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Stress test evaluation of cobalt-enhanced nickel plaque electrodes

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Abstract

The addition of cobalt to the surface of aerospace-quality sintered nickel plaque was observed to improve plate performance during a 5000 cycle stress test. A two-level, four-factor full factorial design matrix was established to compare the performance of cobalt-enhanced nickel plates with standard aerospace quality nickel plates during testing. The three other factors were loading level, current density during electrochemical impregnation and the concentration of KOH electrolyte. Regression analysis after 4000 cycles indicates that nickel plates fabricated from cobalt-enhanced plaque using a low current density during the electrochemical impregnation step provides the best performance during high rate charge/discharge applications. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Sintered nickel electrode; Cobalt additive; KOH electrolyte; Capacity utilization; Stress test; Loading level; Electrochemical impregnation current density

1. Introduction

Cobalt addition to sintered nickel plaque at the sinter/active material interface increases plate utilization and improves cycle life at high discharge rates [1]. The purpose of this paper is to compare the effect(s) of cobalt-enhanced sintered nickel plaque with standard nickel plaque during stress test evaluation of aerospace-quality nickel electrodes. A two-level, four-factor full factorial experimental design matrix was setup to compare the soak/re-sinter cobalt-enhanced nickel plates [2] with standard aerospace-quality nickel plates during stress test. Three other important parameters that influence plate performance during cycling are: the active material loading level (LL, g/cm^3 void volume), KOH electrolyte concentration and the electrochemical impregnation current density (EICD). Stress test evaluation on plates with high and low levels of these three factors and two levels of sintered plaque (cobalt-enhanced vs. standard) was performed.

2. Experimental

2.1. Fabrication of nickel electrodes

Fabrication of sintered nickel plaque and all nickel electrode processing steps was carried out in Yardney's Nickel–Hydrogen Battery Production Facility. Cobalt-enhanced plaque masterplates were prepared using 0.5 M, 1.0 M or 2.0 M $Co(NO_3)_2$ solution by the procedure outlined in a previous paper [1]. Low and high $Ni(OH)_2$ active-material loading levels were controlled to approximately 1.5 and 1.65 g/cm^3, respectively. The EICDs selected for plaque impregnation were 0.25 and 0.50 A/in.^{2} based on submerged plaque masterplate area. Five samples were fabricated for each loading level of each plate type in the full factorial test design matrix. The impregnation times were obtained from an EI loading curve in one or more preliminary runs for each plate type.

2.2. Experimental design

A two-level four-factor full factorial experimental design was selected for evaluation of four parameters that have considerable influence on the nickel electrode performance for aerospace applications. The four independent

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and controlled parameters are: (1) plaque type; (2) EICD; (3) active material loading level; and (4) the KOH electrolyte concentration. Two levels of each parameter were selected, giving a total of 16 combinations: (1) 2 M cobalt-treated and standard plaque; (2) EICDs of 0.25 and 0.50 A/in.^{2} of submerged plaque masterplate; (3) low and high loading levels in the range 1.37 to 1.70 g/cm^3; and (4) KOH electrolyte concentrations of 26% and 31%. The design was duplicated as opposed to replicated and augmented with four additional plates within the range of the experimental design levels. These additional plates were fabricated from 0.5 M and 1.0 M cobalt-enhanced plaque, two plates of each plaque type, making a total of 36 plates for stress test evaluation.

2.3. Cell assembly and test conditions for stress test evaluation

During stress-test evaluation, each plate was centered between two Nickel 200 sheet counter electrodes in an individual cell filled with either 26% or 31% KOH electrolyte. A high rate of charge, overcharge and discharge was selected to bring out differences in plate performance during stress test evaluation. Each plate was charged at the 10 C rate for 12 min, then discharged at the 10 C rate for 6 min or until plate voltage reached -1.6 V (knee voltage at the 10 C discharge rate) vs. the counter electrodes. To obtain maximum plate performance, the KOH electrolyte in each cell was replaced after every 1000 cycles during the first 3000 cycles, then every 500 cycles thereafter. The electrolyte loss is attributed to the large degree of gassing at the cycling rates employed. Water was added, as required, during each 1000-cycle run to maintain adequate electrolyte level in each cell. Flooded capacity measurements were made prior to the stress test, then at 200 and 1000 cycles during the first 1000-cycle run, and after each additional 1000 cycle run. Thickness measurements were made prior to stress test, and after 200, 1000 and 2000 cycles. Flooded capacity measurements were carried out in the stress test cells. The plates were charged at the C rate for 2 h, then discharged at the C rate until plate voltage reached -1.0 V (knee voltage at the 1 C discharge rate) vs. the counter electrodes. The stress test plates were grouped by weight gain capacity (0.289 A h/g active material). Each of the six computer controlled stress test stations could contain up to eight cells connected in series. The 10 C rate for a given station was based on the lowest theoretical capacity in its group of plates. Each plate was ultrasonically rinsed and thoroughly dried before taking thickness measurements and checking for the presence of blisters (small raised blemishes on either side of a plate).

