HEAT STERILIZABLE Ni-Cd BATTERY DEVELOPMENT

Jet Propulsion Laboratory
Contract No. 951972

Report for Second Quarter
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Prepared by

E. J. Rubin - Project Engineer
P. V. Popat - Project Manager

TEXAS INSTRUMENTS INCORPORATED
Research and Development Laboratories
Attleboro, Massachusetts

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Abstract

Optical microscopic studies reveal that during heat sterilization there is no marked corrosion of the nickel support structure although distinctive color and texture changes do occur at both electrodes.

X-ray diffraction, porosity and pore size distribution data indicate changes occuring in the morphology or crystallography of the plates which may adversely affect electrolyte distribution and/or electrochemical efficiency of the cell.

Several sealed rectangular cells have successfully passed two heat sterilization cycles and have delivered over 70% of their rated capacity. However, the charging mode of the heat sterilized cells need to be optimized to avoid excessive voltage and pressure buildup, particularly during the first few post-sterilization cycles.

Both Pellon polypropylene 14019 and FT 2140 separators will be included in the factorial experiment owing to better uniformity of the former and superior strength of the latter. Other important factors, to be further investigated in the factorial design experiment have been identified.

Satisfactory KOH resistant, thermally stable seal has been developed for heat sterilizable sealed cell studies.

I. INTRODUCTION

This is the second quarterly progress report on the heat-sterilizable nickel-cadmium battery development under Jet Propulsion Laboratory Contract No. 951972, sponsored under NASA Contract NAS-7-100, Task Order No. RD-26. The object of this contract is to perform research and development work leading to the design, development, fabrication and testing of sealed, rechargeable, nickel-cadmium cells capable of heat sterilization.

The heat sterilization requirements include testing at 135°C for type approval, and 125°C testing for flight acceptance. At the 135°C sterilization temperature, the heating rate is 19°C/hour. The chamber is held at the same rate at which it was heated. Two such cycles are required. For preliminary testing one 120-hour cycle may be used.

The specific tasks under this contract are divided into three broad catagories: (1) electrochemistry involving statistical and other experiments for characterizing and optimizing electrodes, electrolyte and separators for heat-sterilizable Ni-Cd cell, (2) case design for hermetically sealed, heat-sterilizable cells, and (3) fabrication and evaluation of rectangular, 4 AH sealed cells before and after heat sterilization.

The details of each specific task requirement are given in the First Progress Report and will not be repeated here. The work performed under each of these tasks is described in the following sections.

Results and Discussion

Task I Electrochemical Investigations

1. Separator Optimization

The evaluation of the three best non-woven polypropylene separator materials selected during the first quarter was continued. These three separators are Pellon Polypropylene brands 14019, and FT 2140 and Kendall brand EM 476. The performance of these separators in 4 AH prismatic sealed cells during charge-discharge cycles before and after sterilization was measured. The electrochemical parameters measured were: delivered capacity, end of charge resistance, end of charge voltage and the potential of the negative plate against the nickel can as the arbitrary reference electrode.

Table I gives the performance data for cells made with 14019 before and after sterilization. Table II gives the performance data for FT 2140 and Table III gives the performance data for EM 476. A comparison of the delivered capacities and end of charge resistance values before and after sterilization show the 14019 brand of polypropylene to be superior. The next best separator is FT 2140. No further evaluation of EM 476 is planned. It will be noted that both the end of charge resistance and end of charge voltage increase after sterilization. The higher end of charge voltage is in small part due to higher resistance (ohmic drop) and mostly due to increased polarization. In order to determine the contribution to total polarization by the positive and negative electrode, the potential of

TABLE I

POLYPROPYLENE SEPARATOR EVALUATION - TYPE 14019
CELL CAPACITY DATA

4 Ah Sealed, Prismatic Cells; Charged at 2 amps for 3 hrs
Discharged at 2 amps to 1.0 V Cut Off

CYCLE	CELL IDENTIFICATION						MEAN	STANDARD DEVIATION
	2S	3K	5F	6F	7S	8 Si		
PRE-STERILIZATION CYCLING DATA CELL CAPACITY, AH, DELIVERED								
1	2.284	4.166	3.200	3.318	3.318	3.234	3.587	0.500
2	4.334	4.218	4.134	4.184	4.152	4.166	4.198	0.070
3	4.188	4.016	4.034	4.084	4.066	4.066	4.064	0.032
4	4.052	3.966	3.952	4.000	3.984	3.984	3.989	0.032
5	4.052	4.000	3.966	4.043	4.000	4.000	4.009	0.032
6	4.000	3.984	4.016	4.118	4.066	4.066	4.042	0.055
7	3.900	3.984	3.966	4.052	4.000	4.000	3.984	0.049
8	3.934	4.000	4.016	4.084	4.052	4.066	4.025	0.058
POST-STERILIZATION CYCLING DATA CELL CAPACITY, AH, DELIVERED								
1	3.566	3.434	Shorted	3.618	3.666	3.534	3.564	0.090
2	3.734	3.384		3.800	3.834	3.600	3.670	0.184
*3	2.918	3.034		3.666	3.684	3.518	3.364	0.364
*5	3.400	2.118		3.500	3.584	3.334	3.187	0.604
PRE-STERILIZATION CYCLING DATA END OF CHARGE RESISTANCE (milliohms)								
1	3.53	7.11	6.04	5.92	3.56	6.28	5.51	1.50
2	3.15	6.52	5.70	5.49	2.89	6.75	4.92	1.51
3	3.47	7.10	6.43	6.09	3.35	6.50	5.82	1.68
4	3.70	7.73	6.19	5.85	3.33	6.33	5.52	1.68
5	3.67	7.48	6.64	6.24	3.57	6.24	5.64	1.63
6	3.58	7.26	5.76	5.51	3.22	5.81	5.19	1.52
7	3.89	8.05	6.17	6.03	3.37	6.32	5.84	1.73
8	4.05	7.40	6.41	6.05	3.58	6.59	5.66	1.53
POST-STERILIZATION CYCLING DATA END OF CHARGE RESISTANCE (milliohms)								
1	5.93	7.96		9.25	5.58	7.95	7.33	1.54
2	8.15	10.73		16.72	5.67	10.47	10.35	4.10
*3	2.49	13.48		18.52	12.32	12.30	11.82	5.82
*5	10.65	15.12		13.17	11.21	11.40	12.31	1.83

* Power shutdown during 4th cycle

TABLE I (continued)

POLYPROPYLENE SEPARATOR EVALUATION - TYPE 14019
END OF CHARGE VOLTAGE DATA

CYCLE	CELL IDENTIFICATION						MEAN	STANDARD DEVIATION
	2S	3K	5F	6F	7S	8 Si		
PRE-STERILIZATION CYCLING DATA CELL VOLTAGE, VOLTS								
1	1.495	1.488	1.449	1.451	1.445	1.444	1.462	0.023
2	1.474	1.468	1.463	1.460	1.445	1.462	1.464	0.007
3	1.445	1.443	1.436	1.437	1.434	1.438	1.439	0.004
4	1.457	1.450	1.447	1.457	1.450	1.452	1.452	0.004
5	1.463	1.455	1.450	1.468	1.456	1.460	1.459	0.006
6	1.470	1.460	1.458	1.480	1.473	1.473	1.469	0.008
7	1.357	1.463	1.452	1.472	1.466	1.468	1.446	0.004
8	1.463	1.461	1.445	1.459	1.456	1.458	1.457	0.007
POST-STERILIZATION CYCLING DATA CELL VOLTAGE, VOLTS								
1	1.663	1.697	Shorted	1.690	1.674	1.695	1.684	0.015
2	1.720	1.474		1.762	1.729	1.745	1.741	0.021
3	1.148	1.761		1.771	1.741	1.748	1.634	0.272
4	1.408	1.776		1.766	1.735	1.757	1.688	0.315
PRE-STERILIZATION CYCLING DATA NEGATIVE PLATE vs Ni CAN REF. VOLTS								
1	0.206	0.897	0.836	0.832	0.892	0.856	0.823	0.285
2	0.241	0.908	0.869	0.862	0.878	0.888	0.820	0.247
3	0.267	0.900	0.872	0.868	0.879	0.881	0.800	0.218
4	0.203	0.909	0.886	0.884	0.872	0.885	0.773	0.255
5	0.181	0.913	0.869	0.886	0.890	0.894	0.772	0.265
6	0.222	0.916	0.871	0.866	0.893	0.903	0.782	0.251
7	0.140	0.917	0.871	0.897	0.887	0.889	0.765	0.280
8	0.098	0.914	0.867	0.868	0.879	0.892	0.753	0.293
POST-STERILIZATION CYCLING DATA NEGATIVE PLATE vs Ni CAN REF. VOLTS								
1	0.935	0.984	Shorted	0.944	0.106	0.987	0.791	0.343
2	0.966	1.062		1.032	0.071	1.048	0.834	0.383
*3	0.719	1.086		1.074	0.183	1.068	0.826	0.350
5	0.790	1.106		1.119	0.085	1.099	0.850	0.397

* Power shutdown during 4th cycle

TABLE I (continued)

POLYPROPYLENE-SEPARATOR EVALUATION - TYPE 14019
SECOND POST-STERILIZATION CYCLING

CYCLE	DELIVERED CELL CAPACITY
	3K

1	Not Tested
2	1.118
3	1.052
4	Immediate Death

END OF CHARGE RESISTANCE (milliohms)

1	-----
2	20.84
3	30.48
4	-----

END OF CHARGE VOLTAGE

1	-----
2	1.838
3	1.846
4	-----

NEGATIVE PLATE vs Ni CAN REF. VOLTS

1	-----
2	0.961
3	0.966
4	-----

TABLE II

POLYPROPYLENE SEPARATOR EVALUATION - TYPE FT-2140
CELL CAPACITY DATA

4 Ah Sealed, Prismatic Cells; Charged at 2 amps for 3 hrs
Discharged at 2 amps to 1.0 V Cut Off

CYCLE	CELL IDENTIFICATION						MEAN	STANDARD DEVIATION
	4 FTS	5 FTK	6 FTK	7 FTF	8 FTF	9 FTSi		
PRE-STERILIZATION CYCLING DATA CELL CAPACITY, AH, DELIVERED								
1	3.752	3.284	3.834	4.084	4.334	4.134	3.858	0.372
2	3.752	4.034	3.900	4.234	4.384	4.000	3.963	0.234
3	3.552	3.734	3.700	4.016	4.066	3.534	3.707	0.292
4	3.484	3.518	3.652	3.800	3.618	3.452	3.532	0.155
5	3.518	3.518	3.766	3.952	3.966	3.566	3.520	0.255
6	3.534	3.552	3.800	4.200	4.084	3.734	3.734	0.287
7	3.487	3.500	3.766	4.200	3.918	3.784	3.711	0.276
8	3.534	3.500	3.766	4.134	3.884	3.734	3.686	0.257
POST-STERILIZATION CYCLING DATA CELL CAPACITY, AH, DELIVERED								
1	3.434	**Removed	3.434	3.318	3.400	3.200	3.357	0.100
2	2.800	For	3.166	3.184	3.434	3.084	3.134	0.229
*3	2.818	Physical	3.084	3.385	3.385	3.284	3.191	0.243
5	2.800	Exam.	3.166	3.534	3.366	3.266	3.226	0.274
PRE-STERILIZATION CYCLING DATA END OF CHARGE RESISTANCE (milliohms)								
1	12.17	7.27	6.69	7.16	6.39	6.49	7.03	2.25
2	10.66	7.05	6.66	6.99	6.04	6.52	6.92	1.61
3	11.77	8.38	7.22	7.45	6.69	7.35	7.86	1.66
4	12.00	9.16	7.59	8.21	7.17	7.91	8.34	1.62
5	12.08	8.54	7.51	8.23	7.05	7.75	8.23	1.64
6	11.42	7.93	7.30	8.10	7.39	7.77	7.95	1.49
7	12.57	9.39	8.48	9.46	8.85	8.16	9.06	1.57
8	11.87	8.68	7.54	9.03	7.28	7.32	8.31	1.61
POST-STERILIZATION CYCLING DATA END OF CHARGE RESISTANCE (milliohms)								
1	39.31		45.39	8.78	7.61	9.57	22.13	18.50
2	62.59		54.11	14.58	10.11	12.36	30.75	25.40
*3	50.11		40.48	11.18	9.26	10.16	24.22	19.40
5	49.95		51.92	14.18	9.15	11.07	27.25	21.70

* Power Shutdown 4th Cycle
** Cells 2 FTD, 3 FTS also removed for examination

TABLE II (continued)

