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REACTOR EXPERIMENTAL ENGINEERING DIVISION

THE EFFECT OF METALLURGICAL VARIABLES ON THE CORROSION OF EXTRUDED BERYLLIUM

Arnold R. Olsen

Date Issued

MAR 17 1952

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RESTRICTED DATA

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INTRODUCTION

This report is issued as Part I-C of a series of reports on the corrosion studies for the Materials Testing Reactor*. The data included here has been previously published in various Monthly and Quarterly Progress Reports of the Technical Division.

During the early months of 1949 vacuum cast and extruded beryllium seemed the logical production choice for metal for the Materials Testing Reactor. The reasons for this were reported in ORNL-298. There were, however, several questions as to the effect of various metallurgical treatments on the corrosion characteristics as well as the variation from end to end of a given billet. Consequently, the tests reported here were performed to investigate these variables and their effect on corrosion resistance.

* Other reports to date in this series are:

Part I: Interim report on the Corrosion of Beryllium in Simulated Cooling Water for the Proposed Development Reactor, James L. English, ORNL-298, March 16, 1949.

Part I-A: Initial Corrosion Tests on Brush Process QM and QRM Beryllium, Arnold R. Olsen, ORNL-733, July 14, 1950.

Part I-B: The Corrosion of Beryllium in Simulated Cooling Water for the Materials Testing Reactor, ORNL-772, James L. English, January 23, 1951.

Part II: The Corrosion of 356 Aluminum in Simulated Cooling Water for the Proposed Materials Testing Reactor, James L. English, ORNL-681, July 20, 1950.

ABSTRACT

A single vacuum cast and extruded billet of beryllium was divided into a number of corrosion samples and corrosion tested in simulated Materials Testing Reactor coolant for periods up to one year.

The samples were segregated as to position in the billet and metallurgical treatment; i.e, leading end, center, and trailing end of the billet, directly machined, annealed and machined, and annealed-machined and annealed.

It was found that there is no significant change in corrosion resistance throughout the length of the extrusion.

Overall corrosion attack and pitting rates increased directly with the number of anneals on short time exposures. However, the overall attack on longer term exposures, up to 12 months, is almost identical regardless of whether the sample has been annealed or not.

Further proof of the extreme sensitivity of beryllium to minor changes in the media was also found.

CORROSION TEST CONDITIONS

A complete description of the conditions of testing and the reasons for their establishment is given in ORNL-298.

For the purpose of convenience, a resume of these conditions is included here:

Medium	Deaerated and demineralized water	
	Total Solids	8.0 ppm
	Fe	0.05 "
	Al	0.05 "
	Ca	0.5
	Mg	0.5
	Na	1.0
	CO_2	2.0
	SiO_2	5.0
	Cl	0.2 (max)
	SO_4	5.0
	Cu	0.04
	B	0.0
	pH	5.5-6.5
	Specific Resistance (ohms/cc)	3.5×10^5
Temperature	85°C at atmospheric pressure 515°F at 840 psi gauge	
Hydrogen Peroxide	0.0005M, 0.005M	

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TEST MATERIALS

Types of Materials

Unlike previous tests reported in this series, only one piece of beryllium divided into a large number of specimens was used. One carefully chosen extruded billet was tested.

Since the only intimate contact with a dissimilar metal is to be with the end boxes, and in order to decrease the total number of tests, only couples with 356 aluminum were investigated. Previous reports have adequately covered the effect of 2S aluminum-beryllium couples.

BERYLLIUM

Casting and Extrusion History

Billet No. 785 vacuum melted and cast from "pebble" supplied by Brush was used in this investigation. The analysis of the billet as cast was:

Fe	0.15%	Mn	120 ppm
C	0.04%	Ca	130 ppm
Si	0.2%	Ni	100 ppm
Al	0.11%	Cu	40 ppm
Mg	0.011%	Cr	130 ppm

The 20 lb, sound billet, 6.1" in diameter, was jacketed and heated to 1840°F and held two hours and ten minutes before being extruded. The extruded billet measured 1 3/8" x 3 7/8" x 44.13" in the jacket after trimming. The extrusion ratio was 1:6.45.

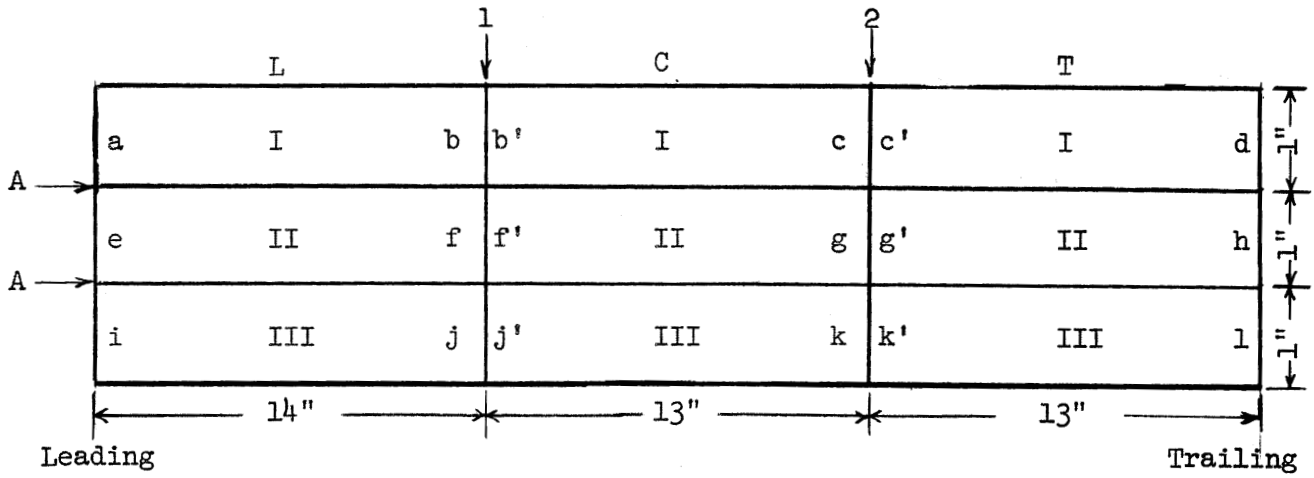
Sectioning and Treatment of the Beryllium Billet

The billet was dejacketed at ORNL and cropped ends were deep etched and examined macroscopically to check the soundness of the extrusion.

Subsequently the billet was divided as shown in Figure 1, which is a copy of the machining order forwarded to the shop.

FIGURE 1

PREPARATION ORDER FOR SAMPLES



1) Make cuts 1 and 2 on each billet marking each piece with appropriate number and positioning letter:

   L Leading end
   C Center
   T Trailing end

   Note: The billet number is stamped on the trailing end of each billet.

2) Make cut A on all sections - labelling all pieces as in step (1).

3) Return piece (II, III) for annealing.

4) Machine piece I to 1" round stock.

5) Machine specimens from round stock to size 1" diameter x 1/4" thick with a 1/4" diameter hole in the center and place in appropriately marked container.

6) When piece (II, III) is returned in the annealed condition make cut B, again properly tagging each piece.

7) Repeat steps 4 and 5 for pieces II and III.

Sectioning and Treatment of the Beryllium Billet (Cont'd)

Annealing of the metal was done in two ways. Annealing before machining was in a muffle furnace under an argon atmosphere at 800°C for one hour. Finished machined samples were annealed in a combustion tube furnace one hour at 825°C in a vacuum of less than 200 microns.

The sample identification is by the small letters shown in Figure 1; thus, A-5 is the fifth sample cut from the a end of piece a-b. Capital letters L, C and T represent the general location in the billet Leading end, Center, and Trailing end, respectively.

Roman numerals are used to identify the type of treatment the sample received:

Condition I: The sample was machined directly from the extruded billet.

Condition II: The billet was annealed prior to the machining of the sample.

Condition III: The billet was annealed, the sample machined, and the finished sample was reannealed.

356 Aluminum

Aluminum casting alloy 356 of the following composition was the only dissimilar metal investigated.

Si	-	7.46%	
Cu	-	0.09%	(0.20 max.)
Mg	-	0.37%	
Mn	-	0.10%	
Fe	-	0.25%	(0.50 max.)
Zn	-	--	(0.10 max.)
Other	-	--	(0.10 max.)
Al	-	Balance	

This alloy was subject to the T-7 heat treatment before exposure:

1) Heat 12 hours at 538°C

2) Quench in boiling water

3) Stabilize at 227°C for 9 hours

CORROSION TESTING METHODS

Test Apparatus

Three types of apparatus were used in testing this material.

A) For the majority of the 60-day tests at 85°C, the arrangement described and pictured in ORNL-298 was used. Briefly, this consists of a five liter, three-necked distilling flask supported on a transite box with a 375 watt infra-red bulb for heating. The temperature is controlled with ± 0.5°C with a thermoregulator-relay system. Air cooled reflux condensers prevent solution loss; hydrogen peroxide additions are made periodically from burettes installed on each test.

B) For the planned interval tests and for 60-day Be-356 Al couple tests, electrically heated water baths holding three 16 liter battery jars were used. The temperature is controlled to ± 1.0°C and excessive solution loss is prevented by aluminum covers. Hydrogen peroxide additions are made through small holes in the covers from pipettes.