2.4. Measurement of sinter corrosion

Eight plates were examined for sinter corrosion after 5000 cycles of stress test evaluation. The active material was removed from plate samples by deloading (dissolving) the active material in an ammonia solution of the disodium salt of ethylenediaminetetraacetic acid (EDTA). Each electrode sample was placed in a beaker with 0.1 g of hydrazine sulfate and 150 ml of the EDTA solution, covered with parafilm and heated to $65 \pm 5°C$ for 20 h. After several rinses in warm D.I. water, the sample was dried and weighed for comparison to the calculated initial sinter weight of a same-size sample based on the initial plate plaque weight and final weight gain after EI, conversion and formation during plate fabrication. The deloading solution was analyzed for Co and Ni by Direct Current Plasma (DCP) instrumentation.

3. Results and discussion

3.1. Experimental design stress test data

Sixteen plates were electrochemically impregnated at a current density of 0.5 A/in.^{2}. These plates were cycled for a total of 4000 cycles. Several of the eight standard plates lost active material and nickel sinter by the end of 3000 cycles. The 10 C rate discharge time had decreased to a range of 3.25–3.78 min for the eight standard plates by cycle 3000. By comparison, the cobalt-enhanced plates were still delivering from 4.88 to 5.92 min of 10 C rate discharge capacity after 3000 stress test cycles. These results indicated that the use of cobalt-enhanced plaque is an important factor in determining plate performance. The 16 plates were then stress tested for an additional 1000 cycles to further evaluate the difference between cobalt-enhanced and standard plates. The average discharge time during this final 1000 cycle run and the capacity measurement after the 4000th stress test cycle were used in the regression analysis.

Twenty plates were impregnated at a current density of 0.25 A/in.^{2}. These plates were cycled for a total of 5000 cycles. The cobalt enhanced plates 551 and 554 in Table 1, maintained an average 6.0-min discharge time during the 5000 cycle stress test while discharge times for the corresponding standard plates, plates 422 and 564 in Table 1, gradually dropped to less than 3.5 min. Flooded capacity utilization for the two standard plates dropped to less than 70% after 5000 cycles while the two cobalt plates maintained utilizations above 100%. These four plates had low $Ni(OH)_2$ loading levels, 1.37–1.43 g/cm^3, and were tested in 31% KOH electrolyte. The cobalt-enhanced plates with low loading levels cycled in 26% KOH, plates 516 and 561 in Table 1, maintained a 6.0-min discharge time after 5000 cycles while the discharge time for the corresponding standard plates, plates 539 and 550 in Table 1, dropped below 3.5 min.

Table 1
Parameter values and four independent variables for 36 stress test plates

Plate S/N	Plaque type, 0 = Std, 0.5, 1, 2 = Co [M]	EICD (A/in.^{2})	Loading level (g Ni (OH_2)/ cm^3 void volume)	Percent KOH	Total discharge time in min after 4000 stress test cycles	Percent utilization after 4000 stress test cycles	Number of blisters after 3000 stress test cycles	Percent change in thickness after 2000 stress test cycles
554	2	0.25	1.37	31	24118.8	108.6	21	−2.0
551			1.43		24117.8	105.6	85	−13.4
516			1.49	26	23798	103.1	3	−16.5
561			1.54		23721	96.9	71	2.3
472			1.68	31	23225	101.7	4	5.4
464			1.65		22841	103.9	60	4.4
449			1.69	26	22658.2	101	27	8.3
461			1.68		22506	102.2	0	4.4
573		0.5	1.51	31	22339	67.4	250[a]	7.7
585			1.55		20329	70.5		0.0
598			1.54	26	21240.2	58.9		1.9
582			1.53		21135.6	60.5		3.3
455			1.67	31	22593.8	71.7		6.7
487			1.70		20311	54.6		4.6
488			1.69	26	20819.6	54.1		2.0
483			1.70		20662.8	52.1		3.7
75	1	0.25	1.44		22928	105.6	7	0.7
74			1.48		22739	104.3	0	−0.3
44	0.5		1.57		23720	98	40	1.0
40			1.59		22996	95.3	17	3.0
564	0		1.40	31	20525.8	76.7	3	−1.3
422			1.40		20118.8	75.2	2	−23.7
539			1.50	26	20567	68.9	0	6.8
550			1.53		20524			0.7
497			1.62	31	20060	72.4	20	4.7
517			1.63		19634		27	3.0
531			1.64	26	20104	65.5	7	3.4
544			1.64		20053	66	28	4.4
14		0.5	1.55	31	18501.8	65.2	35	6.8
18			1.54		18183.8	62.7	16	2.2
24			1.54	26	17962.6	67.3	20	3.2
21			1.54		17780.8	64.4	19	3.5
656			1.64	31	19269.8	69.4	65	12.9
655			1.64		19119.8	67.5	29	5.7
1114			1.66	26	18654.2	70	44	8.2
1122			1.67		18490.6	68.5	68	12.7

[a]Upper limit.
EICD, Electrochemical impregnation current density.
Std, Standard sintered plaque.