POLYPROPYLENE SEPARATOR EVALUATION - TYPE FT-2140
END OF CHARGE VOLTAGE DATA

CYCLE	CELL IDENTIFICATION						MEAN	STANDARD DEVIATION
	4 FTS	5 FTL	6 FTK	7 FTF	8 FTF	9 FTSi		
PRE-STERILIZATION CYCLING DATA								
1	1.506	1.467	1.513	1.492	1.491	1.495	1.502	0.019
2	1.505	1.470	1.525	1.489	1.489	1.511	1.515	0.045
3	1.487	1.462	1.499	1.483	1.483	1.488	1.508	0.062
4	1.480	1.462	1.516	1.495	1.498	1.497	1.501	0.028
5	1.486	1.462	1.535	1.517	1.503	1.498	1.507	0.028
6	1.507	1.476	1.544	1.525	1.593	1.634	1.544	0.052
7	1.532	1.490	1.597	1.454	1.581	1.521	1.533	0.050
8	1.520	1.484	1.570	1.550	1.532	1.530	1.535	0.029
POST-STERILIZATION CYCLING DATA								
1	1.837	Physical	1.738	1.702	1.630	1.746	1.731	0.075
2	1.919		1.766	1.734	1.453	1.742	1.723	0.165
*3	1.681	Testing	1.516	1.570	1.515	1.515	1.559	0.072
5	1.638	**	1.562	1.689	1.523	1.529	1.588	0.072
PRE-STERILIZATION CYCLING DATA NEGATIVE PLATE vs Ni CAN REF. VOLTS								
1	0.844	0.859	0.913	0.924	0.879	0.841	0.880	0.030
2	0.891	0.885	0.931	0.884	0.913	0.887	0.908	0.033
3	0.895	0.876	0.911	0.881	0.899	0.870	0.902	0.031
4	0.891	0.882	0.912	0.891	0.909	0.875	0.899	0.026
5	0.895	0.872	0.922	0.903	0.909	0.884	0.903	0.036
6	0.906	0.876	0.927	0.892	0.942	0.956	0.922	0.047
7	0.910	0.887	0.951	0.908	0.932	0.896	0.926	0.054
8	0.897	0.881	0.935	0.921	0.899	0.898	0.921	0.057
POST-STERILIZATION CYCLING DATA NEGATIVE PLATE vs Ni CAN REF. VOLTS								
1	1.036	1.177	1.017	0.929	0.897	0.990	1.008	0.090
2	1.127	1.232	0.840	1.187	0.657	1.109	1.024	0.208
*3	1.497	3.235	0.784	1.089	0.850	0.885	1.390	0.858
5	0.911	-----	0.849	1.007	0.861	0.892	0.904	0.056

* Power Shutdown 4th Cycle
** Cells 1 FTD, 2 FTD, 3 FTS also removed for Physical Testing

TABLE II (continued)

POLYPROPYLENE-SEPARATOR EVALUATION - TYPE FT-2140
SECOND POST-STERILIZATION CYCLING

CYCLE	DELIVERED CELL CAPACITY			
	6 FTK	7 FTF	8 FTF	9 FTSi
*1	0.966	2.266	3.434	3.184
2	1.884	Out-No Cap	2.952	2.800
3	1.384	"	2.866	2.700
4	1.300	"	2.918	2.766
	END OF CHARGE RESISTANCE (milliohms)			
1	41.02	19.94	11.06	15.72
2	47.49	---	10.02	13.20
3	56.00	---	10.08	13.00
4	28.86	---	10.91	14.16
	END OF CHARGE VOLTAGE			
1	1.490	1.582	1.599	1.628
2	1.744	---	1.541	1.566
3	1.730	---	1.537	1.550
4	1.732	---	1.550	1.559
	NEGATIVE PLATE vs Ni CAN REF. VOLTS			
1	1.037	0.937	0.939	0.940
2	1.079	---	0.906	0.908
3	1.052	---	0.909	0.900
4	1.070	---	0.921	0.907

* Cycle #1 - 0.500 amps for 20 hrs
Remaining Cycles - Charge @ 2 amps for 3 hrs

TABLE III

POLYPROPYLENE SEPARATOR EVALUATION - TYPE EM-476

4 Ah Sealed, Prismatic Cells; Charged at 2 amps for 3 hrs
Discharged at 2 amps to 1.0 V Cut Off

CYCLE	CELL IDENTIFICATION					MEAN	STANDARD DEVIATION
	1 EMS	2 EMD	3 EMSi	4 EMK	6 EMF		
PRE-STERILIZATION CYCLING DATA CELL CAPACITY, AH, DELIVERED							
1	3.234	3.918	3.418	3.652	3.084	3.475	0.331
2	3.100	3.966	3.884	3.684	3.234	3.574	0.388
3	2.984	3.800	3.652	3.434	3.400	3.454	0.310
4	2.866	3.343	3.206	3.366	3.266	3.228	0.220
5	2.952	3.734	3.400	3.852	3.218	3.431	0.371
6	2.918	2.984	3.584	3.684	3.118	3.258	0.352
7	2.866	3.266	3.566	3.618	2.866	3.236	0.363
8	2.884	3.566	3.552	3.500	2.900	3.280	0.355
POST-STERILIZATION CYCLING DATA CELL CAPACITY, AH, DELIVERED							
1	3.200	Removed	3.284	Removed	Removed		
2	3.153	For	2.566	For	For		
*3	3.018	Physical	2.266	Physical	Physical		
5	2.818	Exam.	2.934	Exam.	Exam.		
PRE-STERILIZATION CYCLING DATA END OF CHARGE RESISTANCE (milliohms)							
1	8.80	7.22	11.40	10.93	10.72	9.81	1.76
2	10.29	8.90	14.13	11.67	10.19	11.03	1.99
3	9.37	8.72	13.73	11.87	10.62	10.86	2.00
4	9.31	10.77	14.15	12.07	10.74	11.45	1.81
5	8.45	10.14	14.43	11.93	11.23	11.23	2.22
6	8.70	18.03	15.52	10.88	10.43	12.71	3.90
7	9.34	13.38	15.72	12.49	14.13	13.01	2.38
8	9.20	11.96	14.48	11.73	12.67	12.01	1.55
POST-STERILIZATION CYCLING DATA END OF CHARGE RESISTANCE (milliohms)							
1	10.42	13.91					
2	13.67	22.70					
*3	13.98	25.31					
5	14.51	24.89					

* Power Shutdown 4th Cycle

TABLE III (continued)

POLYPROPYLENE SEPARATOR EVALUATION - TYPE EM-476
END OF CHARGE VOLTAGE DATA

CYCLE	CELL IDENTIFICATION					MEAN	STANDARD DEVIATION
	1 EMS	2 EMD	3 EMSi	4 EMK	6 EMF		
PRE-STERILIZATION CYCLING DATA							
1	1.522	1.542	1.493	1.519	1.495	1.514	0.020
2	1.527	1.643	1.509	1.518	1.496	1.546	0.063
3	1.518	1.687	1.504	1.503	1.568	1.554	0.074
4	1.521	1.586	1.519	1.509	1.666	1.560	0.075
5	1.524	1.665	1.525	1.507	1.575	1.559	0.064
6	1.456	1.515	1.543	1.509	1.537	1.512	0.035
7	1.500	1.596	1.555	1.545	1.532	1.546	0.035
8	1.558	1.688	1.559	1.534	1.535	1.575	0.064
POST-STERILIZATION CYCLING DATA							
1	1.711	Removed	1.708		Removed		
2	1.765	For	1.759	Shorted	For		
3	1.684	Physical	1.580		Physical		
*		Testing			Testing		
5	1.589		1.569				
PRE-STERILIZATION CYCLING DATA NEGATIVE PLATE vs Ni CAN REF. VOLTS							
1	.873	.881	.883	1.455	1.506	1.120	0.296
2	.899	1.008	.893	.922	.226	.798	0.267
3	.892	1.042	.884	1.449	1.054	1.064	0.205
4	.890	.903	.889	1.441	1.085	1.042	0.213
5	.895	1.024	.897	1.439	1.653	1.182	0.309
6	.472	.870	.914	1.459	1.638	1.072	0.423
7	.906	.915	.923	1.480	.941	1.033	0.224
8	.911	1.038	.925	1.472	.963	1.062	0.210
POST-STERILIZATION CYCLING DATA NEGATIVE PLATE vs Ni CAN REF. VOLTS							
1	.170	Removed	.958		Removed		
2	1.056	For	1.038	Shorted	For		
*3	.972	Physical	.893		Physical		
5	.396	Testing	.900		Testing		

* Power Shutdown 4th Cycle

TABLE III (continued)

POLYPROPYLENE-SEPARATOR EVALUATION - TYPE EM-476
SECOND POST-STERILIZATION CYCLING

CYCLE	DELIVERED CELL CAPACITY	
	1 EMS	3 EMSi
*1	2.166	2.066
2	1.966	1.684
3	1.834	1.600
4	1.734	1.700

END OF CHARGE RESISTANCE (milliohms)

1	34.53	30.76
2	28.76	28.53
3	29.86	29.67
4	35.10	34.37

END OF CHARGE VOLTAGE

1	1.565	1.547
2	1.597	1.610
3	1.600	1.608
4	1.611	1.619

NEGATIVE PLATE vs Ni CAN REF. VOLTS

1	0.855	0.913
2	0.895	0.819
3	0.904	0.685
4	0.913	0.635

* Cycle #1 - .500A for 20 Hrs.
Remaining Cycles - Charge @ 2amps for 3 Hrs.

the negative versus the can as arbitrary reference potential was measured. It appears that it is the cadmium (negative) plate that makes the greater contribution to the higher polarization after sterilization. This may be partly due to the fact that during sterilization the excess free cadmium metal (charge adjustment to make the cell positive limited during discharge and to increase oxygen recombination during overcharge) is oxidized and this increases its impedance. This is discussed further in the section on microscopic studies.

Some of these cells, after post-sterilization cycling, were subjected to a second heat sterilization cycle. Cell characterization following this second sterilization cycle indicated a greater failure frequency for the cells containing 14019 separator compared to cells containing FT 2140 separator. These data are also included in Tables I II and III respectively. Further work concerning the failure mode of cells after second sterilization will be necessary before making a final decision on the separator selection. Preliminary indications are that the shorting mode of failure, after second sterilization, is much lower with cells containing FT 2140. This is consistent with better physical and mechanical properties of FT 2140. However, failure could also occur by the loss of electrolyte due to any possible electrolysis of water during charge. This loss of electrolyte can increase the cell resistance during cycling and reduce capacity. It is interesting to note that the cells containing FT 2140 after second sterilization did not show as much capacity loss as did the cells

with 14019 separator. A thorough analysis of changes in the separator during and after sterilization is under way.

2. Optical Microscopy of the Plates

The purpose of this phase of the investigation was to determine what visible changes occurred in the plates as a result of heat sterilization. Since nickel metal is known to corrode in alkaline media according to the reaction:

$$Ni + 2OH^- \longrightarrow Ni(OH)_2 + 2e^-$$

both the nickel mesh and sintered nickel are subject to attack. In fact during the manufacturing process of the positive plate a noticeable proportion of the nickel sinter is attacked. This reaction is accelerated by temperature and thus the heat sterilization process (with the positive and negative electrodes short circuited) may cause a further attack of the substrate. The consequence of the corrosion of the nickel support structure would be a loss of strength of the plate and loss of some electronic conductivity and contact resulting in higher resistance and lower utilization of active material (loss in capacity).

To determine these possible effects, both the positive and negative plates were examined in various stages:

1. as recieved from the manufacturing process
2. after cycling in a sealed cell
3. after heat sterilization

4. after heat sterilization and cycling in a sealed cell

In Figures 1 through 4 the full cross sectionsof the positive and negative plates are shown. These views include:

1. the longitudinal and cross sectional areas of the woven nickel mesh support

2. the sintered nickel substrate (white areas)

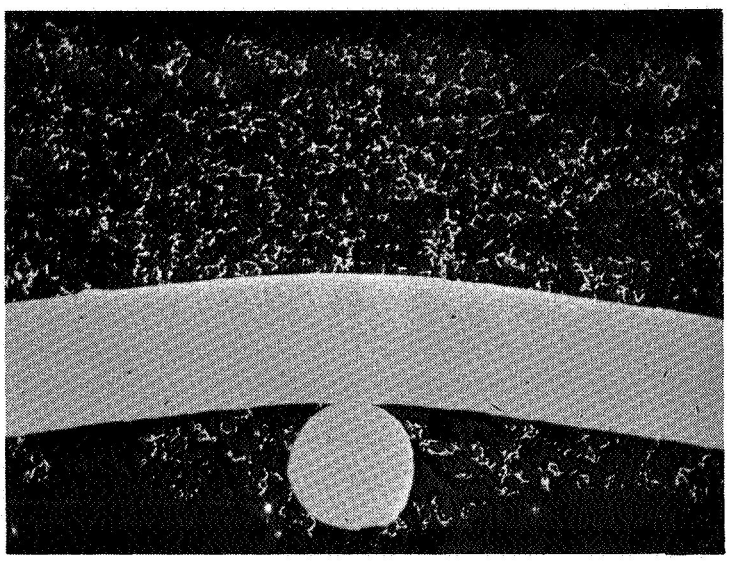
3. the porous active material (gray and black areas)

There are visible changes shown in these figures notably in the positive electrode. The texture and coloring of the positive active material undergoes several transformations. After sterilization the positive (nickel oxide) plate has a blue-gray coloration (appears gray in the photo-micrographs) as opposed to the normal black color. The negative (cadmium) electrode also undergoes a change in coloration from a dark to a light gray. These color, shade and texture changes are more readily visible in Figures 5 through 8 which were taken at a higher magnification. These figures (1 through 8) show no appreciable loss in the nickel sinter structure through corrosion.