C) The high temperature, high pressure tests were made in a large autoclave fitted with three stainless steel containers, each separately sealed. Temperature variation on the outside of the pressure wall is ± 3°F which is estimated as maintaining the inside temperatures at ± 1°F.

Types of Tests

All tests were conducted under essentially static conditions. The only flow being caused by convection currents from the heating, which prevented the solution around the samples from being completely stagnant.

Tests of beryllium alone were conducted by hanging each sample separately from a pyrex glass hook, while couple tests were fashioned by bolting a piece of beryllium to a sample of 356 aluminum using a bolt and nut of the aluminum alloy. This assembly was then hung from a glass hook.

No practical means of totally insulating the beryllium from the stainless steel was found for the high temperature tests. However, it has been shown previously that coupling effects can be reduced to near extinction by the use of nichrome supporting wires.

All tests were conducted with the samples totally immersed in the media.

Solution Control and Maintenance

The solutions in all 85°C tests were changed weekly to minimize corrosion product build up and specific ionic depletion. With the high temperature tests, the original solution was used for the entire testing period.

pH checks and adjustments were made daily. Control being the addition of 0.1 normal sodium hydroxide or nitric acid.

There was no hydrogen peroxide in the high temperature tests. At 85°C the concentration could not be perfectly maintained. However periodic additions of 4% H_2O_2 solution to type A tests and 30% H_2O_2 to type B tests held the concentration within reasonable limits. In addition, each solution was analyzed daily and corrections made accordingly.

A detailed analysis of the decomposition variables for hydrogen peroxide has been reported previously.

CORROSION DATA FOR 60-DAY TESTS IN 0.0005M H_2O_2 SOLUTIONS AT 85°C

Two samples from the leading end, center and trailing end of the billet were tested for each condition. All tests were run in "A" type apparatus for 60 days at 85°C with the hydrogen peroxide concentration at 0.0005 molarity.

These data are listed in Table 1 and graphed in Figures 2 and 3. The results indicate that the leading end of the billet is slightly superior from the overall corrosion standpoint. Pitting susceptibility is apparently independent of location along the billet. As for the effects of heat treatment, both the as-machined and annealed-machined-annealed condition show better overall corrosion resistance than the annealed-machined condition. Susceptibility to pitting attack is markedly increased under these conditions by annealing. Both pit frequency and pit depths were higher by factors of 2 to 3 for Conditions II and III over Condition I.

[redacted]

TABLE 1

CORROSION OF MACHINED AND HEAT TREATED EXTRUDED BERYLLIUM IN 0.0005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

Sample	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
A-7	I	2.6	2.4	4.6
A-17	I	1.6	5.6	1.0
C-7	I	2.1	3.5	7.0
C-17	I	1.7	2.2	6.0
D-7	I	2.1	3.2	8.5
D-17	I	2.0	5.7	8.1
F-7	II	7.4	12.0	6.5
F-17	II	5.6	5.8	12.1
G-7	II	5.6	6.6	10.1
G-17	II	5.7	4.5	10.2
H-7	II	6.0	3.0	7.9
H-17	II	3.7	6.5	4.0
J-7	III	3.9	1.8	1.6
J-17	III	4.7	10.5	0.3
K-7	III	4.8	2.6	8.6
K-17	III	4.7	5.0	8.5
L-7	III	3.2	10.0	8.6
L-17	III	3.4	7.0	1.7

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[redacted]

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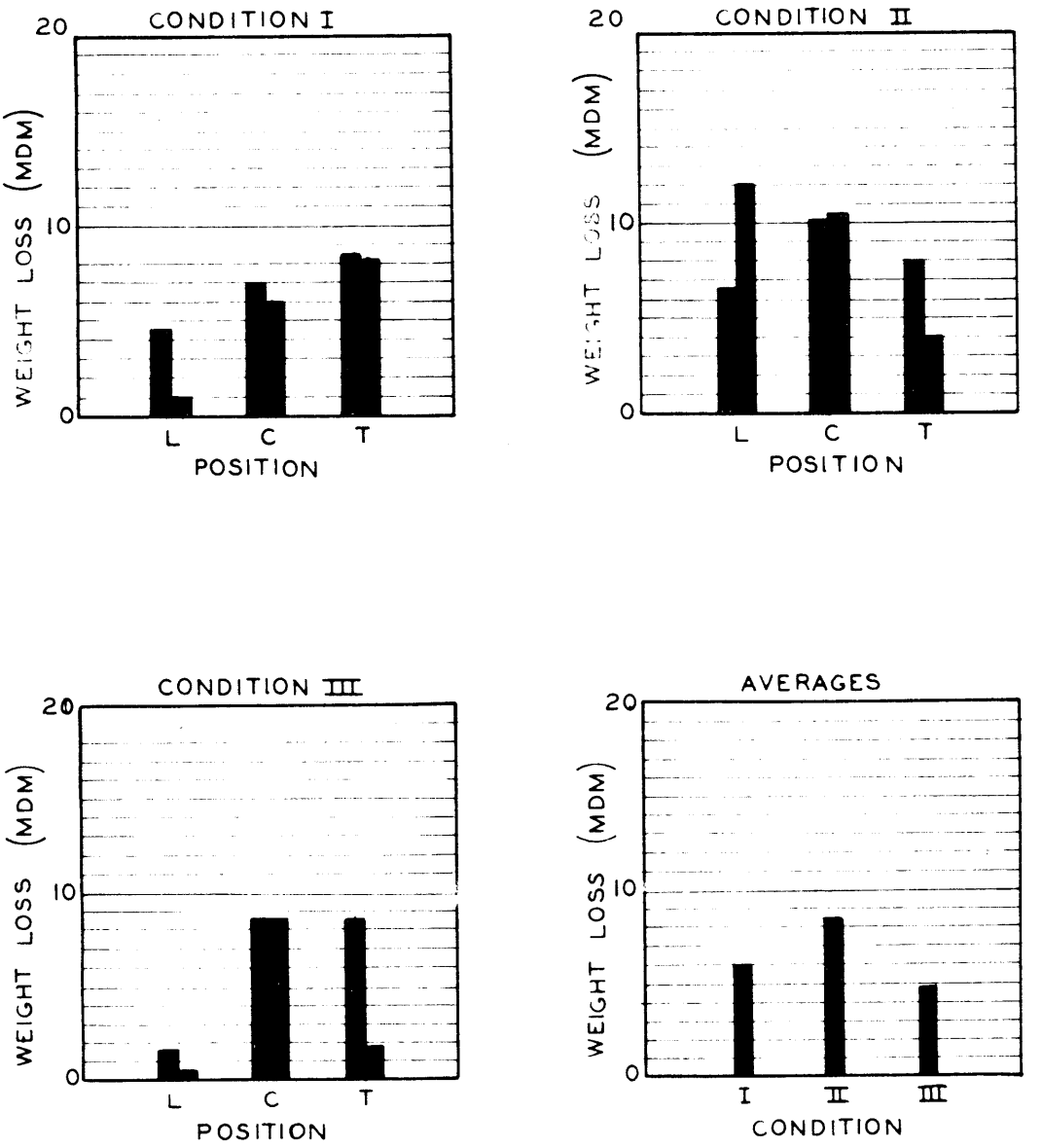
FIGURE 2