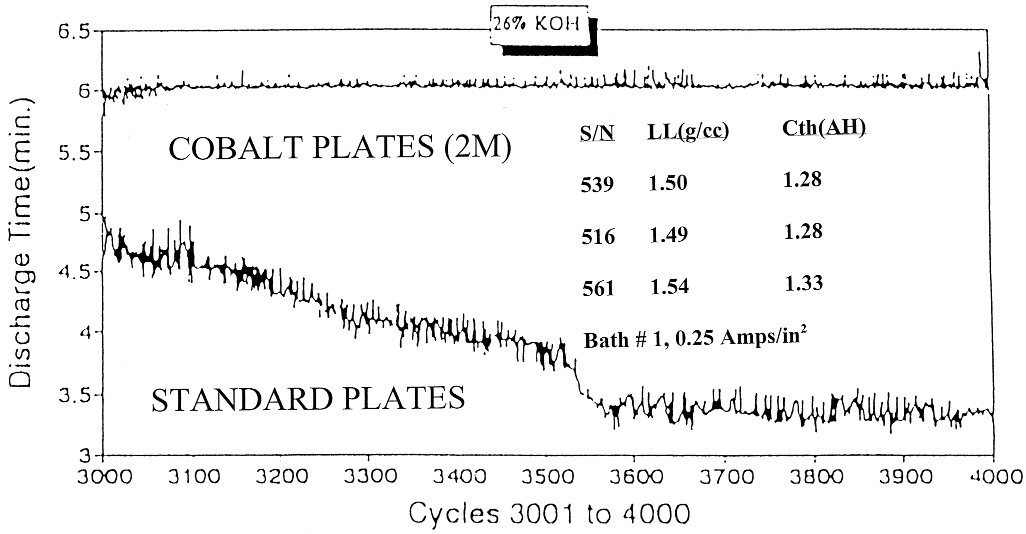

Fig. 1. Discharge time vs. stress test cycle in 26% KOH electrolyte for low loading standard and cobalt-enhanced plates from 3001 to 4000 cycles.

Discharge time vs. stress test cycle number from cycle 3001 through cycle 4000 is plotted in Figs. 1 and 2 for selected combinations of plaque type, stress test electrolyte concentration and active material loading level. The common factor for each group of four plates is the low EICD used during plate fabrication. In each of these figures, plates fabricated with cobalt-enhanced plaque discharged, on average, a full 6 min at the 10 C rate while discharge times for the standard plates in Fig. 1 decreased from between 4 and 5 min during cycle 3001 to between 3 and 3.5 min during cycle 4000 and in Fig. 2, the standard plate discharge times remained between 3 and 4 min throughout the 1000-cycle interval.

3.2. Analysis of plate performance after 4000 stress test cycles

A summary of the parameter values for the four independent variables and data for the four dependent variables for each of the 36 plates is presented in Table 1. The dependent variables used for regression analysis were: (1) total discharge time during the 4000 stress test cycles; (2)