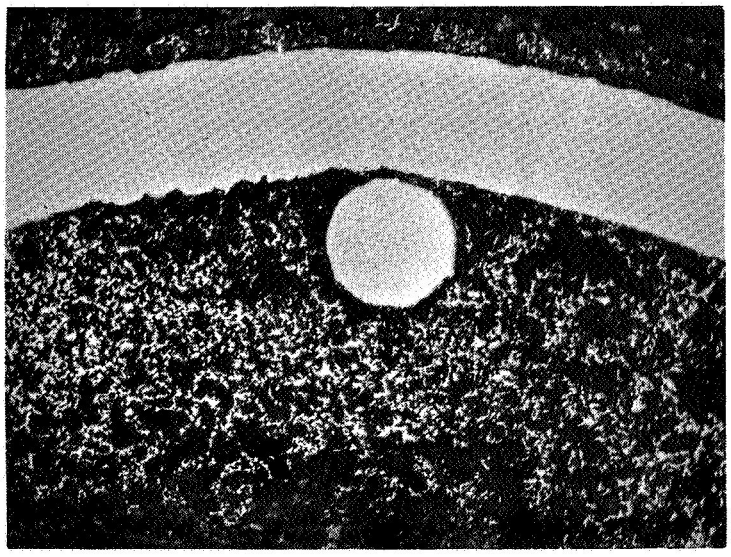
Since corrosion quite often occurs at grain boundaries, these samples were etched to show the microstructure of the nickel wire grid. Photographs of these samples are shown in Figures 9 through 12. There is some evidence of grain boundary attack of the wire as shown in Figure 9. This slight intergranular corrosion occurred in the manufacturing process but there is no further corrosion of the grid both before and after the heat sterilization cycle.