COMPARATIVE CORROSION RATES OF EXTRUDED BERYLLIUM IN 0.0005 M H_2O_2 SOLUTIONS

TEMPERATURE - 85°C
TIME ———— 1462 HOURS
DUPLICATE SAMPLES



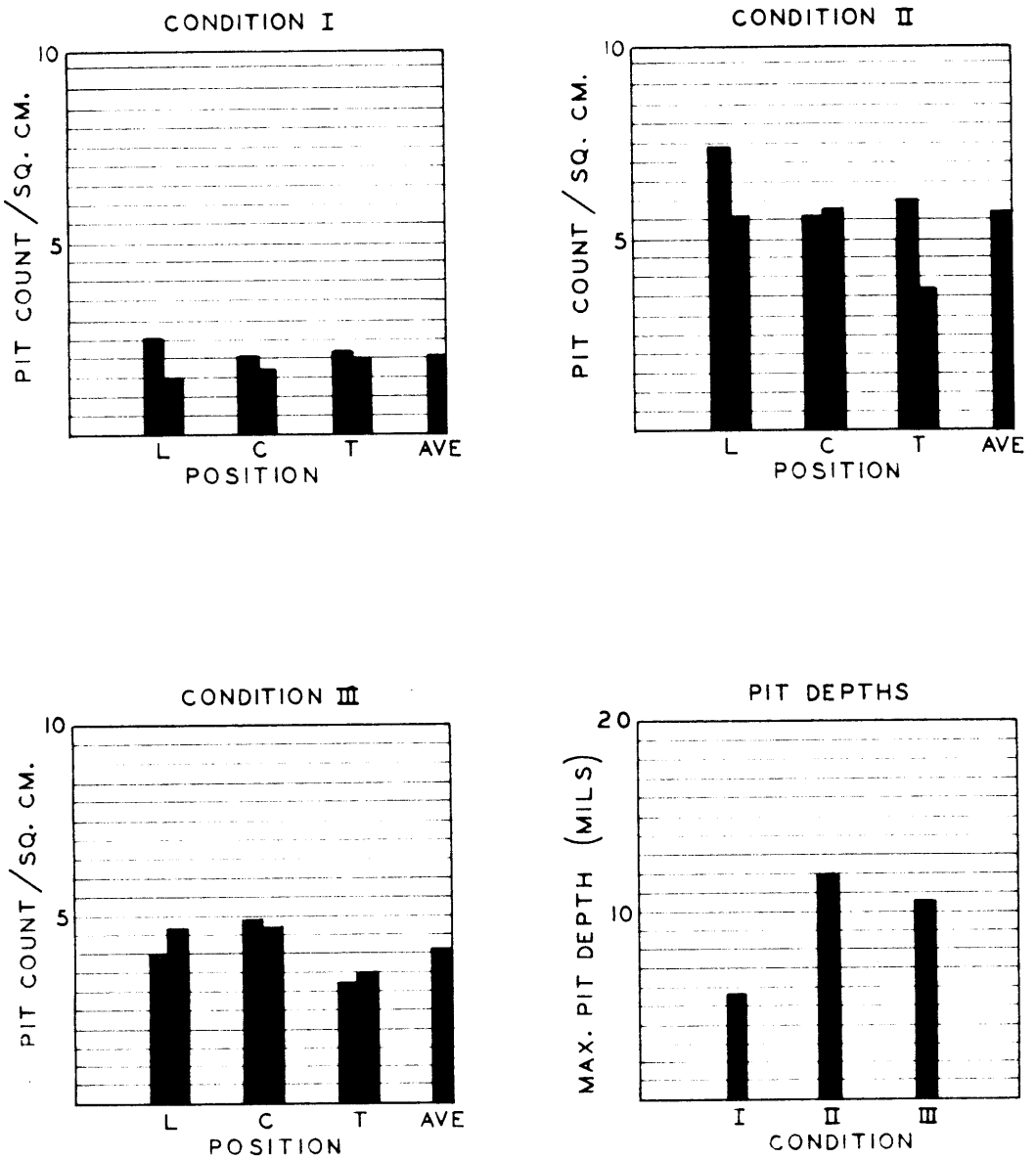
LEGEND
L - LEADING END
C - CENTER
T - TRAILING END
I - AS MACHINED
II - ANNEALED - MACHINED
III - ANNEALED - MACHINED - ANNEALED

FIGURE - 3

[redacted] Dwg. 13598

COMPARATIVE PITTING RATES OF EXTRUDED BERYLLIUM IN 0.0005 M H_2O_2 SOLUTIONS

TEMPERATURE - 85°C
TIME - 1462 HOURS
DUPLICATE SAMPLES



LEGEND

L - LEADING END
C - CENTER
T - TRAILING END
I - AS MACHINED
II - ANNEALED - MACHINED
III - ANNEALED - MACHINED - ANNEALED

CORROSION DATA FOR 60-DAY TESTS IN 0.005M H_2O_2 SOLUTIONS AT 85°C

Two samples from each of the three positions in each of the three conditions were exposed in type "A" apparatus to 0.005 molar hydrogen peroxide concentrations at 85°C for 60 days.

The results of this series of tests are listed in Table 2, and Figures 4 and 5 graphically illustrate this data. Overall corrosion rates are definitely higher in this higher peroxide solution. The effects of position from leading end to trailing end are completely masked by the variations in samples. These results indicate, however, that the overall attack is more severe in the case of annealed samples (Condition II and III). Pitting frequency increases from Condition I to III to II, with the maximum pit depth being nearly equal, but slightly decreasing from Condition I to III. Comparison with pitting attack in the lower H_2O_2 solution shows a marked decrease in frequency with little change in the depth of pits for the higher peroxide content.

TABLE 2

CORROSION OF MACHINED AND HEAT TREATED EXTRUDED BERYLLIUM IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

Sample	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
A-6	I	1.0	10.7	12.1
A-16	I	0.8	5.8	13.4
C-6	I	1.2	10.0	5.8
C-16	I	0.6	6.3	9.1
D-6	I	0.2	4.0	6.4
D-16	I	0.3	6.5	10.6
F-6	II	5.1	7.5	10.0
F-16	II	2.6	5.6	14.0
G-6	II	2.6	4.9	17.1
G-16	II	3.5	5.0	7.3
H-6	II	3.5	9.7	10.4
H-16	II	3.6	4.4	18.0
J-6	III	0.7	8.0	8.8
J-16	III	1.7	6.5	6.4
K-6	III	3.0	6.0	16.5
K-16	III	1.8	3.7	7.8
L-6	III	2.9	7.7	18.2
L-16	III	1.4	4.7	18.9

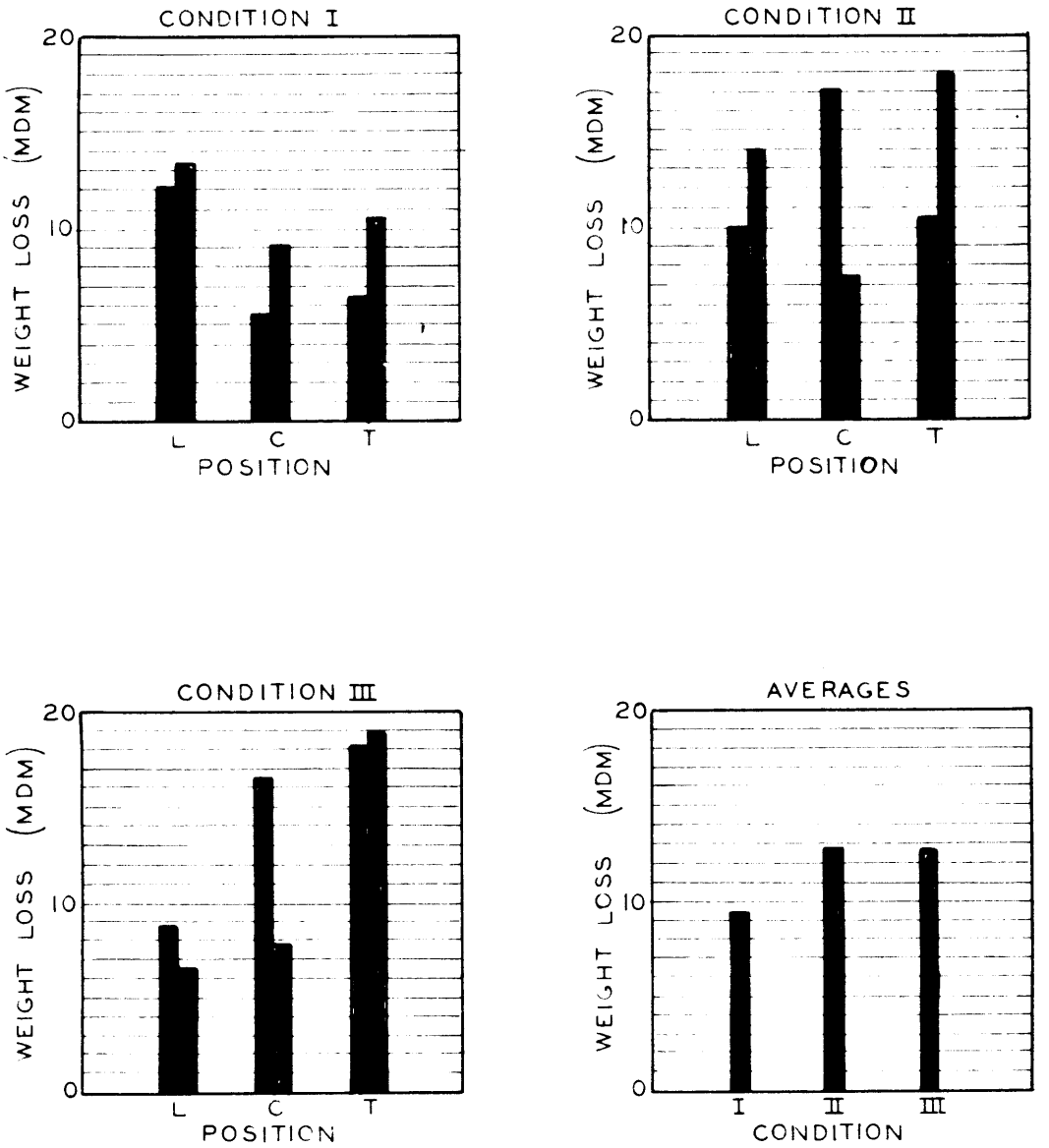
[redacted]

Dwg. 13599

FIGURE 4

COMPARATIVE CORROSION RATES OF EXTRUDED BERYLLIUM IN 0.005 M H_2O_2 SOLUTIONS

TEMPERATURE -- 85°C
TIME---------- 1582 HOURS
DUPLICATE SAMPLE



LEGEND
L - LEADING END
C - CENTER
T - TRAILING END
I - AS MACHINED
II - ANNEALED - MACHINED
III - ANNEALED - MACHINED - ANNEALED