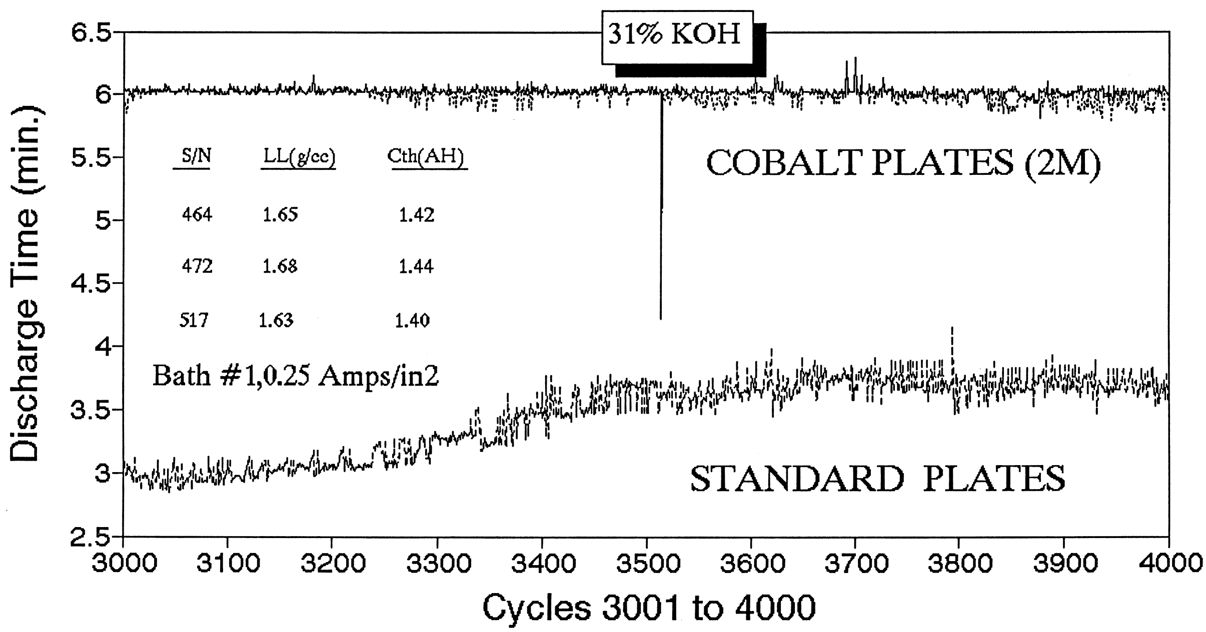

Fig. 2. Discharge time vs. stress test cycle in 31% KOH electrolyte for high loading standard and cobalt-enhanced plates from 3001 to 4000 cycles.

percent utilization of capacity during measurement at the C rate after 4000 stress test cycles; (3) number of blisters observed after 3000 stress test cycles; and (4) percent change in electrode thickness after 2000 stress test cycles. An upper limit of 250 blisters was assigned to the eight plates fabricated from cobalt-enhanced plaque and electrochemically impregnated at a current density of 0.5 A/in.^{2} since it was difficult to count the actual number of blisters. Accurate thickness measurements could not be made on these eight plates and a few of the other plates with many blisters after 3000 cycles, for that reason, the 2000-cycle percent change in thickness values were used for statistical analysis. The statistical and regression analysis was done using Statistica 6.0 by Stat Soft.

Examination of the stress test data in Table 1 shows the following: (1) the best performance based on total discharge time during the 4000 stress test cycles was observed for seven of the eight plates fabricated with the 2.0 M cobalt-enhanced plaque and 0.25 A/in.^{2} EICD combination. For comparison, poor performance was observed for the eight plates fabricated with the standard nickel plaque and 0.5 A/in.^{2} EICD combination; and (2) the best performance based on capacity utilization after 4000 stress test cycles was observed for the eight plates fabricated with the 2.0 M cobalt-enhanced plaque and 0.25 A/in.^{2} EICD density combination. For comparison, the lowest capacity utilization values were observed for eight plates fabricated using the 0.5 A/in.^{2} EICD, three with standard plaque and five with 2.0 M cobalt-enhanced plaque.

These results suggest that plate performance during stress test, and by implication, other high-rate cycling regimes is improved by fabricating plates from cobalt-enhanced plaque and impregnated at a low value of EICD.

Regression analysis on the results for the four independent variables in Table 1 are presented in Figs. 3–6, respectively. The 36 test runs permitted full resolution of the main effects and interactions. Duplicate runs were treated as replicates for the purpose of estimating standard error and the coefficient of multiple determination for regression equation results. This suggests that the resulting regression equation coefficients might be slightly biased. A two-dimensional plot showing the effect of a dominant factor and interaction term on one of the independent variables is presented in Figs. 3–5. In each case, the dominant factor was plaque type, with the plotted range extending from 0.0 for standard plaque to a value of 2.0 for the 2.0 M cobalt-treated plaque. The plotted range for the interaction term extends from the lowest to the highest value for the product of the two factors. Each plot is separated into several narrow ranges of value for the independent variable. A linear regression equation is presented in Fig. 6, since only one factor, the active material loading level, was found to contribute to plate expansion.

In the absence of cobalt-enhanced plaque, plates fabricated with a low loading level from standard plaque using a low EICD would be expected to have the best cycle life performance in a LEO regime at 40% DoD. When cobalt-enhanced plaque is added as the fourth factor, regression