FIGURE 1

AS MANUFACTURED



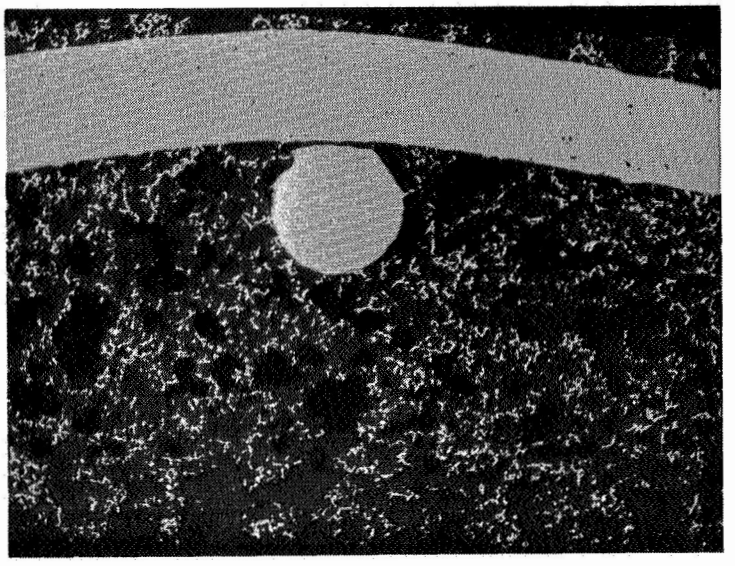
POS 68 MAG. 100X
AS POLISHED



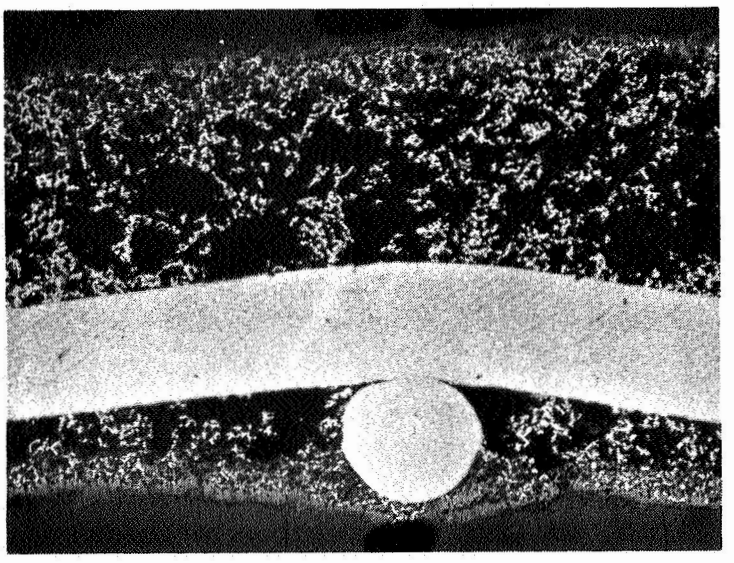
NEG 85 MAG. 100X
AS POLISHED

FIGURE 2

CYCLED PRE-STERILIZED



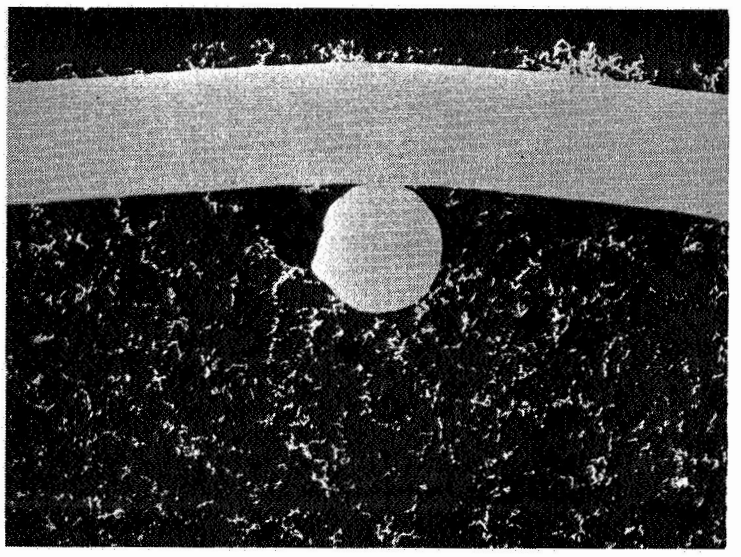
POS 68 100X MAG.
AS POLISHED



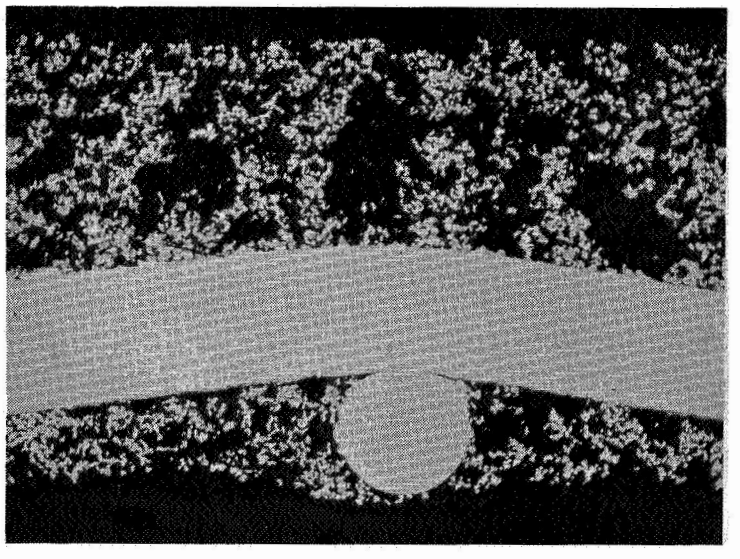
NEG 85 100X MAG.
AS POLISHED

FIGURE 3

STERILIZED



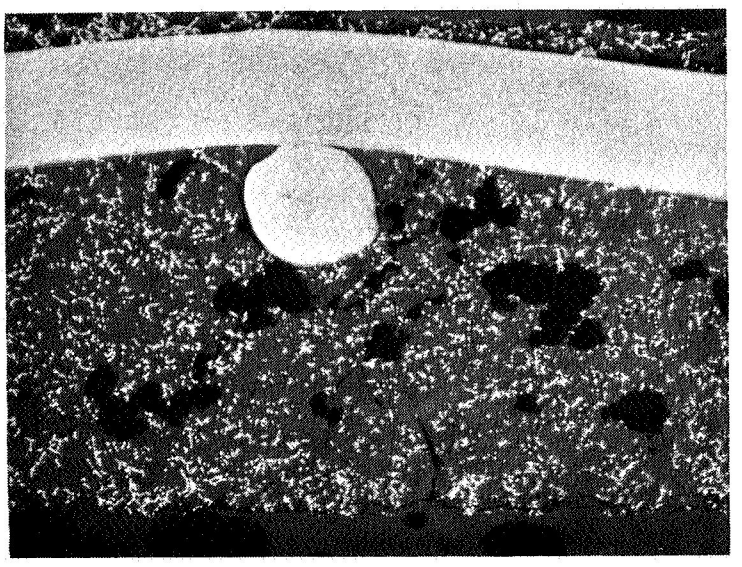
POS 68 100X MAG.
AS POLISHED



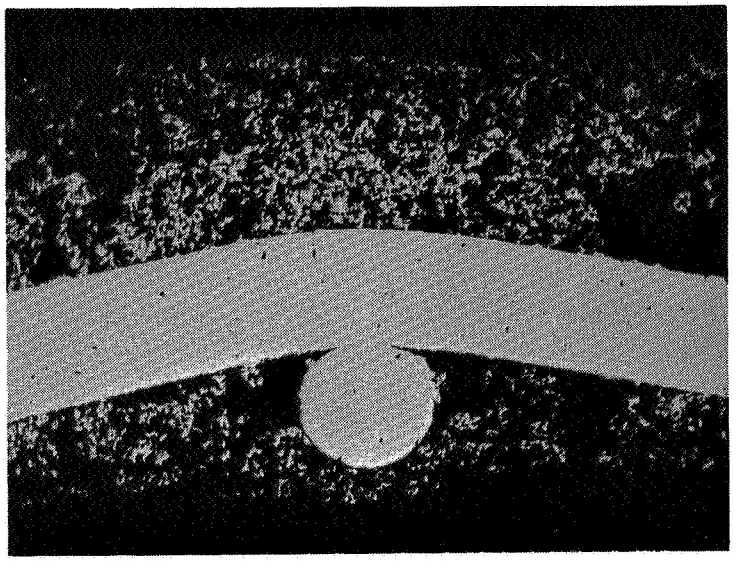
NEG 85 100X MAG.
AS POLISHED

FIGURE 4

STERILIZED & CYCLED



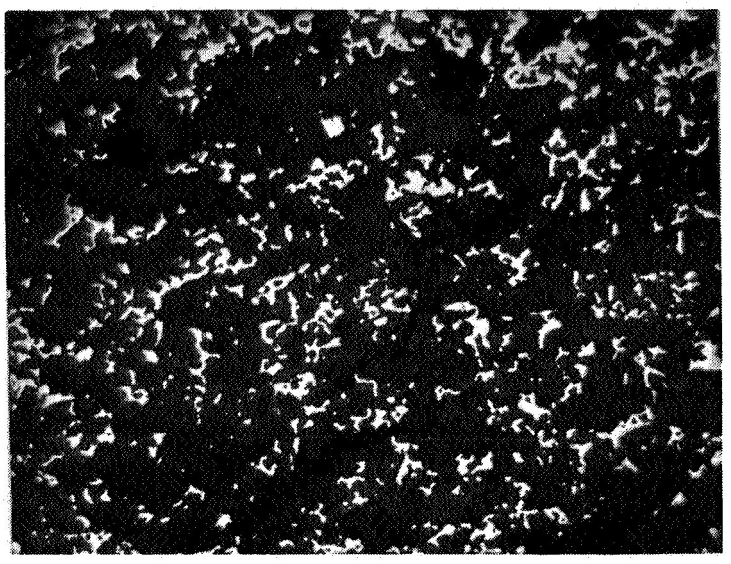
POS 68 500X MAG.
AS POLISHED



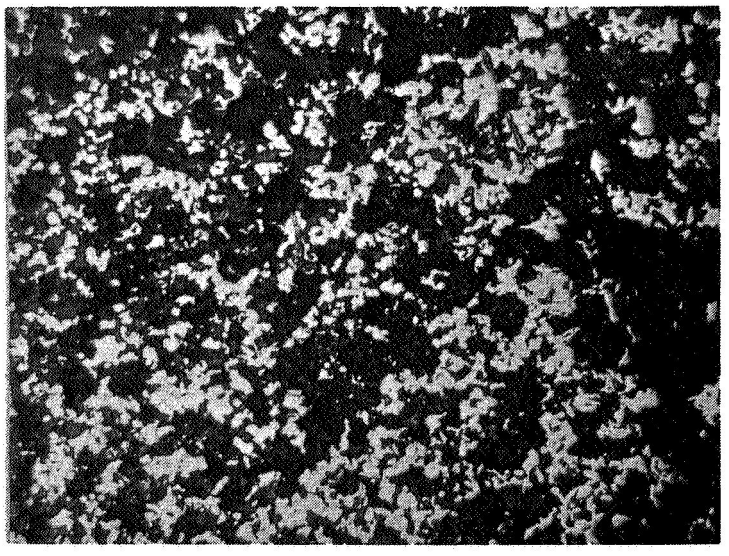
NEG 85 500 X MAG.
AS POLISHED

FIGURE 5

AS MANUFACTURED



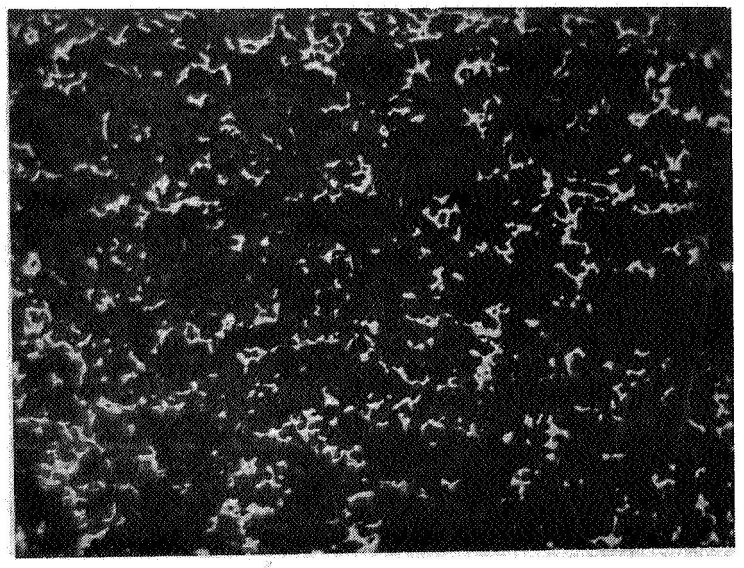
AS POLISHED
POS 68 MAG. 250X



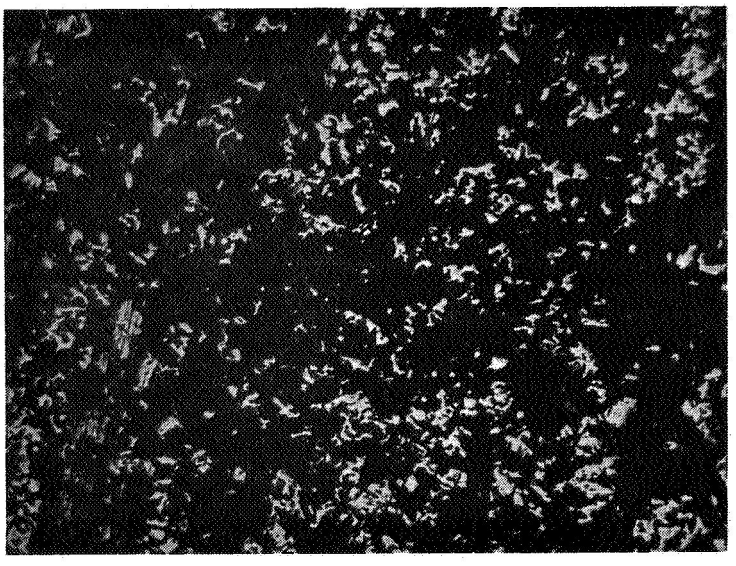
AS POLISHED
NEG 85 MAG. 250X

FIGURE 6

CYCLED PRE-STERILIZED



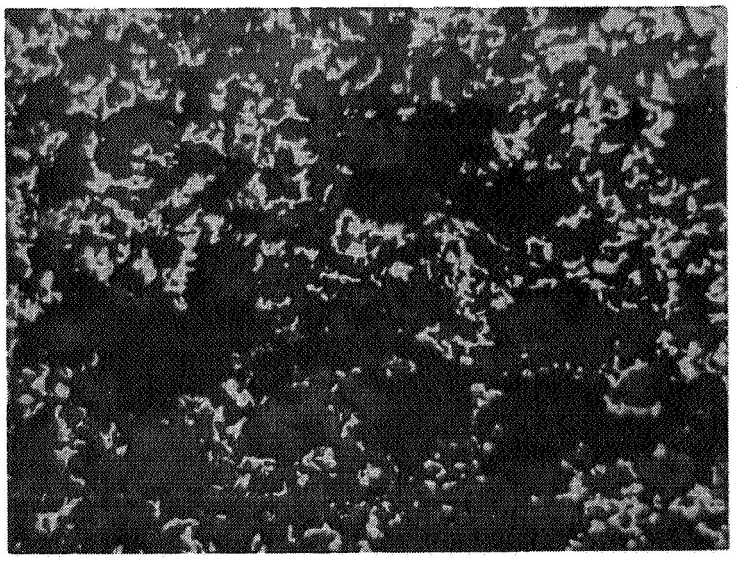
POS 68 250X MAG.
AS POLISHED



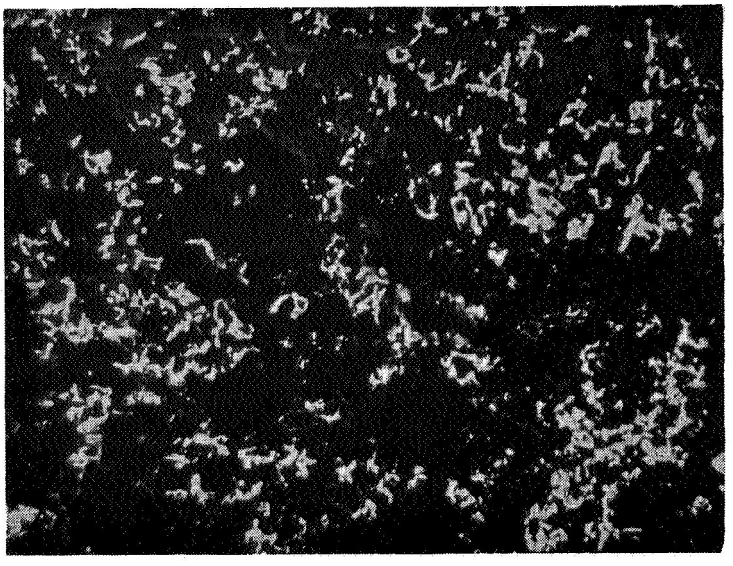
NEG 85 250X MAG.
AS POLISHED

FIGURE 7

STERILIZED



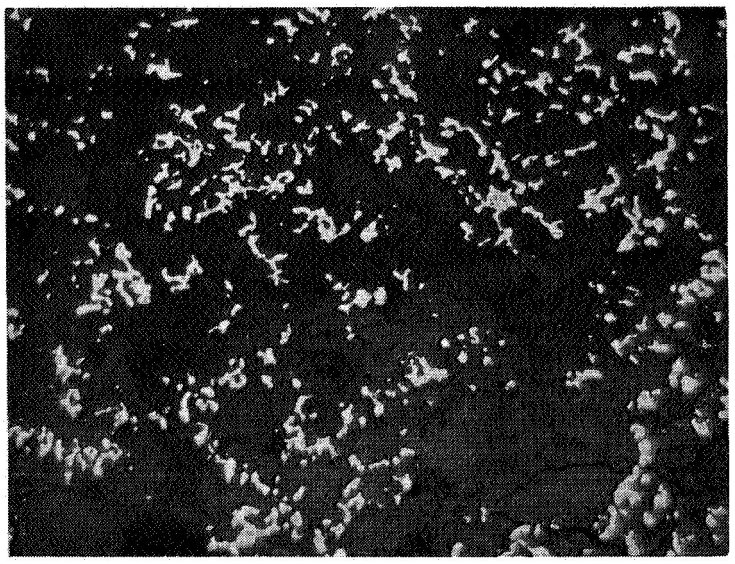
POS 68 250X MAG.
AS POLISHED



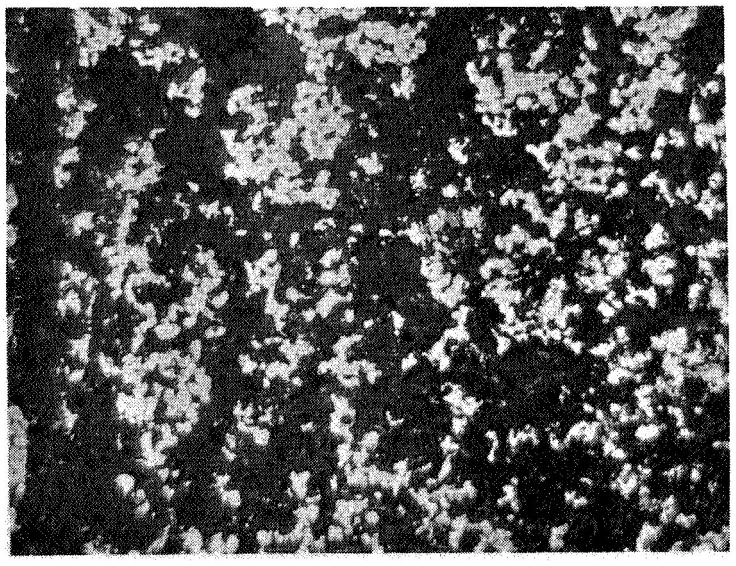
NEG 85 250X MAG.
AS POLISHED

FIGURE 8

STERILIZED & CYCLED



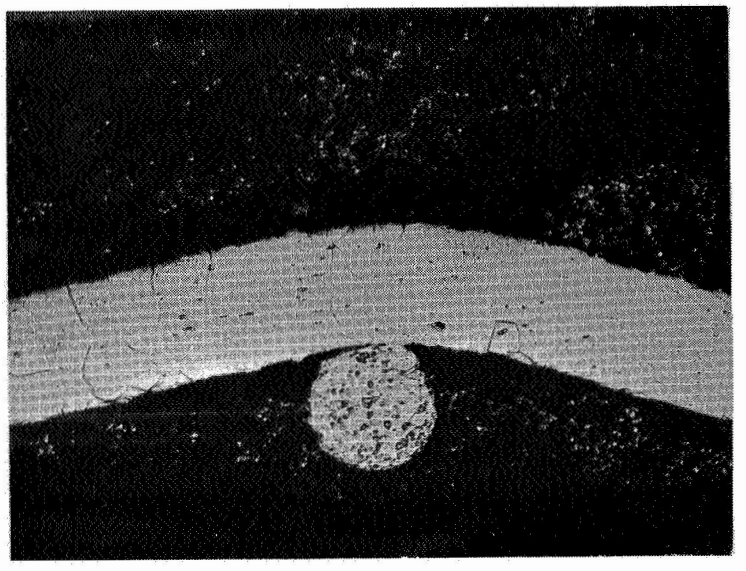
POS 68 250X MAG.
AS POLISHED



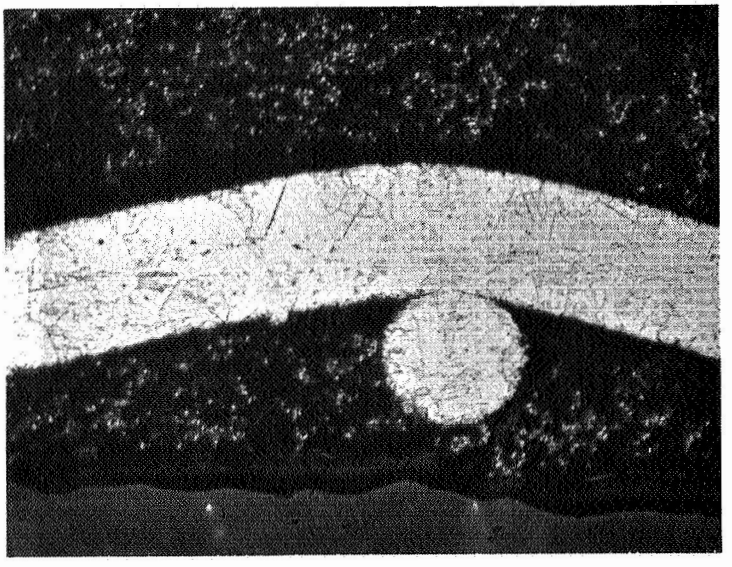
NEG 85 250X MAG.
AS POLISHED

FIGURE 9

AS MANUFACTURED



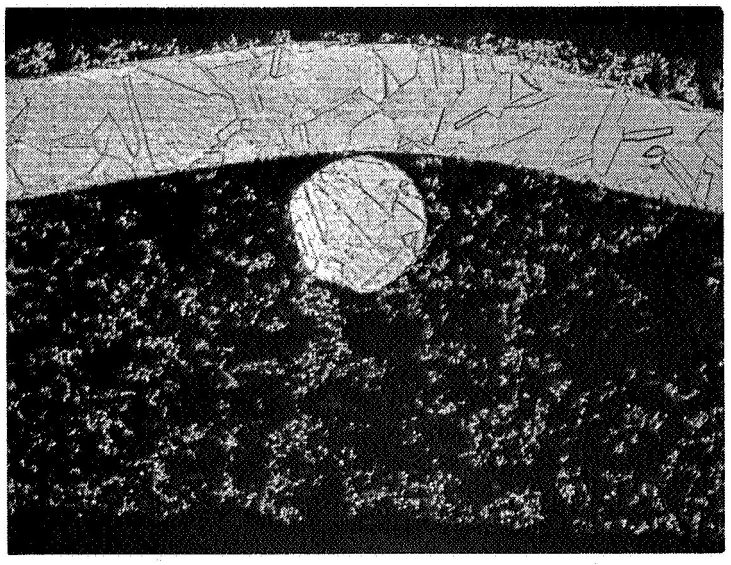
POS 68 MAG. 100X
ETCHED



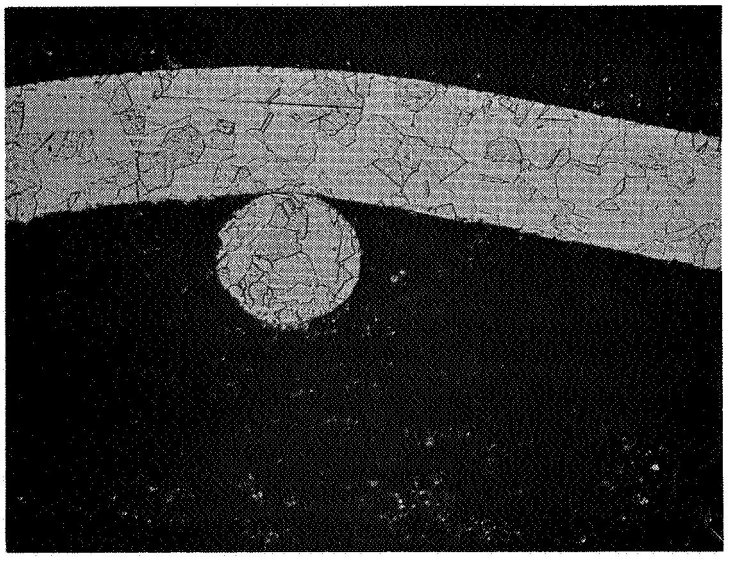
NEG 85 MAG. 100X
ETCHED

FIGURE 10

CYCLED PRE-STERILIZED



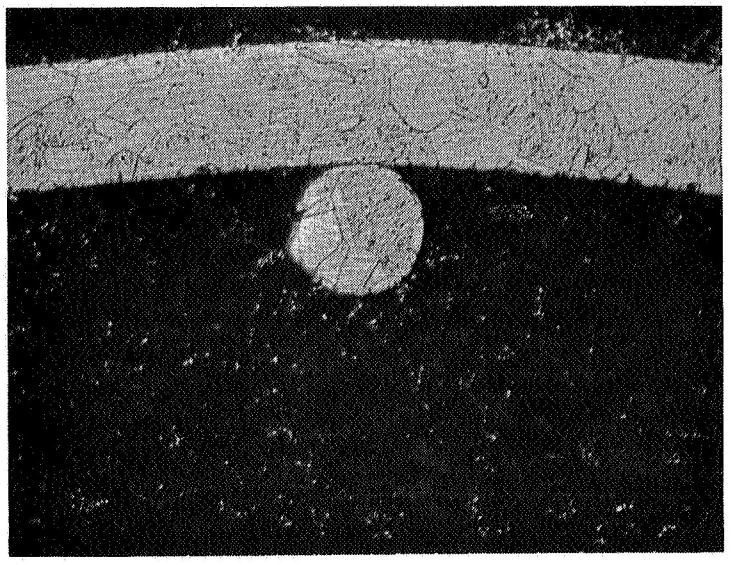
POS 68 100X MAG.
ETCHED



NEG 85 100X MAG.
ETCHED

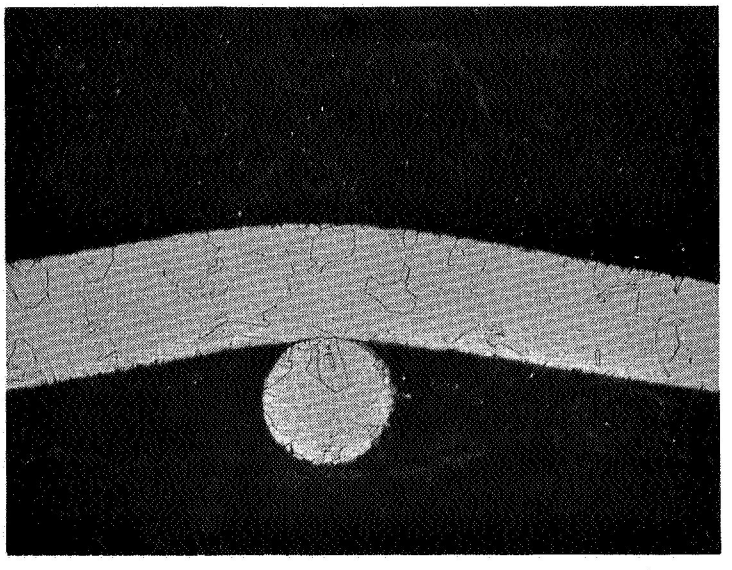
FIGURE 11

STERILIZED



POS 68 100X MAG.

ETCHED

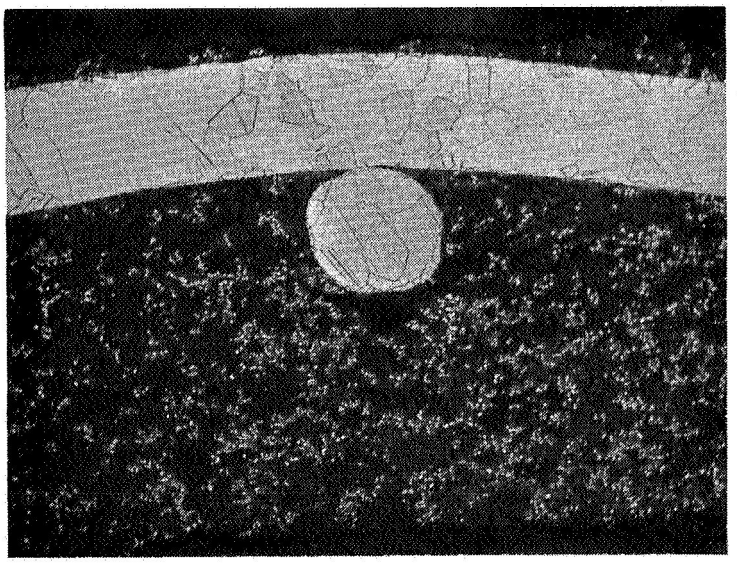


NEG 85 100X MAG.

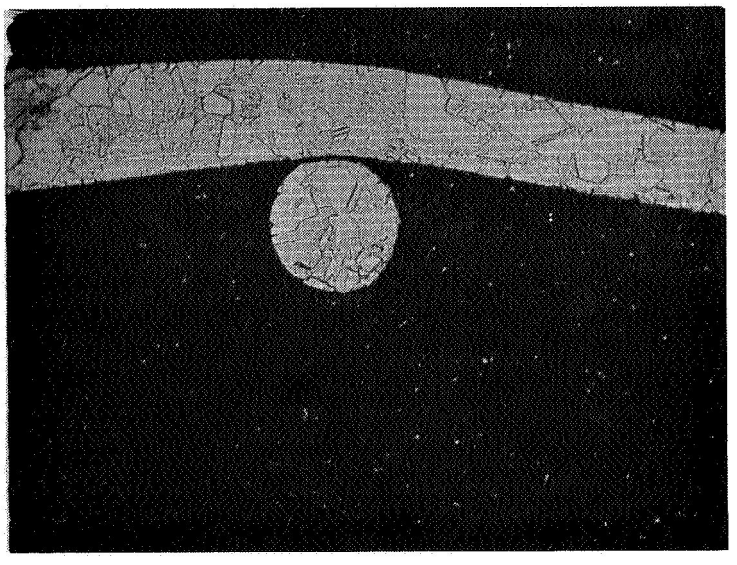
ETCHED

FIGURE 12

STERILIZED & CYCLED



POS 68 100X MAG.
ETCHED



NEG 85 100X MAG.
ETCHED

Thus optical microscopic studies have shown that there is no appreciable attack on the nickel grid or nickel sinter substrate as a result of the heat sterilization process. Also, the color, shade and texture changes which occur in both the positive and negative electrodes are visible.

These changes are believed to be morphological in nature and will be correlated with the other physical and chemical data obtained.