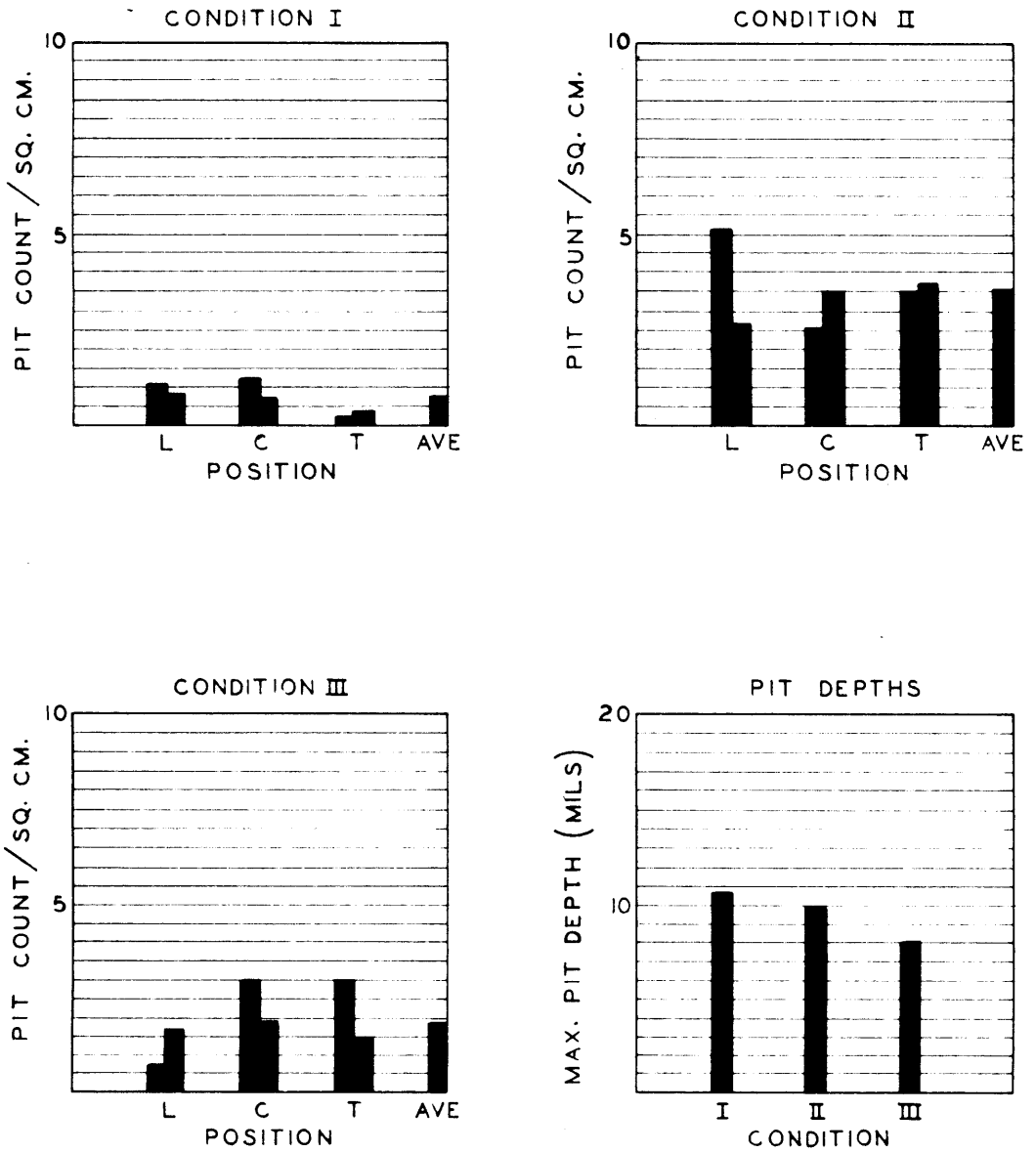
[redacted]

Dwg. 13600

FIGURE - 5

COMPARATIVE PITTING RATES OF EXTRUDED BERYLLIUM IN 0.005 M H_2O_2 SOLUTIONS

TEMPERATURE - 85°C
TIME - 1582 HOURS
DUPLICATE SAMPLES



LEGEND
L - LEADING END
C - CENTER
T - TRAILING END
I - AS MACHINED
II - ANNEALED - MACHINED
III - ANNEALED - MACHINED - ANNEALED

CORROSION DATA ON AS-MACHINED AND ETCHED BERYLLIUM IN 0.005M H_2O_2 SOLUTIONS FOR 60 DAYS AT 85°C

In an effort to determine the effect of the worked surface on corrosion behavior of extruded beryllium, two samples from the leading end, center, and trailing end of the billet in the as-machined condition were treated with the Battelle Etch before exposure.

The results of this test as shown in Table 3 and Figure 6, indicate that the metal has been activated corrosion-wise. There is no difference in the pitting frequency nor the maximum pit depth but the overall corrosion rate is approximately three times as great under this condition as compared to the rates reported in previous section. Whether this activation is effective for long periods of time, unfortunately, could not be determined with the available amount of material.

TABLE 3

CORROSION OF MACHINED AND ETCHED EXTRUDED-BERYLLIUM IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

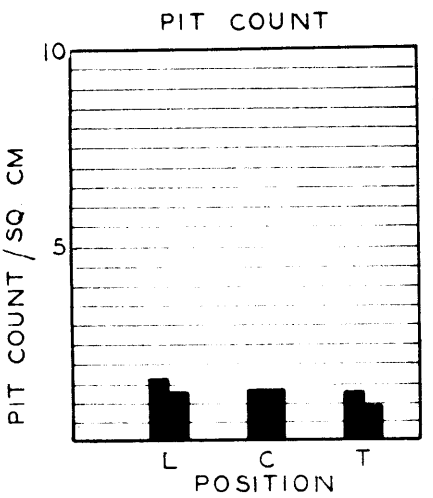
Sample	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
A-4	IV	3.3	6.8	31.3
A-14	IV	2.6	9.5	28.7
C-4	IV	2.7	7.3	28.0
C-14	IV	2.7	8.3	29.8
D-4	IV	2.4	11.8	29.6
D-14	IV	1.8	14.3	29.4

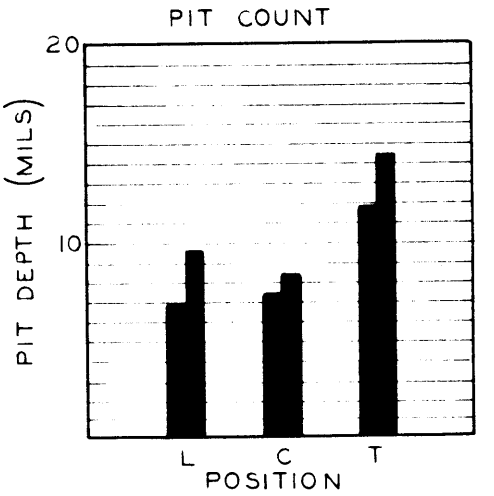
[redacted] Dwg. 13601

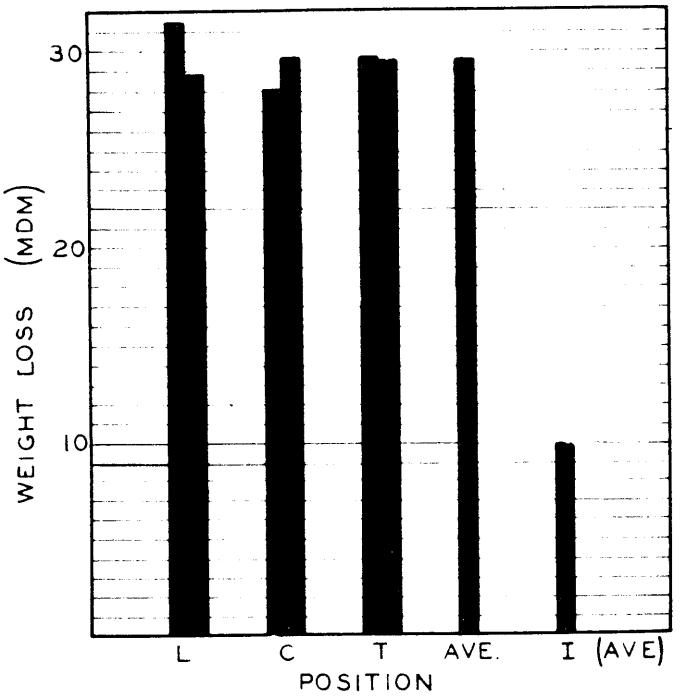
FIGURE 6

CORROSION ATTACK ON EXTRUDED BERYLLIUM IN THE MACHINED AND ETCHED CONDITION IN 0.005 M H_2O_2 SOLUTIONS

TEMPERATURE ---85°C
TIME ----------1439 HOURS
DUPLICATE SAMPLES







LEGEND
L-LEADING END
C-CENTER
T-TRAILING END
I-AS MACHINED

LONG TERM CORROSION TESTS IN 0.005M H_2O_2 SOLUTIONS AT 85°C

Since the chief variable in many forms of corrosion is time, a test was conducted in which six samples of each of the nine variables were tested for periods ranging from two months to twelve months. The planned interval type of test, even using so few samples, should give a result which shows the trend of the corrosion rates and any changes in type of attack with time.