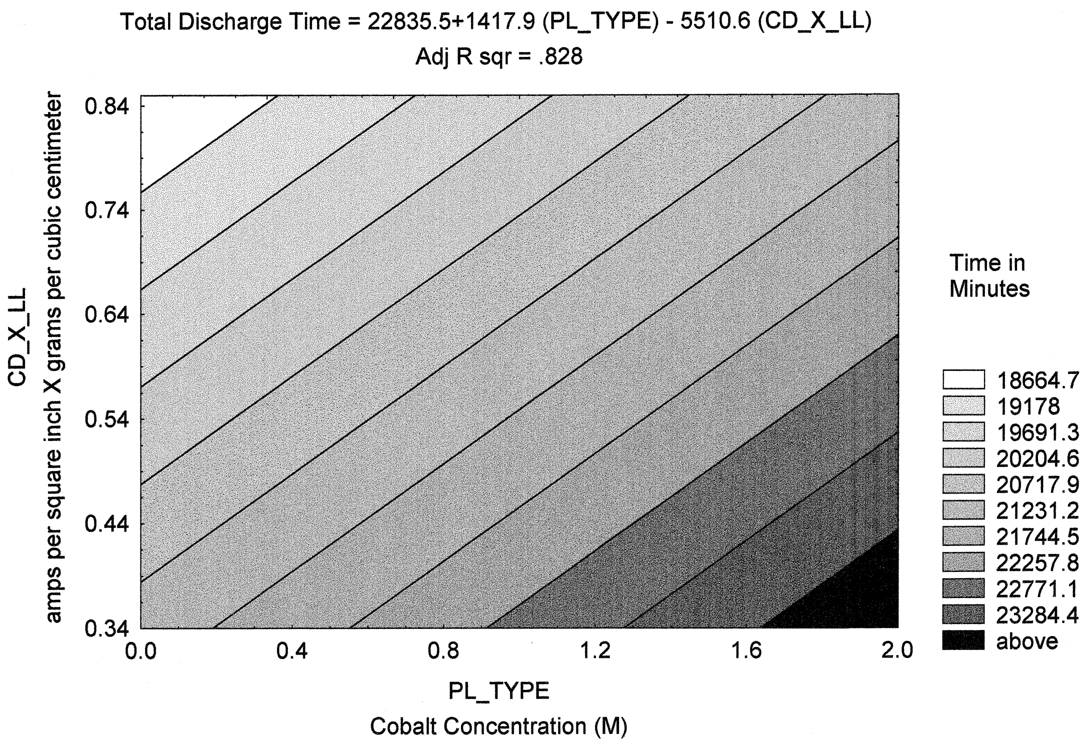

Fig. 3. Total discharge time after 4000 stress test cycles.

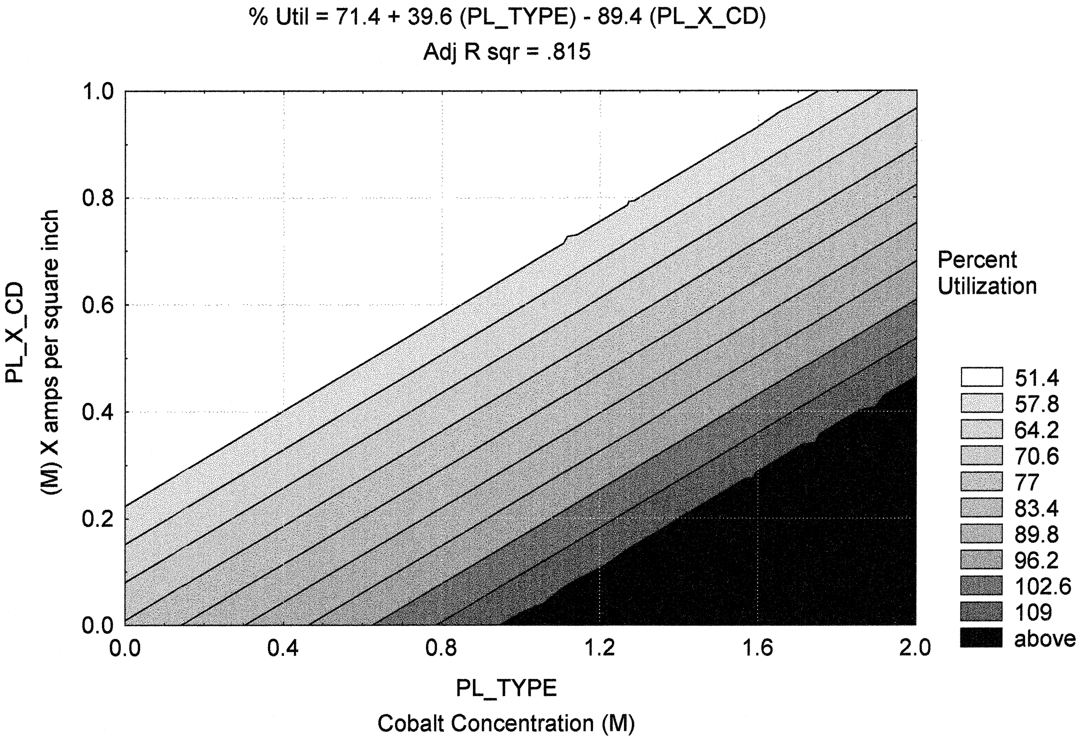

Fig. 4. Percent utilization after 4000 stress test cycles.

analysis provides further insight on plate performance during high rate applications. Three factors, plaque type, EICD and loading level accounted for approximately 83% of the total discharge time during 4000 stress test cycles. From the regression equation in Fig. 3, cobalt-enhanced plaque makes a positive contribution to the total discharge time while the EICD and loading level appear as an interaction term $(CD \times LL)$ that detracts from it. The maximum discharge time was achieved for plates fabricated using 2.0 M cobalt-enhanced plaque, a 0.25-A/in.^{2}