3. Porosity and Pore Size Distribution

The wetting (capillarity) and electrolyte retention properties of porous materials (e.g. battery plates) are affected by: (1) the wetability of the surface (inherent property of the material); (2) the total porosity; and (3) the pore sizes (distribution).

Both porosity and pore size distribution of positive and negative plates were determined at the several stages previously mentioned. An Aminco-Winslow mercury porosimeter, Model No. 5-7118 was used for these determinations. A summary of the porosity data is shown in Table IV.

TABLE IV

Porosity of Dry Positive and Negative Plates

	Condition of Plate When Evaluated			
	As Manufactured	Cycled Pre-Sterilized	Sterilized	Cycled Post-Sterilized
Positive	33.4 %	26.9 %	45.2 %	39.1 %
Negative	55.9 %	57.8 %	55.5 %	58.6 %

These data indicate there are measurable changes in the porosity of the positive plate, whereas the porosity of the negative plate is essentially unchanged, under the conditions studied. In addition, heat sterilization causes a marked and apparently irreversible change in the pore size distribution of the positive plate. These data are shown in Figures 13 and 14. After sterilization, the majority of the pores are one micron or less. Continued cycling after sterilization does not restore the original (pre-sterilization) pore diameter distribution of the positive plate.

Independent studies conducted in these laboratories indicate that in battery plates pores with very small diameters do not effectively participate in electrochemical reactions. Thus a shift to a larger number of very small pores, may cause a loss in useable capacity of a plate. The change in pore diameter distribution also affects the capillarity of the positive plate, which can alter the electrolyte retention of the plate and cause a shift in electrolyte distribution throughout the cell.

One may therefore hypothesize a failure mode for heat sterilized cells according to the following scheme.

(1) the average pore size of the positive plate decreases during sterilization and may hold less electrolyte even though porosity may increase somewhat.

(2) the capacity of the positive plate decreases due to electrolyte limitation. The cadmium plate absorbs some of the electrolyte rejected by the positive plate in the sealed cell.

