16-014	5360 cycles	40 % DOD	50	early 1985	early 198	3 yes
UARS 2-007	5500 cycles	40 % DOD	50	late 1988/1989	late 1989	
UARS 2-021	5700 cycles	40 % DOD	50	late 1988/1989	late 1989	borderlin
COBE 15-005	6000 cycles	40 % DOD	50	mid 1985	late 1985	
EUVE 16-003	10600 cycles	15 % DOD	50	early 1985	early 1986	
GRO 17-063	11800 cvcles	15 % DOD	0	early 1085	0001 otcl	

TABLE 1: LIST OF NASA/GSFC CELLS ANALYZED

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NEGATIVE PLATES

Weight and Thickness

Weight and Thickness

POSITIVE PLATES

SEPARATOR Absorbency

ELECTROLYTE Composition Distribution

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- Tensile Strength
- Active Material Loading
 - Flooded Capacity
 - High Rate Cycling
 - Distribution

Cadmium Distribution

Loading

· Corrosion of Plaque

Tensile Strength

- Active Material

- Cadmium Migration
- Mechanical Strength Resistivity

Wicking

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ELECTRICAL CHARACTERIZATION

C/2 Rate of Discharge

 Charge Retention Charge Efficiency Voltage Recovery

Ni-Cd CELL DPA

07 ESS P	Ah) 0.1 V	63.7	63.0	62.6		73.3
) 2-0) STRI)SS OC	acity (62.1	61.5	61.3		61.1
2201 5501	Cap	1.533	1.570	1.569		1.571
е ш	h) 0.1 V	59.4	56.3	52.8	44.0 83.4	- 56.2
1 17-06 00 LIFI EG LIM	icity (A	58.4	55.2	51.8	43.1	48.7
LO1 N N	Capa EOCV	1.484	1.493	1.489	1.486	
Е 3	(h) 0.1 V	63.7	63.0	62.6	59.1 92.7	- 76.2
T 16-00 500 LIFI OS LIM	acity (A 1.0 V	63.4	62.2	62.1	56.7	62.9
С <u>1</u> 0 10 10	Cap EOCV	1.501	1.519	1.518	1.518	
ω <u>e</u>	Аh) 0.1 V	65.2	62.4	59.4		- 64.0
17 4-06 tery AT NEG LIM	acity (/ 1.0 V	64.5	61.6	58.4		57.2 -
Bat	Cap EOCV	1.487	1.495	1.487		1.484
23	Ah) 0.1 V	66.9	66.9	66.3	59.9 90.3	
T 16-0: ATP VOS LIM	acity (/ 1.0 V	66.3	66.1	65.0	58.6	64.0 -
<u>с</u>	Cap EOCV	1.490	1.503	1.494	1.492	1.492
	DCH	25	25	25	25 n	25 1Ω
	₽₹	2.5	S	5	5 , retur	5
	풍 또	48	16	16	16%	16
	(°C)	10	10	10	10	10
	CYCLE	recond	-	~	• ຕ	4 •

 charge retention test - 72 hour open circuit stand after complete charge percent of capacity remaining ** voltage recovery test - discharged with 1 Ω after power discharge to 1.0 V

then shorted for 16 hours

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CELL	HISTORY	Negative Limited	Nameplate Capacity (Ah)	Reported Fill Volume of 31 % (mL)	Volume (Theor KOH) (mL)	KOH Concentration (w/o)	K2CO3 Concentration (w/o)	Theoretical KOH (w/o)	cc/A
IUE 3-010	ATP		12	46	46.5	24.0	7.1	29.8	2.44
IUE 3-011	ATP		12	46	47.3	20.9	10.3	29.2	QN
TDRSS 15-78	ATP		40	120	123.9	27.3	3.5	30.1	2.06
EUVE 16-053	ATP		50	150	147.6	28.5	4.3	32.0	2.23
EUVE 4-005 backup	ATP	borderline	50	162	165.4	26.1	3.9	29.3	2.10
EUVE 4-068 backup	Battery ATP	yes	50	162	171.3	24.5	5.1	28.7	2.10
GOES 5-110	I AND T		12	30.5	35.1	18.9	11.0	27.8	2.03
TDRSS 8-69	60 cycles		40	120	129.2	21.5	7.4	27.6	2.05
GRO 17-073	576 cycles		50	163	161.8	26.5	5.1	30.7	2.14
EUVE 16-030	2480 cycles	yes	50	153	169.6	23.9	5.2	28.2	1.99
EUVE 16-079	3900 cycles		50	150	153.4	25.1	5.7	29.7	1.99
UARS 2-073	5010 cycles	yes	50	155	160.4	26.0	5.4	30.4	2.26
EUVE 16-014	5360 cycles	yes	50	153	160.7	20.6	5.8	25.6	1.73
UARS 2-007	5500 cycles		50	155	154.8	26.6	7.0	32.2	2.29
UARS 2-021	5700 cycles	borderline	50	163.8	174.9	24.2	6.7	29.7	2.35
COBE 15-005	6000 cycles		50	166	172.8	24.1	6.3	29.2	2.25
EUVE 16-003	10600 cycles		50	150	151.3	24.6	7.2	30.4	2.09