The results of this series are tabulated in Tables 4, 5, and 6. Figures 7, 8, and 9 are graphs of the three conditions of working and three positions in the billet. It would be best to note that each point represents a single sample and thus is not an exact report of the attack to be expected in the given period of exposure.

Figures 10 and 11 compare only the conditions of metallurgical treatment with each point representing the average rate of attack for all three positions in the billet. It can be clearly seen from these that up to one year's exposure the rate of attack, as well as pitting susceptibility, is least for the as-machined condition and increases with the number of anneals.

Figures 12, 13, 14, 15, 16, and 17 are photographs taken of the samples as they were removed from the corroding medium. These clearly show the differences in susceptibility. Figure 18 shows views of the three blisters formed on the samples after one year of exposure. These blisters are the reason for the dotted lines in the previous graphs. It should be noted that one blister occurred on an annealed-machined sample, while two annealed-machined-annealed samples were blistered. None of the as-machined samples were so effected during the 12 months exposure.

TABLE 4

CORROSION OF EXTRUDED BERYLLIUM IN THE AS-MACHINED CONDITION IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR PERIODS UP TO 12 MONTHS

Sample	Exposure Duration (Months)	Material Condition	No. of Pits per specimen	Max. Pit Depth (mils)	Weight Loss (MDM)
A-1	2	I	2	3.4	2.6
A-2	4	I	1	8.7	0.8
A-11	6	I	1	2.5	2.4
A-12	8	I	4	5.2	12.8
A-21	10	I	None	-	0.2
A-22	12	I	2	2.5	0.5
C-1	2	I	2	5.8	2.6
C-2	4	I	1	5.2	1.3
C-11	6	I	2	5.5	2.7
C-12	8	I	4	4.3	11.1
C-21	10	I	4	3.2	10.0
C-22	12	I	4	9.6	10.6
D-1	2	I	2	2.0	13.2
D-2	4	I	4	4.8	11.2
D-11	6	I	1	8.6	1.4
D-12	8	I	1	6.9	1.6
D-21	10	I	3	13.6	7.6
D-22	12	I	2	7.4	1.9

TABLE 5

CORROSION OF EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED CONDITION IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR PERIODS UP TO 12 MONTHS

Sample	Exposure Duration (Months)	Material Condition	No. of Pits per specimen	Max. Pit Depth (mils)	Weight Loss (MDM)
F-1	2	II	2	3.2	11.0
F-2	4	II	6	6.8	9.1
F-11	6	II	5	3.5	11.5
F-12	8	II	6	13.3	11.2
F-21	10	II	3	11.2	13.5
F-22	12	II	4	5.8	11.3
G-1	2	II	1	2.6	12.4
G-2	4	II	3	8.5	12.4
G-11	6	II	5	6.0	10.3
G-12	8	II	4	24.4	10.9
G-21	10	II	2	9.6	8.5
G-22	12	II	2	1.8*	5.1*
H-1	2	II	5	5.5	12.9
H-2	4	II	2	6.6	9.6
H-11	6	II	3	10.2	9.7
H-12	8	II	3	15.2	8.3
H-21	10	II	2	25.6	10.7
H-22	12	II	2	28.0	7.1

*Blister formation

[redacted]

TABLE 6

CORROSION OF EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED-ANNEALED CONDITION IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR PERIODS UP TO 12 MONTHS

Sample	Exposure Duration (Months)	Material Condition	No. of Pits per specimen	Max. Pit Depth (mils)	Weight Loss (MDM)
J-1	2	III	1	11.7	13.2
J-2	4	III	2	3.4	5.9
J-11	6	III	1	3.5	5.8
J-12	8	III	2	1.2	2.4
J-21	10	III	None	-	1.9
J-22	12	III	2	5.8	2.5
K-1	2	III	3	12.0	11.8
K-2	4	III	8	4.0	5.1
K-11	6	III	6	8.9	11.4
K-12	8	III	4	24.6	21.1
K-21	10	III	12	13.6	8.0
K-22	12	III	19	5.6*	5.2*
L-1	2	III	8	7.0	21.5
L-2	4	III	7	11.3	27.2
L-11	6	III	14	7.5	37.4
L-12	8	III	8	29.4	13.2
L-21	10	III	3	13.6	14.7
L-22	12	III	2	9.6*	4.8*

*Blister formation

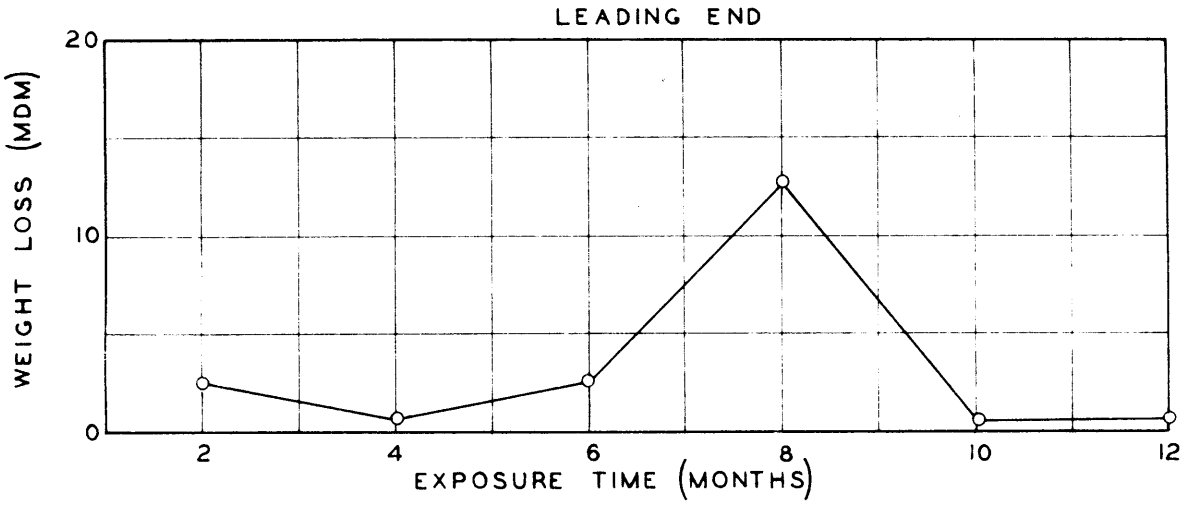
[redacted]