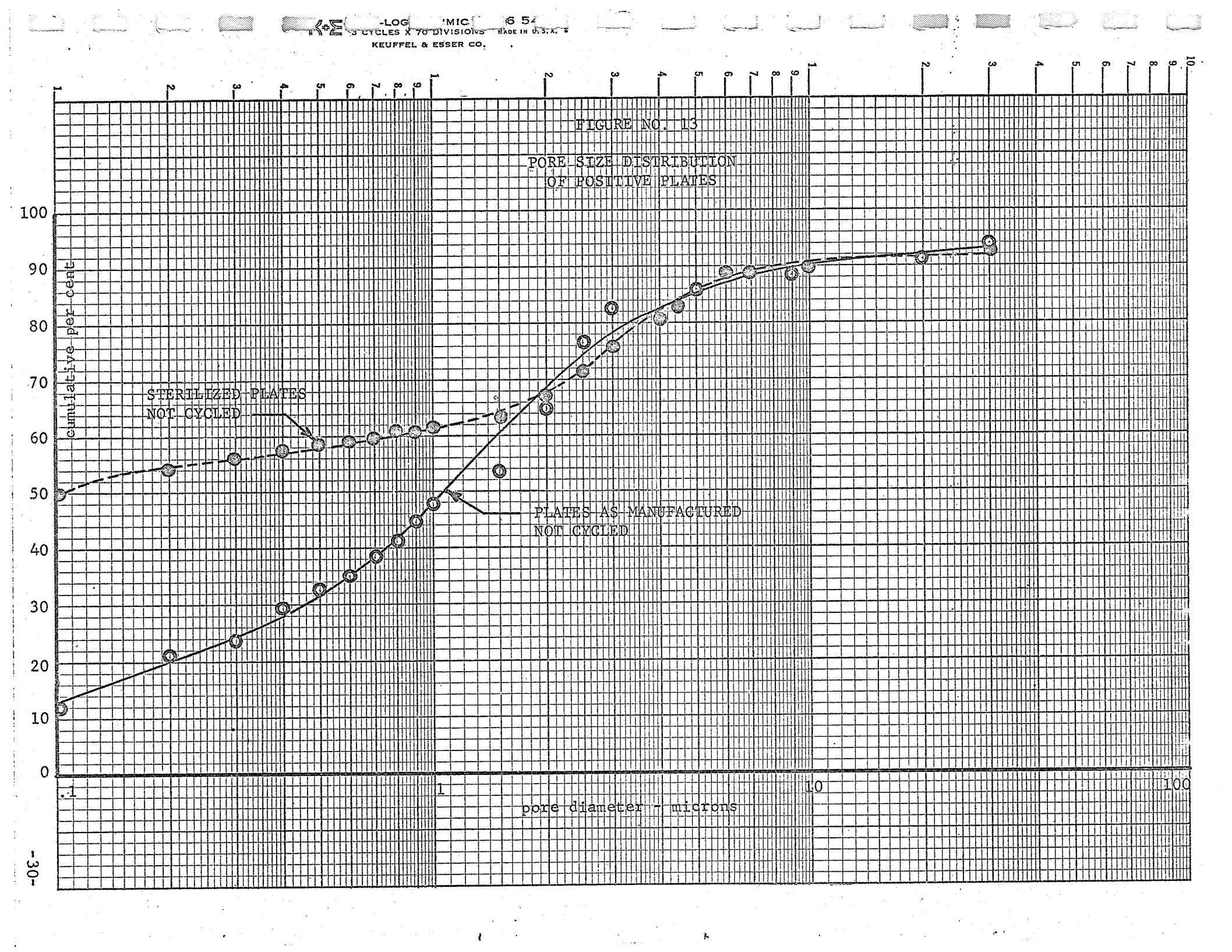
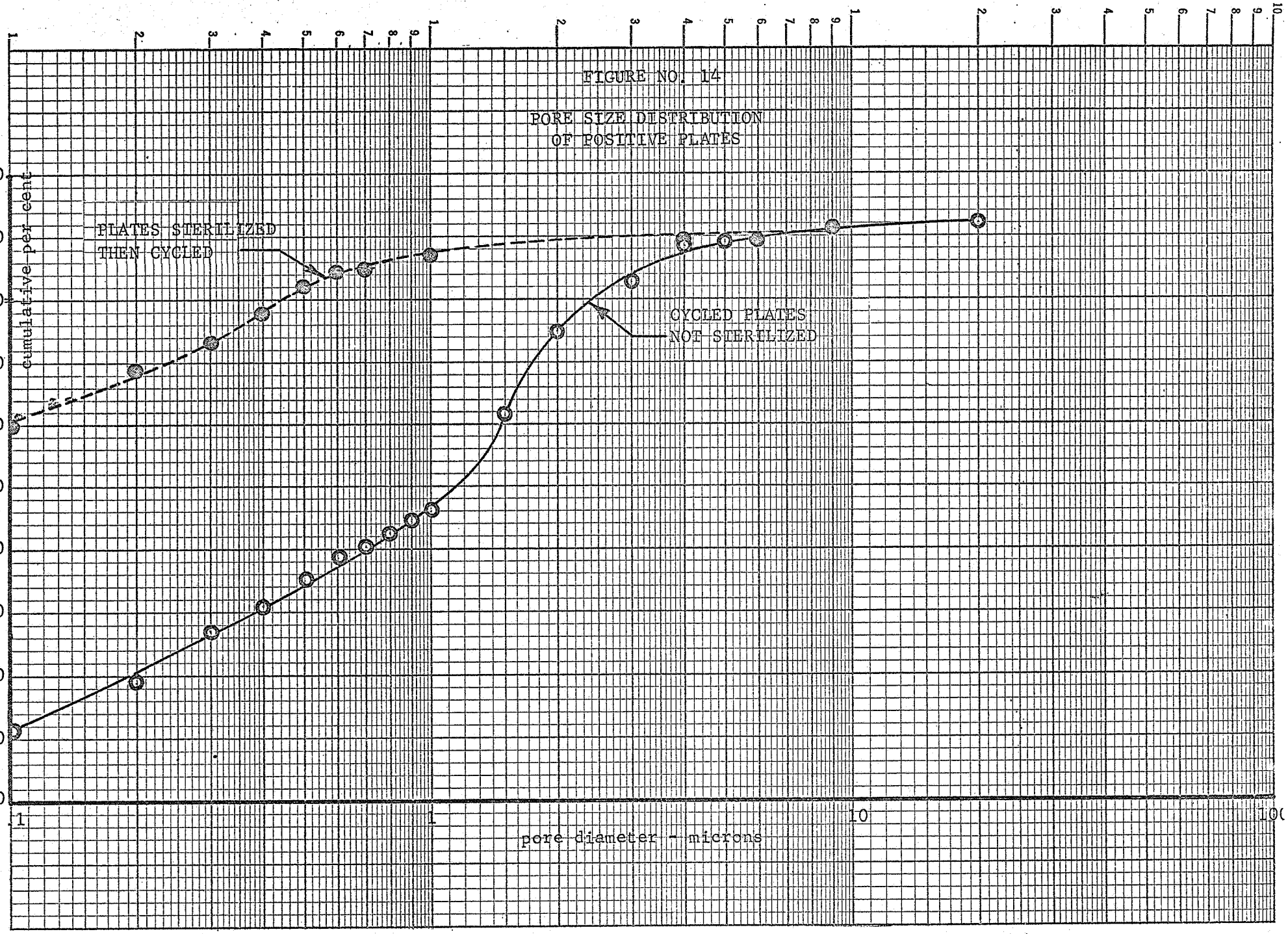

KEUFFEL & ESSER CO.
FIGURE NO. 13
PORE SIZE DISTRIBUTION
OF POSITIVE PLATES
STERILIZED PLATES
NOT CYCLED
PLATES AS MANUFACTURED
NOT CYCLED
cumulative per cent
pore diameter - microns
100
90
80
70
60
50
40
30
20
10
0
.1
1
10
100

FIGURE NO. 14

PORE SIZE DISTRIBUTION
OF POSITIVE PLATES



(3) since positive capacity falls, then for a given coulombic charge input (e.g. 5 ampere-hours) a greater proportion of oxygen gas is evolved.

(4) Since the cadmium plate has absorbed additional electrolyte its oxygen recombination rate is lowered, since recombination rate is controlled predominantly by oxygen diffusion through the electrolyte film.

(5) this results in a rapid buildup of oxygen gas during overcharge.

(6) under these conditions the negative (cadmium) electrode can evolve hydrogen gas, particularly if excess cadmium hydroxide is not accessible for reduction during constant current charging.

This hypothesis is valid even if there are no changes in the porosity or pore diameter distribution of the negative plate. The data presented in Figures 15 and 16 indicate certain shifts in the pore size distribution of the negative plate occurs during heat sterilization. These changes are apparently reversible and upon cycling (after sterilization) the pore size distribution apparently returns to its original (pre-sterilization) values.

To verify the above hypothesis, further studies concerning the electrolyte retention by the separators and the plates before and after heat-sterilization are now in progress. In addition, measurement of the oxygen-free and hydrogen-free capacities of plates prior to and subsequent to heat sterilization is planned for the next quarter.

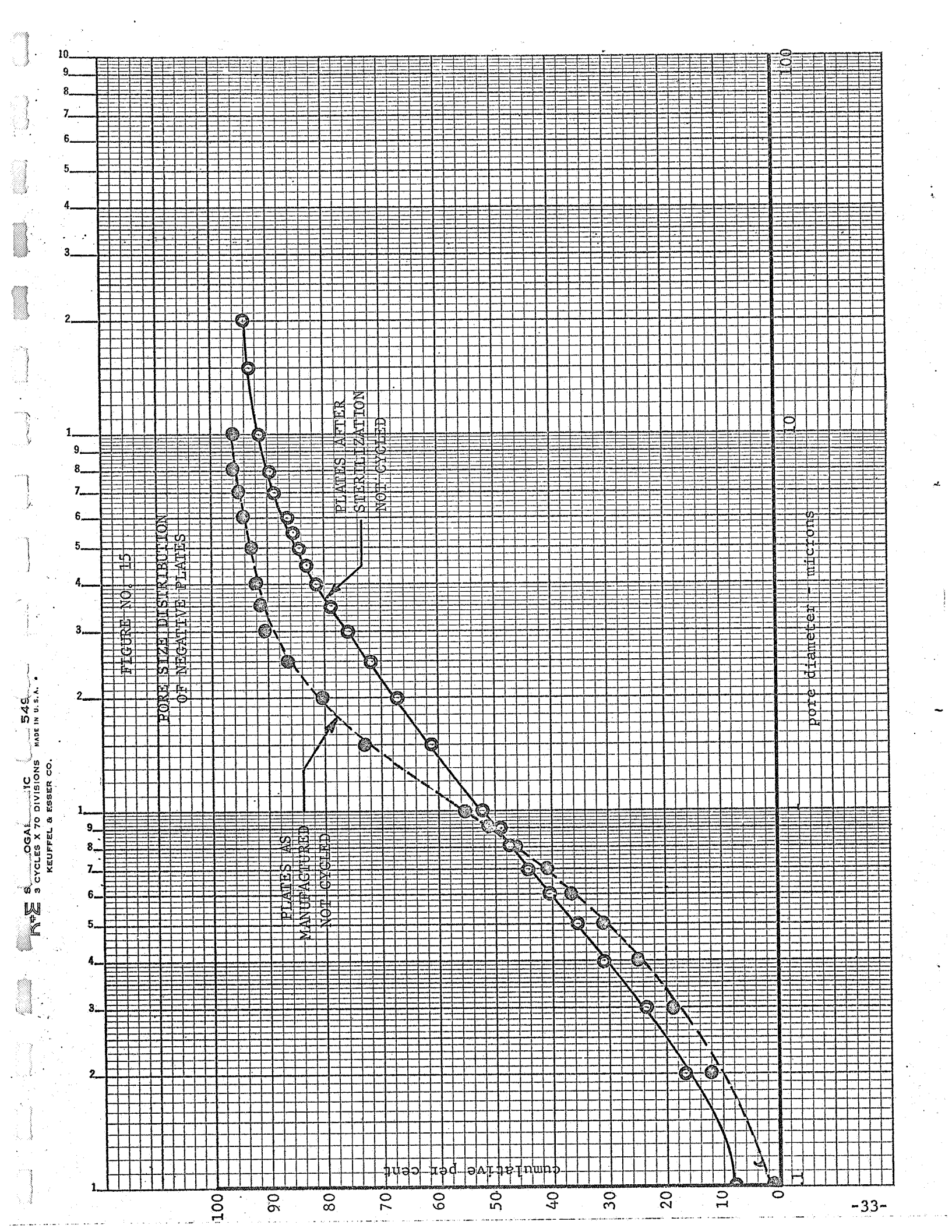
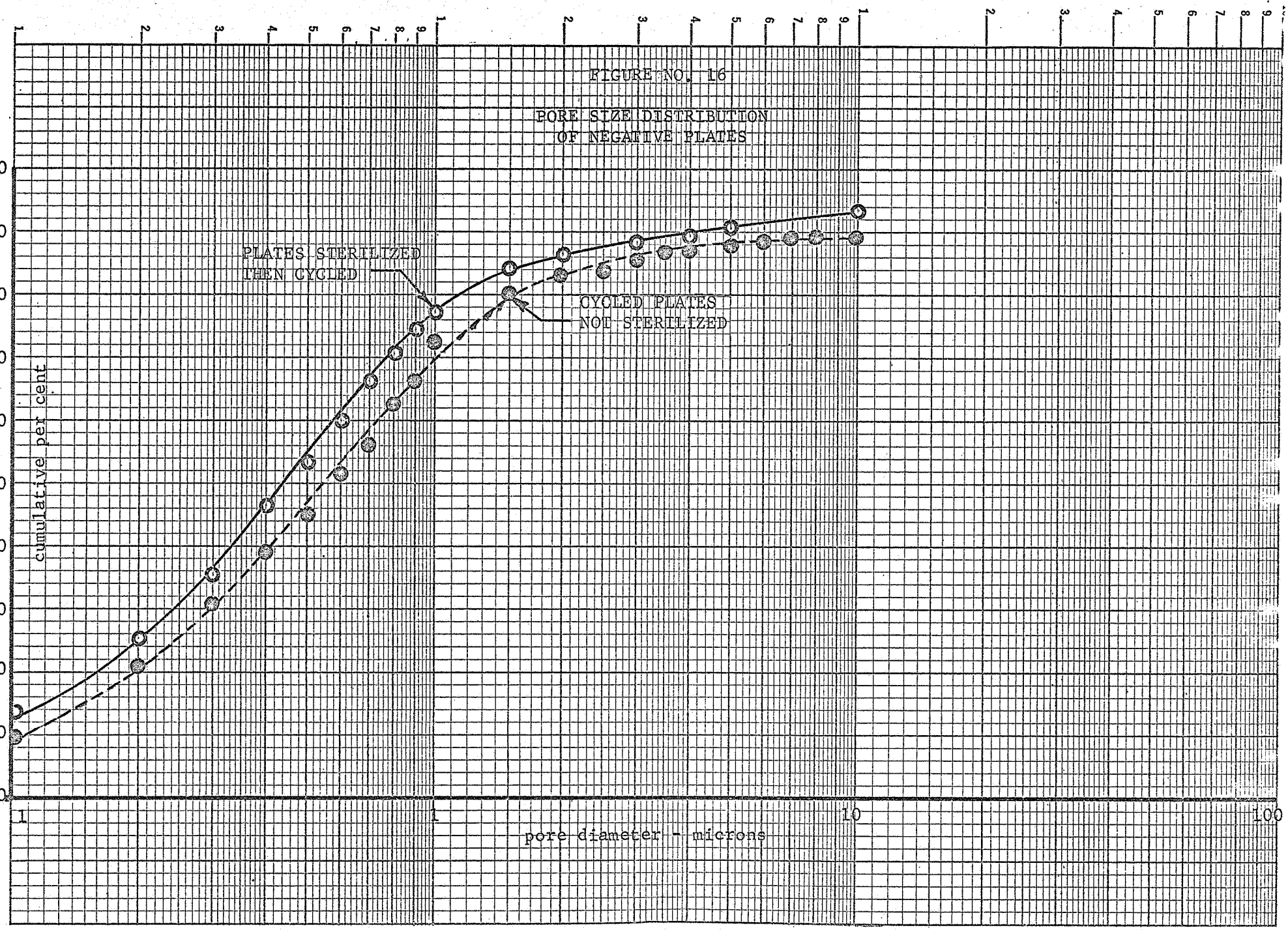