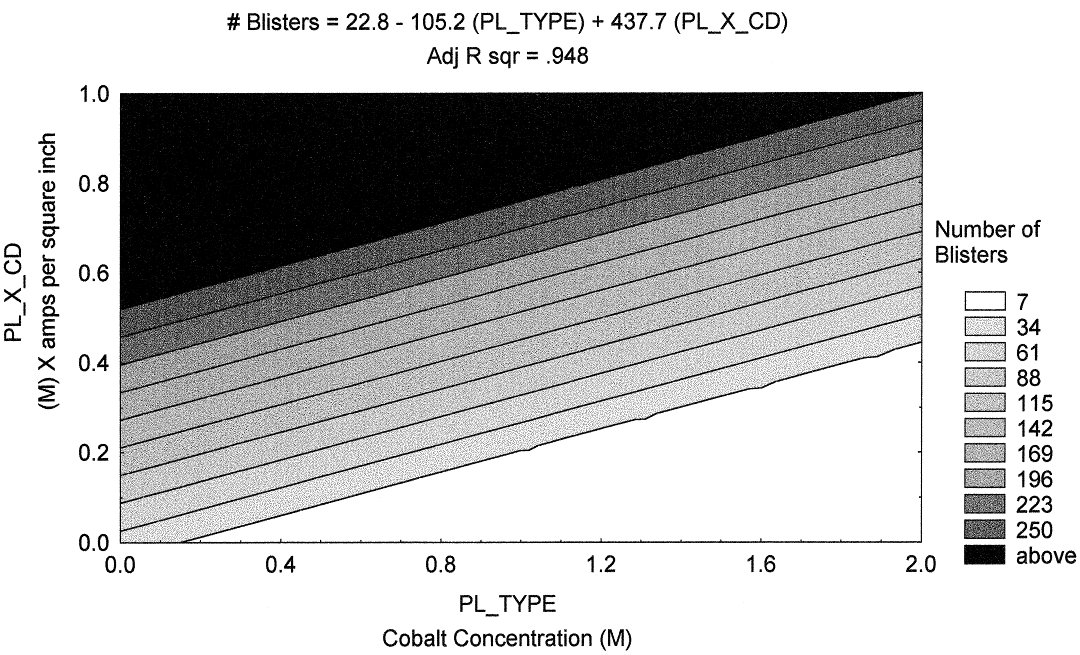

Fig. 5. Number of blisters after 3000 stress test cycles.

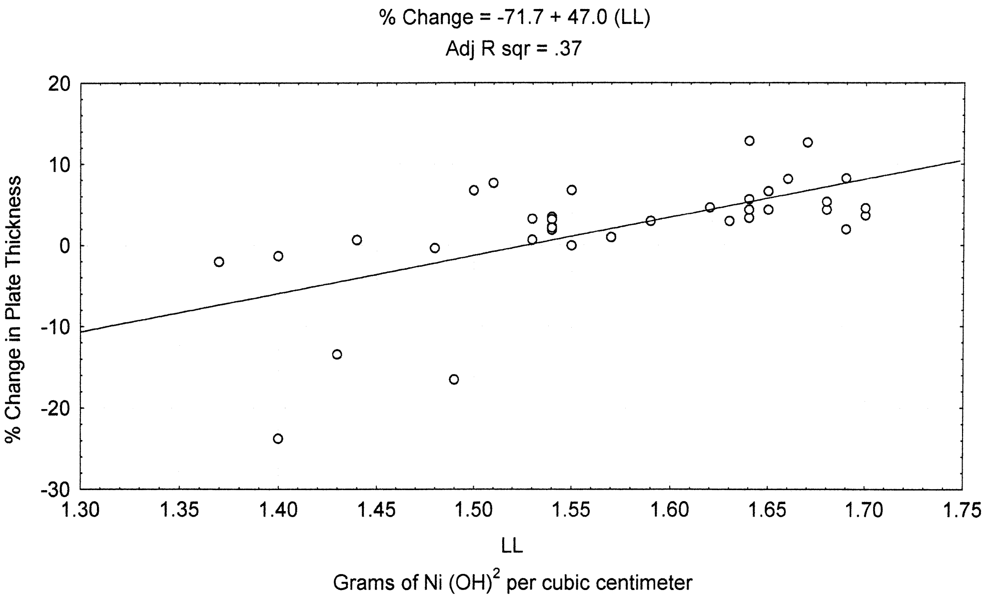

Fig. 6. Percent change in electrode thickness after 2000 stress test cycles.

EICD and, for the top four plates, low values of the loading level, in the range 1.37–1.54 g/cm^3. Two factors, plaque type and EICD accounted for approximately 82% of the capacity utilization following 4000 stress test cycles. In the regression equation of Fig. 4, cobalt-enhanced plaque appears to be the most important factor, but because of the interaction term between plaque type and EICD, PL × CD, plate fabrication requires a low EICD along with cobalt-enhanced plaque to achieve maximum capacity utilization. The absence of the loading level parameter from the regression equation in Fig. 4 can be attributed to the relatively low charge/discharge rate used during capacity utilization measurements, i.e., the 1 C rate vs. 10 C rate during stress test.