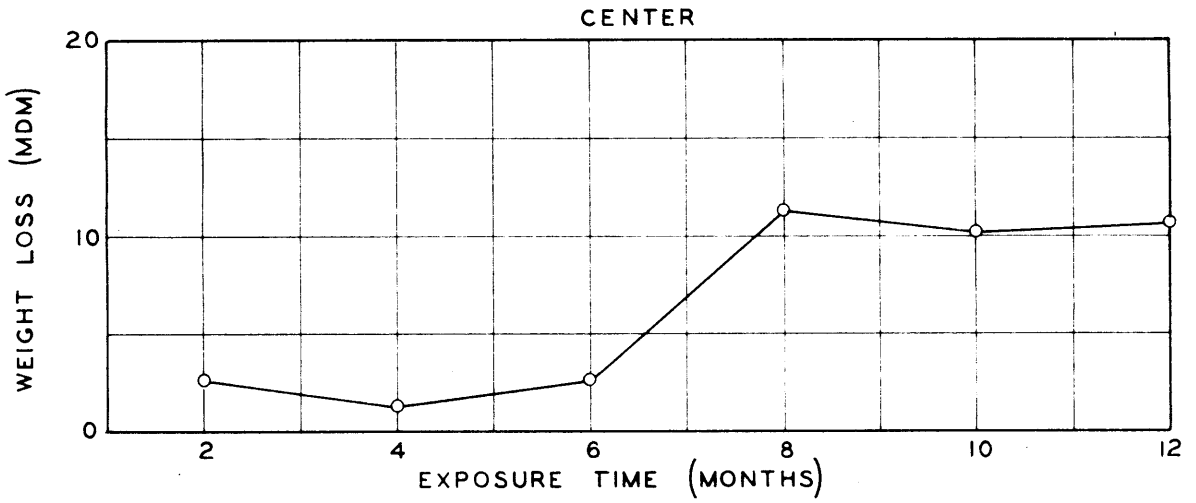
[redacted] Dwg. 13602

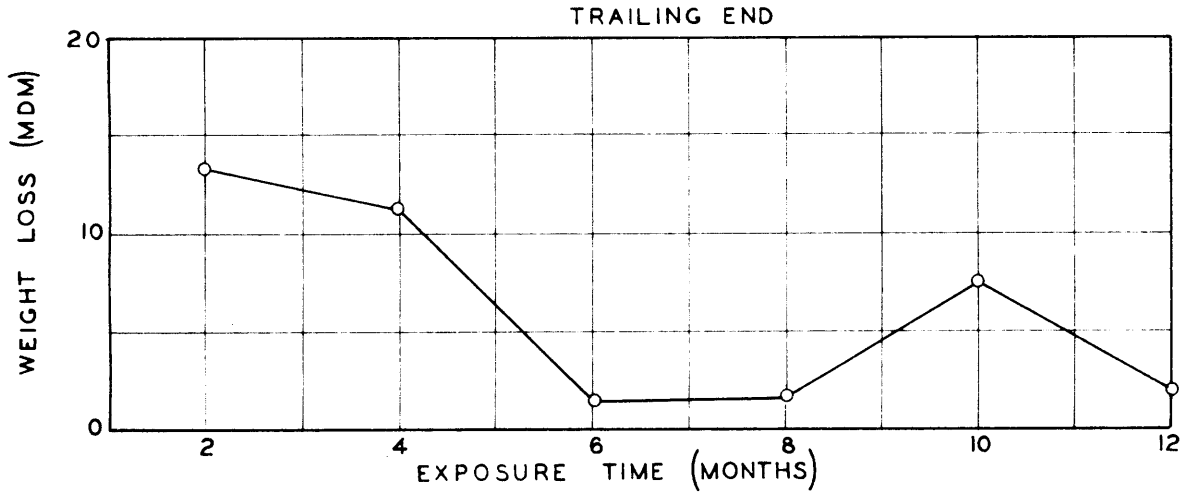
FIGURE 7

CHANGE IN CORROSION RATE WITH TIME FOR EXTRUDED BERYLLIUM IN THE AS-MACHINED CONDITION IN 0.005 M H_2O_2 SOLUTIONS

TEMPERATURE-85°C





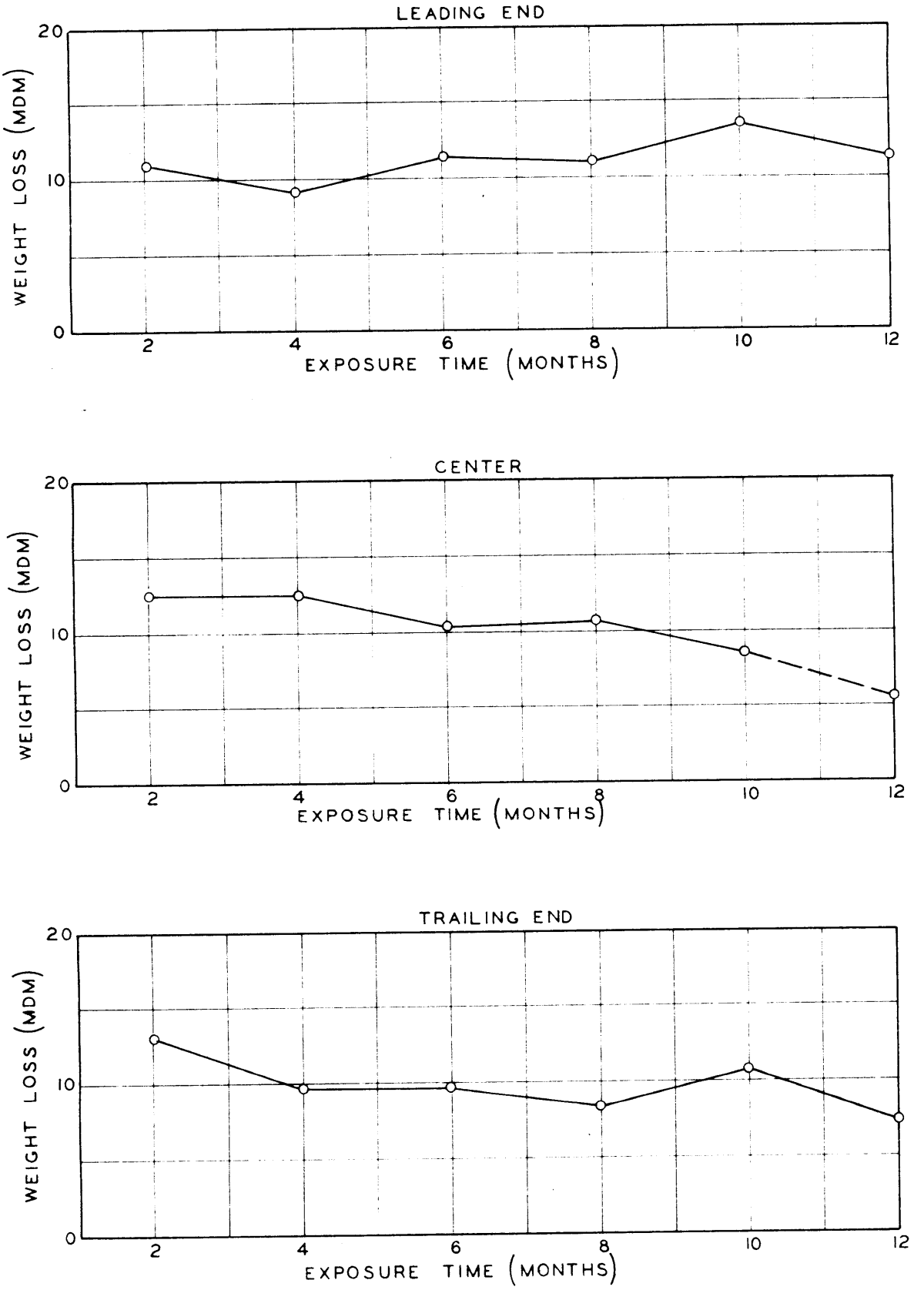


[redacted] Dwg. 13603

FIGURE 8

CHANGE IN CORROSION RATE WITH TIME FOR EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED CONDITION IN 0.005 M H_2O_2 SOLUTIONS

TEMPERATURE - 85°C



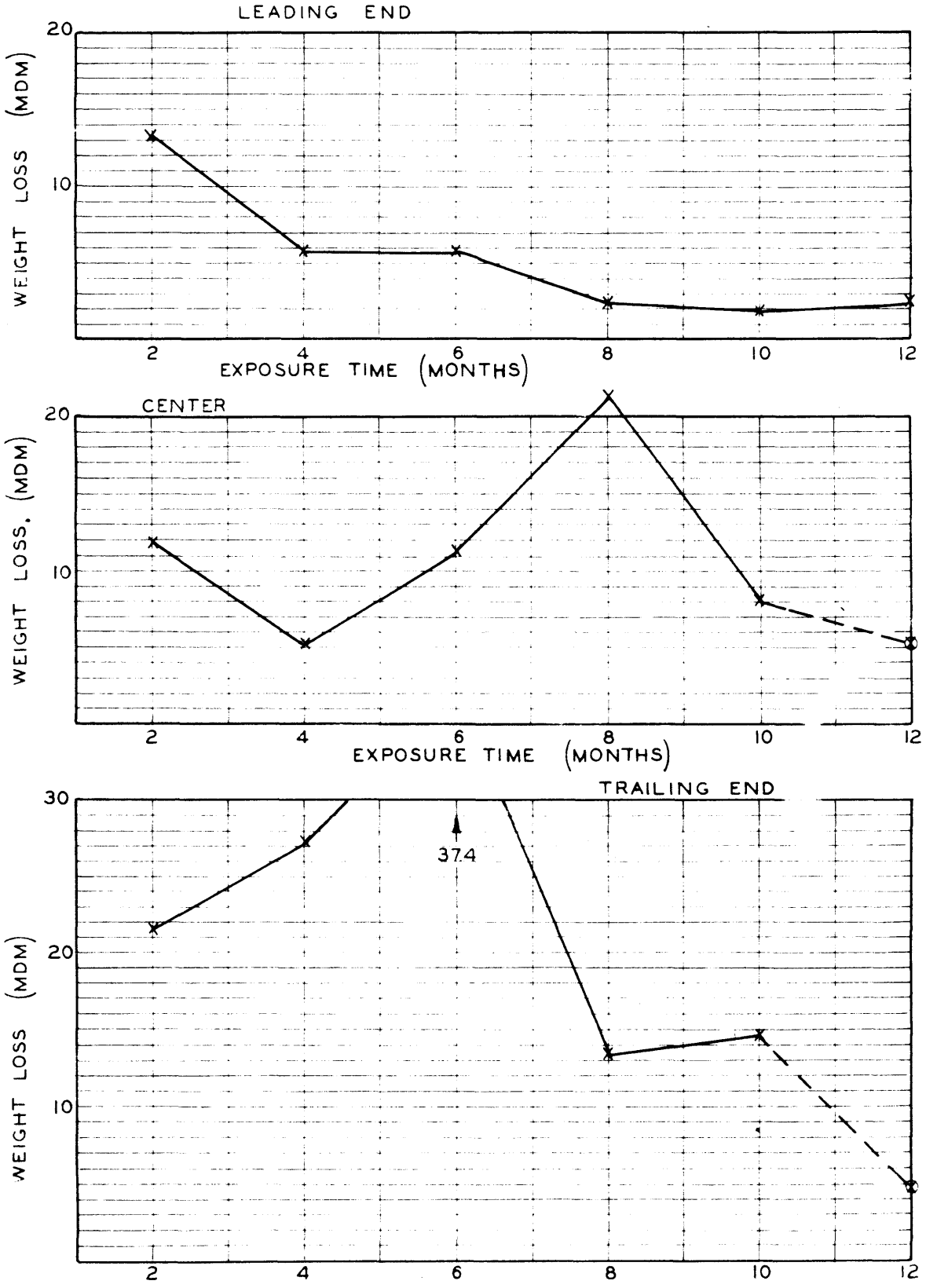
[redacted]