FIGURE NO. 15
PORE SIZE DISTRIBUTION
OF NEGATIVE PLATES
PLATES AFTER
STERILIZATION
NOT CYCLED
PLATES AS
MANUFACTURED
NOT CYCLED
cumulative per cent
pore diameter - microns
100
90
80
70
60
50
40
30
20
10
0
10
1
KEUFFEL & ESSER CO.
MADE IN U.S.A.
3 CYCLES X 70 DIVISIONS
549

FIGURE NO. 16

PORE SIZE DISTRIBUTION
OF NEGATIVE PLATES



4. X-Ray Diffraction

X-ray diffraction spectra were taken of positive and negative electrodes at various stages (of condition) previously mentioned. Spectra were taken using a Norelco diffractometer Model No. 5-210M. The scan rate for these initial runs was 2 degrees per minute using CuKα radiation. The major peaks and relative intensities of these spectra are presented in Table V. These data are being analyzed and compared to values available in the literature.

A preliminary analysis of the data shown in Table V reveals changes in bandwidth, intensity and slight shifting in some reflection peaks indicating that crystallographic or morphological changes occur in both positive and negative plates as a result of the heat sterilization process. However the x-ray spectrum of the positive plate taken after sterilization and subsequent cycling is similar to spectra obtained before heat sterilization. This indicates that during cycling after sterilization the crystallography or morphology of the positive plate returns to its original state (i.e., before heat sterilization). The same effect is not apparent in the negative electrode since spectra obtained after cycling following the sterilization process retain many of the intensity and bandwidth changes characteristic of the post-sterilization spectra. Therefore heat sterilization produces long term crystallographic changes in the negative electrode which may impair its electrochemical efficiency.

Future work includes continued analysis of these and other necessary spectra.

TABLE V

X-RAY DIFFRACTION DATA - NEGATIVE PLATES

Plate Condition											
As Received			Cycled			Sterilized			Sterilized and Cycled		
2θ	I	B.W.	2θ	I	B.W.	2θ	I	B.W.	2θ	I	B.W.
18.85	▷100	---	18.80	15.9	1.5	18.80	▷100	---	18.75	▷100	---
29.45	85.0	1.5	29.50	10.8	1.3	29.45	▷100		29.45	▷100	---
31.90	8.4	1.4	31.80	34.8	1.8	-----	----	---	31.75	8.0	1.7
-----	----	---	-----	----	---	33.00	5.0	2.0	-----	----	---
35.30	▷100	---	*34.70 35.20	31.2 19.8	1.6 2.3	35.20	▷100	---	35.20	▷100	
38.40	39.0	1.8	38.35	▷100	---	38.20	19.3	1.9	38.30	29.8	1.7
44.50	2.0	2.0	44.50	2.0	2.0	44.40	8.6	1.3	44.50	6.0	1.5
47.90	6.0	1.8	47.80	18.6	2.0	-----	----	---	47.80	6.2	1.7
49.10	53.8	1.9	49.00	6.0	1.8	49.00	82.0	1.7	49.00	66.0	1.7
52.40	29.3	2.0	52.40	3.8	2.2	52.30	48.6	1.5	51.25	36.0	1.8
56.20	25.4	2.0	56.15	5.0	2.0	56.10	53.0	1.5	56.10	35.0	1.8
59.0	5.0	1.8	-----	----	---	58.80	6.4	2.0	58.70	7.4	1.4
61.20	13.0	2.3	61.10	19.5	2.3	61.20	14.2	1.7	61.15	16.8	1.6
62.40	6.4	4.7	62.35	19.0	2.0	-----	----	---	62.35	7.8	2.0
64.75	16.5	2.0	64.70	3.0	1.4	64.60	29.4	2.0	64.50 64.70	22.5 64.7	2.0
66.70	8.5	2.5	66.60	3.0	2.0	66.60 66.80	17.0 15.0	1.4 1.6	66.60	11.0	2.0
77.20	12.8	2.6	-----	----	---	67.15	24.0	1.9	67.20	19.3	1.8
71.80	5.0	2.0	71.70	18.4	2.4	-----	----	---	71.70	6.8	2.8
74.60	10.4	2.5	75.70	12.0	2.4	74.40	20.5	2.0	74.50	14.3	2.0
82.60	6.5	2.8	82.40	4.0	2.5	82.60	8.2	2.8	82.40 82.80	8.50 6.20	2.0 2.0
84.65	5.0	2.6	84.50	2.0	2.0	84.60 84.80	11.3 7.5	1.8 2.0	84.60	7.5	3.0
87.70	10.6	2.7	87.70	2.8	3.0	87.60 87.90	21.8 14.8	1.4 1.0	87.60 87.90	13.0 12.2	2.8
90.00	5.0	3.0	-----	----	---	89.20 90.00	7.40 9.00	3.5 3.0	89.20 89.90	6.50 8.10	2.5 3.0

2θ = Reflection angle, I = Relative intensity, B.W. = Band width at 1/2 I

* $\frac{34.7}{35.2}$ for example indicates doublet

TABLE V (continued)

Plate Condition											
As Received			Cycled			Sterilized			Sterilized and Cycled		
2θ	I	B.W.	2θ	I	B.W.	2θ	I	B.W.	2θ	I	B.W.
X-RAY DIFFRACTION DATA - NEGATIVE PLATES											
93.00	3.4	2.7	93.00	6.0	3.3	-----	----	---	-----	----	---
96.80 97.10	12.1 11.0	2.0 1.5	96.90 97.20	4.60 3.00	3.0 3.0	96.90 97.20	18.5 14.60	1.8 1.5	96.80 97.10	12.0 9.3	3.2
-----	----	---	97.80	4.0	4.7	-----	----	---	-----	----	---
-----	----	---	-----	----	---	99.50	6.0	3.0	99.30	5.1	2.0
102.50	4.0	2.0	-----	----	---	102.60	9.0	3.0	102.60	6.1	2.0
104.60	4.0	2.0	-----	----	---	104.20	9.7	4.0	104.20	7.0	3.5
-----			106.60	7.4	4.6	-----	----	---	106.60	5.8	3.0
X-RAY DIFFRACTION DATA - POSITIVE PLATES											
19.1	42.0	6.3	19.0	55.5	5.2	19.2	⩾100	---	19.0	77.4	2.7
33.1	16.0	3.2	33.2	28.0	3.8	33.0	57.7	3.0	33.0	24.7	3.0
38.5	39.0	5.4	38.5	57.8	6.7	38.5	⩾100	---	38.4	56.0	4.2
44.5	⩾100	---	44.6	⩾100	---	44.5	⩾100	---	44.6	⩾100	---
51.8	⩾100	---	51.9	⩾100	---	51.9	⩾100	---	51.9	⩾100	---
59.1	10.0	5.5	59.2	19.5	4.0	59.0	36.4	3.0	58.9	13.8	6.0
62.7	7.8	5.0	62.8	11.4	4.2	62.6	28.0	3.0	62.5	10.0	7.0
69.7	4.4	12.0	69.7	5.0	8.0	69.4	6.0	3.0	69.0	7.0	7.0
----	----	---	----	----	---	70.4	11.8	3.6	70.2	10.0	6.0
72.7	4.5	6.0	72.7	5.6	7.5	72.7	13.3	4.2	72.5	9.0	6.0
* 76.2 76.4	40.4 42.4	2.5	76.4 76.5	39.3 39.3	2.5	76.4 76.5	43.3 43.8	2.7	76.4 76.6	63.6 43.5	2.1
82.5	3.0	5.0	82.5	4.0	3.8	82.5	7.8	4.4	82.5	5.0	5.5
92.8 93.1	47.0 44.0	3.0	92.9 99.2	46.0 38.6	3.0	92.8 93.1	48.0 38.9	3.2	92.8 93.2	67.8 45.6	2.5
98.2 98.6	14.5 12.4	3.2	98.4 98.8	15.0 12.2	3.0	98.4 98.6	17.7 15.2	3.3	98.4 98.8	22.0 15.7	3.0
100.8	2.0	5.0	100.9	3.0	3.5	100.7	9.0	5.6	----	----	---

2θ = Reflection angle, I = Relative intensity, B.W. = Band width at 1/2 I

* $\frac{76.2}{76.4}$ for example indicates doublet

5. Other Testing

Characterization of heat sterilized plates using electron microscopy and B.E.T. (surface area) methods were initiated this quarter. Due to the rough and porous nature of battery plates, special techniques have to be developed for the electron microscopic examinations.

B.E.T. (surface area) measurements normally require that samples be prepared by heating (above 100°C) for several hours under vacuum (~50 microns or less). Heating of battery plates to these temperatures would cause (a) some decomposition and/or loss of water and subsequent change in structure and (b) simulation of a sterilization process so that pre-sterilization characteristics of the plates could not be accurately measured. During this quarter a preconditioning cycle for plates was developed and will be used next quarter for the determination of B.E.T. surface areas on both positive (nickel) and negative (cadmium) plates.

6. Planning and Selection of Factors for the Design of Statistical Experiments

One of the tasks in the development of satisfactory heat sterilizable, sealed Ni-Cd battery is to perform planned statistical studies to determine the effects of the following variables on the cell performance.