The same two factors account for 95% of the blisters that occurred during stress test. For standard plaque, the number of blisters that developed during 3000 stress test cycles was on the low end, but showed an increase with increase in current density. The maximum number of blisters occurred for the cobalt-enhanced plaque and high EICD combination, which shows up as the interaction term with large positive coefficient in the regression equation, Fig. 5.

Several factors contribute to blister formation [3]: (1) plaque strength; (2) charge rate and the percent of overcharge during the charge step; (3) plate loading level; and (4) the current density used in electrochemical impregnation of active material into the plaque. These factors have an effect on the reversible and irreversible expansion that occurs during cycling. The active mass, expanding during discharge and contracting during charge, exerts a cyclic force on the supporting plaque structure. The cyclic strain

Table 2
Summary of plate capacity utilization for 20 design matrix plates fabricated with 0.25 A/cm^2 EICD

Plate S/N	Percent utilization for 8 standard and 12 cobalt-enhanced plates									
	539/550	516/561	422/564	551/554	531/544	449/461	497/517	464/472	40/44	74/75
Plaque type 0 = Std 0.5, 1, 2 = Co [M]	0	2.0	0	2.0	0	2.0	0	2.0	0.5	1.0
Average LL (g/cm^3)	1.51	1.51	1.40	1.40	1.64	1.68	1.62	1.66	1.58	1.46
KOH (%)	26	26	31	31	26	26	31	31	26	26
Average capacity after cycle[a]										
0	135	132	133	130	118	135	138	135	129	129
200	139	138	139	138	129	140	139	139	135	137
1000	134	128	128	128	130	134	129	136	121	130
2000	112	115	97.4	103	120	123	110	127	106	113
3000	87.8	97.7	86.1	105	86.1	120	77.1	105	88.2	103
4000	68.9	100	76.0	107	65.7	102	72.4	103	96.6	105
5000	67.0	99.7	67.0	107	59.0	101	59.7	76.0	77.1	101

[a]Average value of each experimental design plate and its duplicate.
EICD, Electrochemical impregnation current density.

Table 3
Sinter corrosion in standard and cobalt-enhanced plates after 5000 cycle stress test

Plate S/N	Plate parameter		KOH concentration (2) (%)	Sinter weight (3) prior to stress test (g)	Sinter weight loss (4) after stress test	Sinter corrosion (%)	Deloading (5) solution analysis (g)		Co as % of Ni
	Plaque type (1)	LL (g/cm^3)					Ni	Co	
561	Co	1.54	26	0.676	0.094	13.9	0.292	0.033	11.3
551	Co	1.43	31	0.767	0.126	16.4	0.327	0.035	10.7
461	Co	1.68	26	0.747	0.123	16.5	0.339	0.044	13.0
472	Co	1.68	31	0.758	0.126	16.6	0.353	0.041	11.6
550	Std	1.53	26	0.693	0.083	12.0	0.330	0.020	6.1
564	Std	1.40	31	0.696	0.115	16.5	0.323	0.016	4.9
544	Std	1.64	26	0.702	0.138	19.6	0.355	0.021	5.9
517	Std	1.63	31	0.684	0.118	17.2	0.350	0.019	5.4

(1) Std, Standard sintered plaque; Co, cobalt-enhanced plaque surface.
(2) Stress test electrolyte.
(3) Calculated weight is based on initial plate plaque weight and final plate weight gain after formation.
(4) Same size sample was used to calculate the sinter weight in (3). This sample was deloaded and weighed to determined sinter weight loss due to high rate stress test cycling.
(5) Deloading solution was analyzed for Co and Ni by DCP.

produced on this structure gradually weakens the electrical and mechanical properties of the plate, causing failure. Cyclic strain is greatest during the end of discharge [4]. The breakdown in mechanical properties along with gassing during overcharge can lead to blister formation. The most likely explanation for the increased blistering of cobalt-enhanced plates is that these plates experienced more strain on the supporting plaque structure due to their longer discharge times than the standard plates.

The loading level parameter accounts for only 37% of the observed change in plate thickness after 2000 stress test cycles. Part of the reason for the low value of R^2 was the difficulty in making accurate thickness measurements as they depend on the extent of discharge and amount of rinsing and drying. The linear regression equation in Fig. 6 suggests that plate expansion increased with loading level regardless of plaque type, EICD or electrolyte concentration.