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TABLE 4: CADMIUM MIGRATION INTO THE SEPARATORS

		TOTAL IN A	LL SEPARATORS	HEAVIEST AREA
CELL	HISTORY	(g/cell)	(mg/sep. area)	(mg/cm^2)
TDRSS 15-78	ATP	0.40	0.16	0.7
EUVE 16-53	ATP	0.51	0.11	2.6
EUVE 4-05 backup	ATP NL	0.40	0.09	6 .0
EUVE 04-068 backup	Battery ATP NL	0.61	0.13	1.6
GOES 05-110	I and T	0.10	0.05	0.4
TDRSS 8-69	60 cycles L	0.76	0.31	0.6
GRO 17-073	576 cycles S	0.49	0.11	0.9
EUVE 16-30	2480 cycles S NL	0.51	0.11	0.7
EUVE 16-79*	3900 cycles L	1.82	0.40	9.6
UARS 2-073	5010 cycles L NL	1.28	0.28	5.9
EUVE 16-014	5360 cycles S NL	0.68	0.15	1.1
UARS 2-007	5500 cycles S	12.10	2.65	7.6
UARS 2-021	5700 cycles S NL	1.37	0.30	1.5
COBE 15-5	6000 cycles S	1.00	0.22	8.3
EUVE 16-003*	10600 cycles L	2.86	0.63	9.9
GRO 17-063 *	11800 cycles L NL	1.14	0.25	11.7

* = separator sticking may effect results for Cd in all separators. NL = capacity limited by the negative electrode

- S = stress cycling, 40 % DOD
- L = life cycling, 15 % DOD



					Cinter	- Cubatrato	Porosity	Porosity	Load	ling Total	Utiliza	ation
Identification	History	2(UO)IN (%)	cu(UU)z	cu(On)z (%)	3111E	(%)	(%)	anhei	(3/CC)	(3/cc)	(%)	(%)
IUE 3-10	ATP	44.5	2.7	3.1	28.3	19.8	87.8	80.2	1.93	2.25	76.8	74.0
TDRSS 15-78	ATP	41.3	2.6	5.6	30.6	19.8	86.8	79.2	1.82	2.18	92.9	94.4
EUVE 16-53	ATP	41.4	2.1	4.4	28.4	20.0	87.6	79.7	1.84	2.29	89.7	87.3
EUVE 4-05 backup	ATP	44.7	2.6	3.6	28.2	17.7	87.9	81.0	1.94	2.34	83.2	75.0 NL
EUVE 4-68 backup	BATT	44.9	2.6	3.6	28.7	17.3	87.8	81.0	1.94	2.34	84.1	75.1 NL
GOES 5-110	I and T	42.4	2.8	3.4	33.1	16.4	85.9	79.6	1.87	2.23	83.0	83.0
TDRSS 8-69	60 Life	43.2	2.9	4.3	32.1	17.4	85.3	78.3	2.06	2,40	72.9	75.8
GRO 17-73	576 Stress	43.9	1.9	5.0	28.9	18.5	87.4	80.0	1.95	2.34	84.1	17.3
EUVE 16-30	2480 Stress	43.7	1.6	5.9	28.3	18.8	87.2	79.6	2.01	2.43	78.7	63.0 NL
GRO 16-79	3900 Life	43.5	2.4	4.7	28.4	19.1	87.5	79.9	1.94	2.35	86.4	83.4
UARS 2-73	5010 Life	41.9	2.2	4.5	30.6	19.1	87.1	79.8	1.81	2.17	83.1	71.4 NL
EUVE 16-14	5360 Stress	42.5	2.1	7.3	28.7	18.3	87.06	79.6	2.0	2.44	78.2	63.3 NL
UARS 2-07	5500 Stress	41.6	2.2	5.5	29.8	19.4	87.7	80.4	1.74	2.13	84.6	84.4
UARS 2-21	5700 Stress	42.6	2.4	5.5	28.4	18,4	88.2	81.1	1.79	2.24	84.2	78.6 NL
COBE 15-5	6000 Stress	42.0	2.2	5.7	29.3	20.3	87.7	79.9	1.80	2.15	85.0	78.3
EUVE 16-03	10600 Life	41.7	2.1	6.7	29.4	18.1	87.0	79.8	1.88	2.37	85.4	84.4
GRO 17-63	11800 Life NL =	46.3 negative	2.1 limited	5.0	27.4	19.1	88.2	80.7	2.01	2.32	81.0	59.7 NL