A. Concentration of KOH (2 levels)

B. Addition of Li^+ Na^+ and Bi^{+++} ions and surfactants (2 types) to the electrolyte

C. Addition of Tl^+ and In^{+++} ions to the negative plate

D. Addition of Co^{++} ions to the positive plate

E. Various separators (2 levels)

Assuming only two levels of electrolyte concentration and two types of separator material, a two level of factorial experiment with only two cells of each kind (one control, one for sterilization) with the twelve factors listed above would require over 4,000 cells. . . . This does not include the important design factors such as the amount of electrolyte (% pore fill), the charge adjustment on the negative [$Cd:Cd(OH)_2$ ratio] or the ratio of the total cadmium to nickel hydroxide and cell geometry factors. It is therefore necessary to screen the effects of many of these variables in preliminary screening experiments, so that the variables showing very significant and reproducible effects can be incorporated in the final factorial experiments.

A meaningful factorial experiment requires a fairly accurate knowledge of the experimental error and a high degree of reproducibility of cells. TI

Prismatic production cells, based upon non-woven nylon (polyamide) separator material have achieved a high degree of reproducibility and uniformity. However nylon separator thermally decomposes and/or dissolves in KOH during the heat sterilization treatment. Before considering the factorial experiment any further, it is necessary to fulfill the following requirements for heat-sterilizable, sealed Ni-Cd cells:

1. Establish the reproducibility and uniformity of cells with heat-sterilizable polypropylene separators. Both Pellon 14019 and FT 2140 show fairly good reproducibility and uniformity in the initial experiments with prismatic cells. Cylindrical cells used in the initial screening tests with these separators have not exhibited the required degree of uniformity and since the contract requirements specify prismatic cells, no further work with cylindrical cells is planned.

2. A heat sterilizable seal that will withstand corrosive and electrochemical environments and also maintain its hermeticity during the heat sterilization treatment and the cycling requirements. Glass to metal seals coated with several layers of cured KEL-F withstand these conditions satisfactorily and this seal is therefore selected for the factorial experiments.

3. Development of a reproducible heat-sterilizable, hermetically sealed standard cell (control cell) against which further development and progress can be measured. This requires, in addition to the satisfactory separator and seal development stated above, the determination and control of the following cell variables:

A. Can material, stainless steel and size as described in the first quarterly progress report.

B. Electrode plate size and numbers of positive and negative plates per cell as reported in Task II of this progress report.

C. Electrolyte concentration (30% KOH).

D. Amount of electrolyte for optimum capacity, resistance and oxygen recombination rates during overcharge.

E. Geometrical factors, particularly core compression which has an effect on delivered capacity, resistance and rates of recombination of oxygen.

F. Cell assembly (fabrication) procedure to give a highly reliable, high quality cell.

Variables A, B, C, and F are now essentially fixed as indicated. This is based upon our earlier studies and the results obtained during this contract work. Cell assembly procedure is described under the task on cell fabrication and testing. Work is now in progress on item D to determine the optimum electrolyte amount for high capacity, low resistance and high oxygen recombination rate during overcharge. Prismatic cells with varying amounts (60%, 70%, 80%, 90% of free pore volume) have been fabricated and are under test now. In addition to capacity, voltage and resistance data, rate of pressure increase during charge and the rate of pressure decay during stand are obtained to get the rate constant for oxygen recombination with various % pore fills. These data will be presented in the next quarterly progress report.

Narrowing down other factors for the statistical experiments:

Thallium Ion Addition to the Negative Plate

It was originally planned to incorporate thallium hydroxide in the negative plate to improve its performance characteristics. The idea of using thallium ion addition was a logical follow-up of the indium ion addition which has been described by other workers as having a beneficial effect on the negative plate properties since thallium (At. No. 81) is the next element in the family of similar elements Ga, In, Tl. However, the primary requirement in the addition of ions to positive or negative plates is that the hydroxide of the metal ion to be added should be highly insoluble. Any appreciable solubility of the metal hydroxide in the negative plate could result in shorting by the plating of dissolved metal ions during charging sequence. A literature search of the solubility of the thallium hydroxide was therefore undertaken. "A Comprehensive Treatise on Inorganic and Theoretical Chemistry" by Mellor, Vol. 5, page 431, gives the following data for the solubility of thallium hydroxide in water as a function of temperature:

Solubility (grams/liter) of TlOH in Water

Temp(°C)	0	18.50	29.00	32.10	40.0	54.30	78.5	99.5
S(g/l)	25.44	34.44	39.85	41.14	49.5	64.97	103.3	148.3

The aqueous solution of thallium hydroxide is strongly alkaline. Because of the relatively high solubility of the thallium hydroxide its addition to the negative plate is not expected to have any permanent beneficial effect since as soon as it is oxidized during the discharge cycle, it will dissolve out in the battery electrolyte. For this reason,

it is decided not to include thallium ion addition in the factorial experiment but determine its effect in initial screening experiments.

Indium Ion Addition to the Negative Plate

Special negatives impregnated with a solution of 5% indium nitrate added to the standard cadmium nitrate impregnation liquor will be compared against the negatives not containing any indium in preliminary screening experiments. If indium ion addition is found to have permanent beneficial effect on the performance of the negative plate, it will be incorporated in fractional factorial experiment. A preliminary review of literature does indicate beneficial effect of indium ion addition to the negative plate under certain conditions.

Cobalt Ion Addition to the Positive Plate

Special positive plates impregnated with a solution of 5% by weight of cobalt nitrate added to the standard nickel nitrate impregnation liquor will be compared against the positives not containing any cobalt in preliminary experiments. If cobalt ion is found to have permanent beneficial effect on the performance of the positive plate, it will be investigated further. In the past, in some company funded programs, we have investigated the effect of cobalt addition. Its main effect, under the conditions of our factorial experiment was to enhance the corrosion of the sinter and thereby increase the capacity. However the corrosion of the sinter weakens the support mechanically and may also increase its resistance. There is a large amount of work on cobalt addition reported in the literature and this is being collected and reviewed now.

Bismuth Ion Addition to the Electrolyte

Appreciable solubility of the bismuth hydroxide in the potassium hydroxide electrolyte is a pre-requisite for studying the effect of bismuth ion on the performance of sealed Ni-Cd cells.

Solubility data for bismuth hydroxide in KOH solutions reported by Seidell in "Solubilities of Inorganic and Metal Organic Compounds", Volume 1, page 437 are given below. The concentration of KOH in terms of grams of KOH per liter reported by Seidell is converted to molarity for easy comparison with Ni-Cd battery electrolyte.

Molarity of KOH	0.5	1.0	2.0	3.0	4.0	5.0	6.0	8.0	10.0
Sol. g/l (20°C)	0	trace	0.037	0.074	0.100	0.124	0.137	0.137	0.174
Sol. g/l (100°C)	0.188	0.249	0.373	-----	0.622	0.622	-----	1.494	2.054

These data show that at room temperature in 6 to 8 molar KOH, the solubility is only about (0.137)(100)/(1000)(1.37)g/cc or 0.01 per cent. This maximum amount of bismuth ion that can be incorporated in the electrolyte appears to be too low to have any permanent beneficial effect on the performance of Ni-Cd cells. The effect of trace quantities of bismuth hydroxide addition to the electrolyte will be investigated in separate small experiments.

Electrolyte Concentration Effect and Addition of Li^+ and Na^+ Ions

Previous experience in our laboratory and some published data indicate that the following electrolyte concentrations and additives should be further investigated for their effect on heat sterilizable battery performance:

1. KOH electrolyte concentrations, 30 and 34 weight per cent.

2. Binary electrolyte with 24% KOH containing 20g/liter of lithium hydroxide.

3. Binary electrolyte with 24% KOH containing 48 grams per liter of sodium hydroxide.

The design of factorial experiments with factors and levels selected, through preliminary fractional factorial experiments, in underway now.

Literature Review:

In order to take full advantage of the published literature on the effects of the above factors on the performance of sealed nickel-cadmium cells, a comprehensive review of the subject is undertaken and will be presented in the next quarterly report. The effects reported in the literature will be compared and correlated with the data obtained during this contract work. The effects found significant will be incorporated into the design of final cells.

Tasks II and III: Cell Case, Fabrication and Test

The details of the design and construction of the stainless steel cases and tops as well as glass to metal seals coated with KOH resistant polymeric materials for the rectangular, 4 ampere hour cells were given in the 1st Q Progress Report. An examination of the glass to metal seals coated with KOH resistant Durafilm K as well as KEL F polymeric materials, after two heat sterilization cycles has indicated that the adherence of KEL F polymer coating is somewhat better than the Durafilm K coating. Therefore the Kel F coating is selected for the final design of the cells.

Cell components (positive plate material, negative plate material, separators, leads, etc.) for approximately 300 rectangular cells have been received.

The electrode sizes are as follows:

Positive plates: 2.25" x 1.812"; .025" nominal thickness.

Negative plates: 2.25" x 1.812"; .025" nominal thickness.

Number of plates per cell: 9 positive plates and 10 negative plates. Some cells have been prepared with 8 positives and 9 negatives and one cell with 10 positives and 11 negatives. Nineteen plate construction, which is the standard for most rectangular production cells appears to give optimum performance and is relatively easy to assemble compared to 21 plate construction. Therefore nineteen plate construction is adopted as the standard(control) cell for further studies.

Cell Fabrication Procedure

Each cell assembly for the production of rectangular 4 AH cells used in this program involves the following operations:

1. Preparation of top cover assembly described in the 1st Q Progress Report.
2. Preparation of two terminal sub assemblies (glass to metal seals) also

described in the 1st Q Progress Report.

3. Leak check for each terminal.
4. Welding the terminal sub assemblies to the top cover.
5. Leak checking the top assembly for weld integrity.
6. Coating the glass to metal seals with thermally stable, KOH resistant polymer coating by an outside vendor.
7. Cutting the positive and negative plates to size, quality control checks for burrs, loose wires etc.
8. Welding nickel tabs to positive and negative plates.
9. Separator assembly (wrapping the separator around each positive and negative plate)
10. Core compression under controlled conditions (~400 psia)
11. Trimming the package and checking for any possible short circuits.
12. Welding the positive and negative tabs to the cover terminals.
13. Q.C. checking for short circuits in the cell package.
14. Inserting the cell package into the steel case.
15. Q.C. checking for any possible short circuits in the cell package.
16. Welding the top cover to the case .
17. Q.C. checking for any possible short circuits during welding.
18. Q.C. checking for weld integrity.
19. Electrolyte addition (predetermined fill level).
20. Cell resistance check.
21. Attaching pressure relief vent or pressure gage as needed.
22. Cells ready for conditioning and tests.

The test procedure for these cells is described in the Task I under electrochemistry. Approximately 100 of such rectangular cells are now under construction for the first factorial experiment.

Conclusions

1. Optical microscopic studies of both positive and negative plates reveal that there is no marked corrosion of the nickel support structure upon heat sterilization.

2. The color, shade and texture of the positive and negative plates are altered during heat sterilization.

3. The porosity and pore size distribution of the positive (nickel oxide) plate changes during heat sterilization. This may unfavorably affect the electrolyte distribution in the cell and result in high gas pressure during overcharge.

4. X-ray diffraction data indicate that either morphological or crystallographic changes occur in the negative (cadmium) electrode during heat sterilization. These effects are not easily reversed and may impair the electrochemical efficiency of this electrode.

5. Both Pellon polypropylene 14019 and FT 2140 separators will be included in the factorial experiment owing to better uniformity of the former and superior strength of the latter.

6. Several sealed, rectangular, 4 Ah cells made with these separators have successfully passed two heat-sterilization cycles and have delivered over 70% of their rated capacity. However, the charging mode of the heat-sterilized cells need to be optimized to avoid excessive voltage and pressure buildup, particularly during the first few post-sterilization cycles.

7. Glass to metal seals specially coated with KOH resistant, thermally stable KEL-F polymer satisfactorily withstand heat sterilization without leakage and will be used in all further investigations of heat sterilizable batteries.

8. Important factors, to be further investigated in factorial design experiment have been identified.

Conferences

Drs. Ralph Lutwack, Aiji A. Uchiyama and Gordon L. Juvinall from the Spacecraft Power Section, Jet Propulsion Laboratory, visited our Research and Development Laboratories on Friday, October 6, 1967. They reviewed the contract work as well as our electrochemical test facilities and Ni-Cd battery production facilities in connection with spacecraft power requirements.

Dr. Ralph Lutwack from the Jet Propulsion Laboratory visited the Research and Development facilities of Texas Instruments Incorporated, Attleboro, Massachusetts, on December 13, 1967 to review the progress of the contract work.