3.3. Plate capacity utilization during 5000 cycle stress test

A summary of average plate capacities as a function of cycle number are summarized in Table 2 for the plates fabricated with the low EICD (0.25 A/in.^{2}). The data shows that the maximum capacity occurred after 200 stress test cycles. At this point, the average capacity utilization ranged from a low value of 129% to a high value of 140%. Capacity utilization differences between plates made with standard plaque and the 2.0 M cobalt-treated plaque start to show up after 2000 stress test cycles. This difference increases with each additional 1000 cycle run. After 5000 cycles, the average capacity utilization for the standard plates ranged from a low value of 59.0% to a high value of 67.0%. By comparison, the average capacity utilization for six of the eight plates made with 2.0 M cobalt-treated plaque, ranged from a low value of 99.7% to a high value of 107%. The average capacity utilization was 76.0% for the other two cobalt-enhanced plates. Both of these plates had a high loading level of active material and were tested in 31% KOH electrolyte, not the best combination for maximum plate performance.

3.4. Sinter corrosion after 5000 cycles

Eight nickel electrodes, four fabricated with standard plaque and four with cobalt-enhanced plaque, were examined for sinter corrosion after completing 5000 stress test cycles. These plates were electrochemically impregnated at a 0.25 A/in.^{2} current density. The results presented in Table 3, indicate that the degree of sinter corrosion that occurred during the stress test is the same for both plaque types within experimental error. The presence of additional cobalt at the sinter/active material interface does not appear to increase the rate of sinter corrosion during high rate cycling.

4. Conclusions

Addition of cobalt to the surface of sintered nickel plaque improves plate performance during stress testing, and by implication, during other high rate applications. The evidence suggests that cobalt forms a nickel/cobalt alloy which improves plaque strength, as observed during plate handling. Each plate was inserted into or removed from a cell container a total of 11 times during the 4000 stress test cycles, or 13 times for the plates cycled for 5000 cycles. Several of the standard plate tabs, but none of the cobalt-enhanced plate tabs broke away from the plate coined tab area during handling after 3000 or 4000 cycles.

One concern is that addition of cobalt to the plaque surface would increase the rate of plaque corrosion during high rate applications. The results suggest that the presence of cobalt at the plaque surface/active material interface did not increase the rate of plaque corrosion during stress test evaluation.

The stress test results indicate that the combination of a high EICD and high loading level is detrimental to plate performance during extended periods of high rate cycling even if the plate is fabricated from cobalt-enhanced plaque. Several of the standard and 2.0 M cobalt-treated plates fabricated at high current density (0.5 A/in.^{2}) lost active material and nickel sinter after 3000 stress test cycles. A similar result was not observed for the 20 plates fabricated at 0.25 A/in.^{2} current density after 3000 cycles. However, after 4000 cycles, active material and nickel sinter loss was observed for cobalt-enhanced plates fabricated at low current density. The two plates affected, 464 and 472, had a high loading level and were cycled in 31% KOH electrolyte.

The best results obtained during high rate cycling were with plates fabricated from cobalt-enhanced plaque to a low loading level value using the low EICD. These four plates, cycled in either 26 or 31% KOH electrolyte for 5000 cycles, did not lose active material or nickel sinter. Moreover, capacity utilization for these plates ranged from 96.2 to 108% after 5000 stress test cycles.

A non-uniform distribution of active material over the sintered plaque internal surface area occurs during plate fabrication in a bath using a high value of EICD, a result which can lead to blister formation, especially during high rate stress test cycling. The number of blisters is observed to decrease for plates fabricated in a bath with the low value of the EICD. This result can be attributed to the uniform distribution of active material, a factor that should reduce the build-up of cyclic strain. For the best plate performance combination, cobalt-enhanced plaque and low EICD, the number of blisters after 3000 stress test cycles ranged from 0 to 85 blisters. The plates fabricated from standard plaque and low EICD had the fewest number of blisters, i.e., less than 30 blisters per plate. However, because these plates did not provide as much capacity, i.e., they did not experience cyclic strain to the same extent as the cobalt-enhanced plates, they had fewer blisters after 3000 stress test cycles.

One explanation for the results observed during stress test evaluation is the addition of cobalt to the surface of the sintered nickel plaque prevents the plaque surface/active material interfacial resistance from increasing as the nickel sinter corrodes during periods of high rate cycling to the same degree as would be expected to occur in the absence of cobalt. The corroded nickel containing cobalt is charged and discharged during cycling along with the original active material. The presence of cobalt in the active material has been found to decrease both the proton diffusion resistance and the charge transfer resistance [5]. This decrease in resistance makes the electrode less susceptible to a buildup of residual capacity, by allowing a lower oxidation state to be reached before the isolating layer forms at the boundary between the current collector and the active material. The improvement due to cobalt is similar to the difference in performance of standard nickel plates containing active material with and without a few percent of cobalt. Under flooded electrolyte conditions, these plates provide 130% to 140% and from 95% to 100%, respectively. The presence of cobalt at the plaque surface should likewise improve the charge/discharge performance of corroded nickel sinter.

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