Dwg. 13604

FIGURE 9

CHANGE IN CORROSION RATE WITH TIME FOR EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED ANNEALED CONDITION IN 0.005 M H_2O_2 SOLUTION

TEMPERATURE -- 85°C

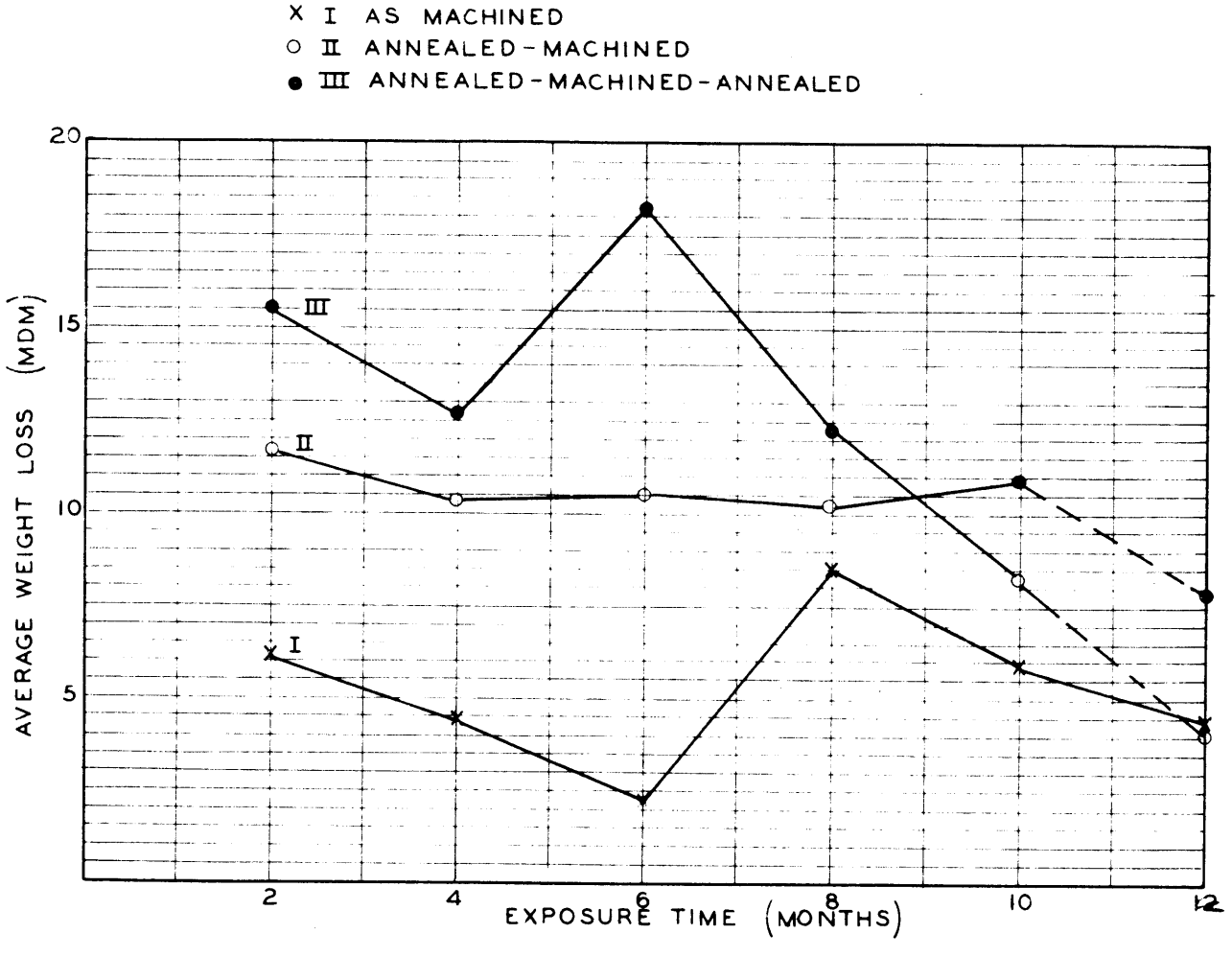


[redacted]

Dwg. 13605

FIGURE 10

COMPARATIVE CORROSION RATES WITH TIME FOR EXTRUDED BERYLLIUM IN 0.005 M H_2O_2 SOLUTIONS

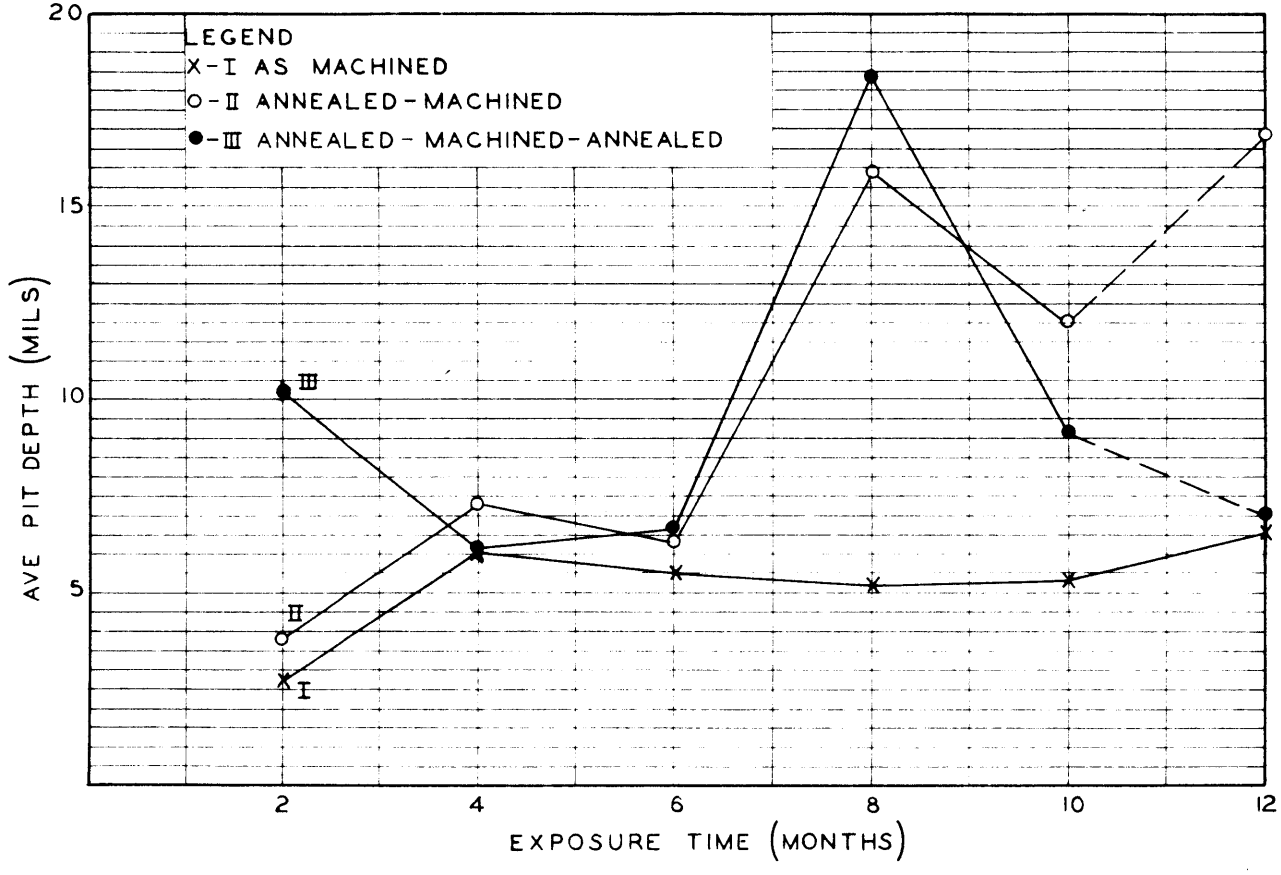


[redacted]