TABLE 5: POSITIVE PLATE ANALYSIS COMPARISON

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Ni Ni(OH)2 Fe Loading (g Cd (%) (%) (%) Cd(OH)2 Cd
35.3 3.7 14.1 0.341
34.5 2.8 15.0 0.331
31.1 3.6 14.0 0.356
33.2 3.8 14.4 0.321
34.3 4.1 13.8 0.276
33.9 1.6 15.6 0.296
33.7 3.9 13.7 0.339
35.9 3.4 12.8 0.323
34.1 3.8 13.7 0.284
32.1 3.7 14.7 0.335
35.2 4.5 13.3 0.277
36.4 4.4 14.2 0.283
36.5 3.7 14.3 0.285
36.3 4.4 13.5 0.251
33.1 4.7 14.7 0.269
34.4 3.6 14.6 0.305
35.2 4.2 12.4 0.291

TABLE 6: NEGATIVE PLATE ANALYSIS SUMMARY



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ORIGINAL PACE IS OF POOR QUALITY



NEC 2.73 = 17 1206/15 A GENERAL PAOR G GENERAL PAOR G CF POOR QUALARY

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OF POOR GEALBY





General Topic Session

GELEVEL FUNCTION FIGURE 12: SEM Analysis of Bottom of Negative Plate #15 - UARS Lot 2 S/N 21



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TABLE 7: PRECHARGE AND OVERCHARGE PROTECTION

		Chemical		Negative			•	Humanitable
		Negative	Cell	Flooded		Precharge	Overcharge	Unavailable Disabourad Cd
		Capacity	Capacity	Capacity		Total	Protection	Uischarged Co
Identification	History	(Ah)	(Ah)	(Ah)		(Ah)	(An) 10.05	<u>(All)</u>
IUE 3-10	ATP	34.8	13.96	22.42		4.27	10.05	10.32
			40.1%	64.4%		12.3%	28.9%	10.770
EUVE 16-053	ATP	129.11	62.81	100.81		21.10	26.95	18.28
			48.6%	78.1%		16.3%	20.9%	14.2%
EUVE 4-005	ATP	138.84	57.78	86.7		32.68	23.36	24.82
backup			41.6%	62.4%	NL	23.5%	16.8%	17.9%
EUVE 4-068	Battery	133.39	57.21	78.12		39.85	20.99	14.56
backup	ATP		42.9%	58.6%	NL	29.9%	15.7%	10.9%
TDRSS 15-78	ATP	106.98	47.86	61.81		11.16	13.29	34.67
			44.7%	57.8%		10.4%	12.4%	32.4%
GOES 5-110	I and T	27.09	12.63	19.16		8.27	3.36	5.93
			46.6%	70.7%		30.5%	12.4%	21.9%
TDRSS 8-69	60 Life	103.08	55.46	72.75		11.85	8.34	27.43
			53.8%	70.6%		11.5%	8.1%	26.6%
GRO 17-073	576 Stress	134.35	59.8	81.43		32.06	21.5	20.99
			44.5%	60.6%		23.9%	16.0%	15.6%
GRO 16-030	2480 Stress	130.79	50.42	68.1		45.55	5.29	29.53
			38.6%	52.1%	NL	34.8%	4.0%	22.6%
GRO 16-079	3900 Life	125.81	65.13	96.28		15.91	22.25	22.52
			51.8%	76.5%		12.6%	17.7%	17.9%
UARS 2-073	5010 Life	123.82	51.39	76.17		39.64	17.03	15.76
			41.5%	61.5%	NL	. 32.0%	13.8%	12.7%
GRO 16-014	5360 Life	126.18	47.07	68.88		43.7	7.71	25.41
			37.3%	54.6%	NL	. 34.6%	6.1%	20.1%
UARS 2-007	5500 Stress	s 118.60	61.09	87.55		34.85	3.16	19.50
			51.5%	73.8%		29.4%	2.7%	16.4%
UARS 2-021	5700 Stress	s 124.57	55.35	77.35		53.69	9.97	5.56
			44.4%	62.1%	NL	43.1%	8.0%	4.5%
COBE 15-05	6000 Stres	s 129.46	59.56	70.91		37.90	1.78	30.22
			46.0%	54.8%		29.3%	1.4%	23.3%
GRO 16-003	10600 Life	128.34	62.93	82.28		42.36	12.93	10.12
			49.0%	64.1%		33.0%	10.1%	7.9%
GRO 17-063	11800 Life	e 135.67	48.67	72.07		38.38	12.58	33.17
			35.9%	53.1%	N	L 28.3%	9.3%	24.4%

Percentages based on overall negative capacity

NL = negative limited on discharge

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Nickel-Hydrogen Storage / Capacity Fade Session

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