Dwg. 13606

FIGURE II

COMPARATIVE PIT DEPTHS WITH TIME FOR EXTRUDED BERYLLIUM IN 0.005M H_2O_2 SOLUTIONS



UNCLASSIFIED
PHOTO NO. 6-2859

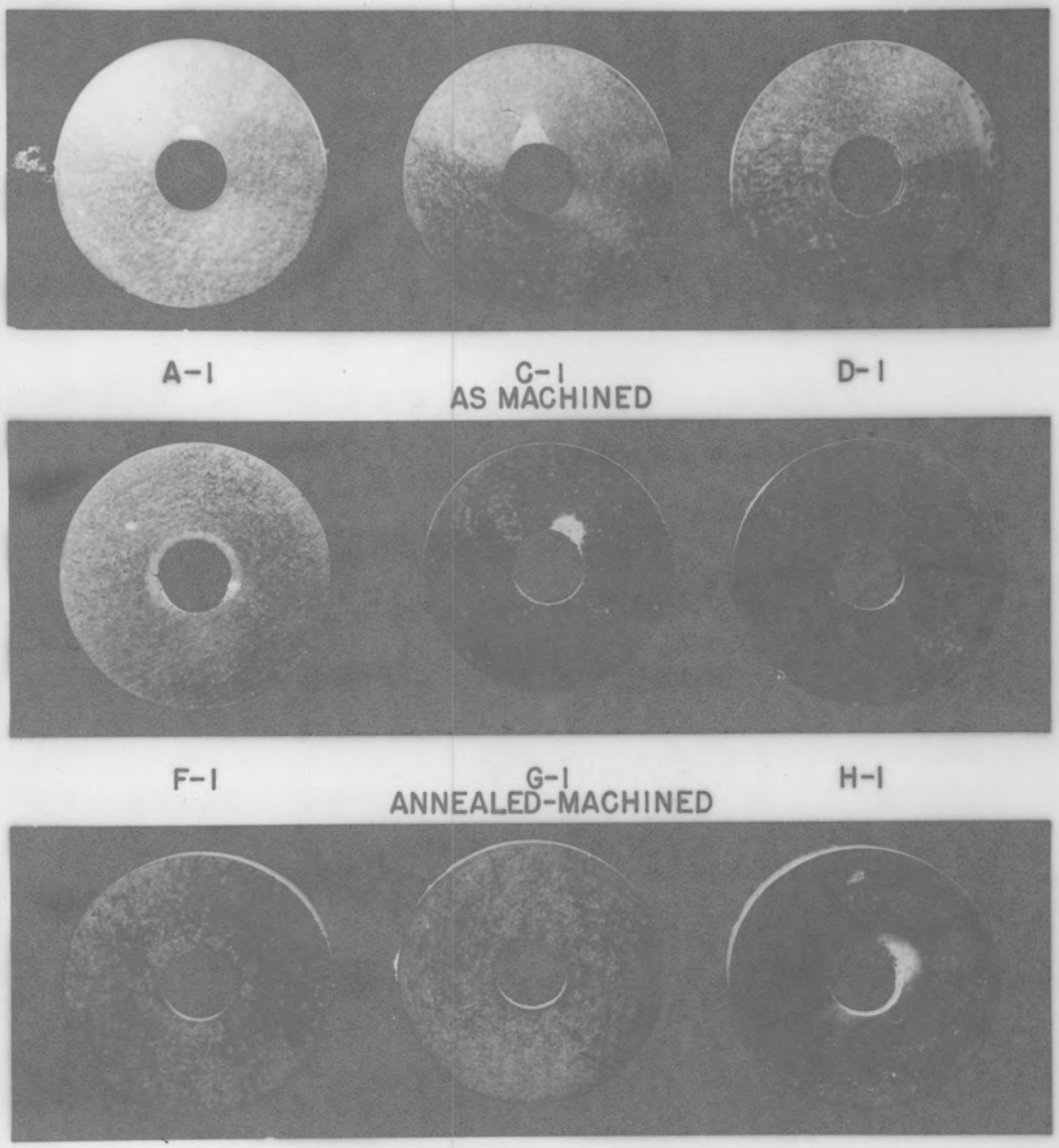
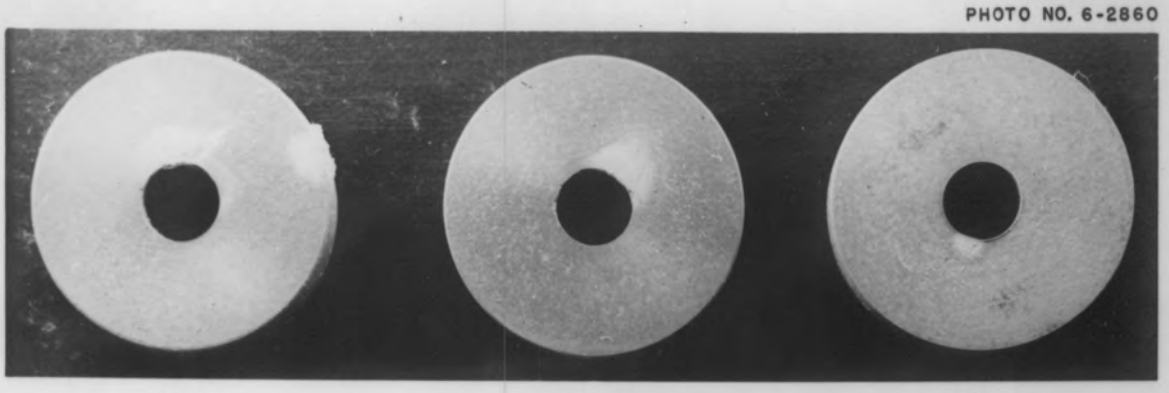
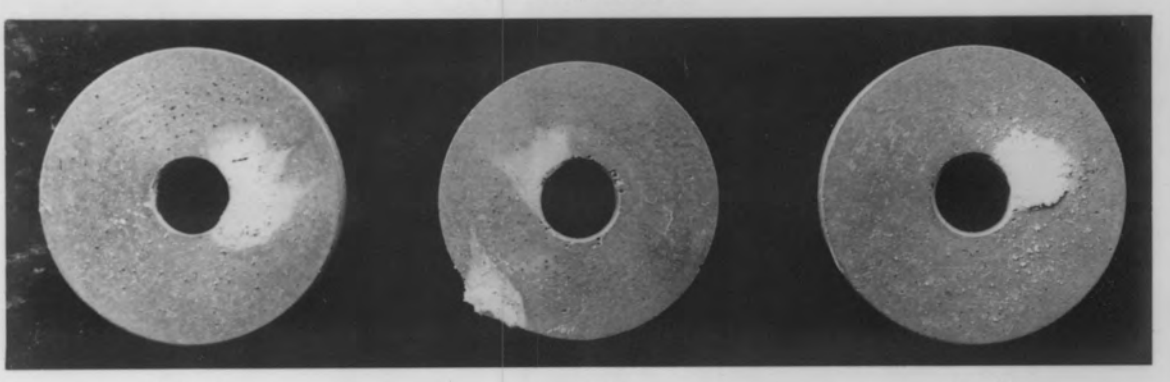


FIGURE 12. PHOTOGRAPHS OF EXTRUDED BERYLLIUM SAMPLES AS REMOVED, AFTER EXPOSURE TO 0.005 M H_2O_2 SOLUTIONS AT 85°C FOR 2 MONTHS (MAG. 1.5x)

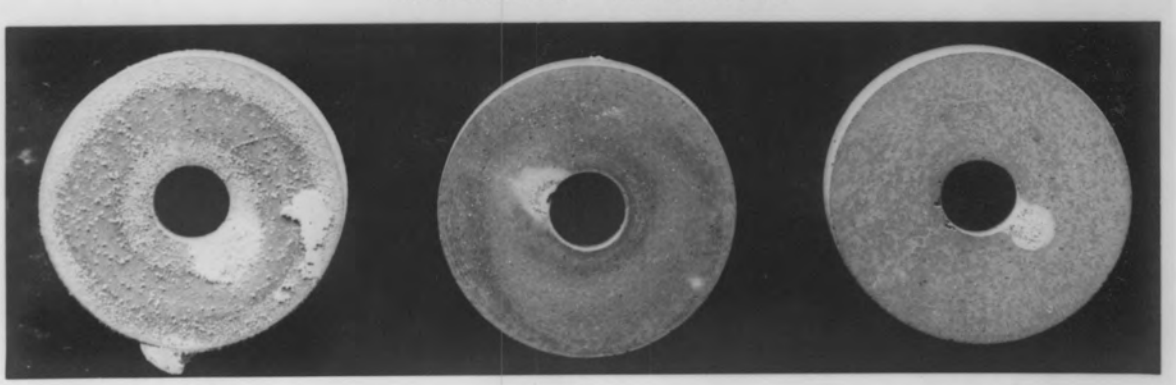
UNCLASSIFIED
PHOTO NO. 6-2860



A-2 C-2 D-2
AS MACHINED



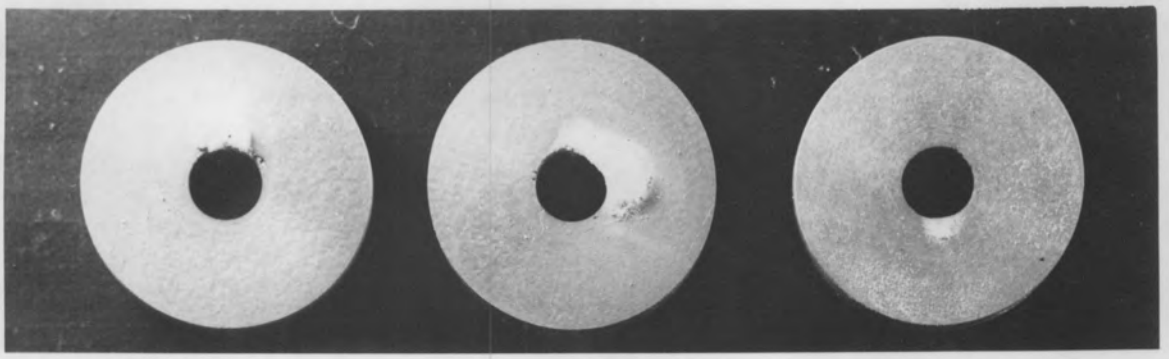
F-2 G-2 H-2
ANNEALED - MACHINED



J-2 K-2 L-2
ANNEALED-MACHINED-ANNEALED

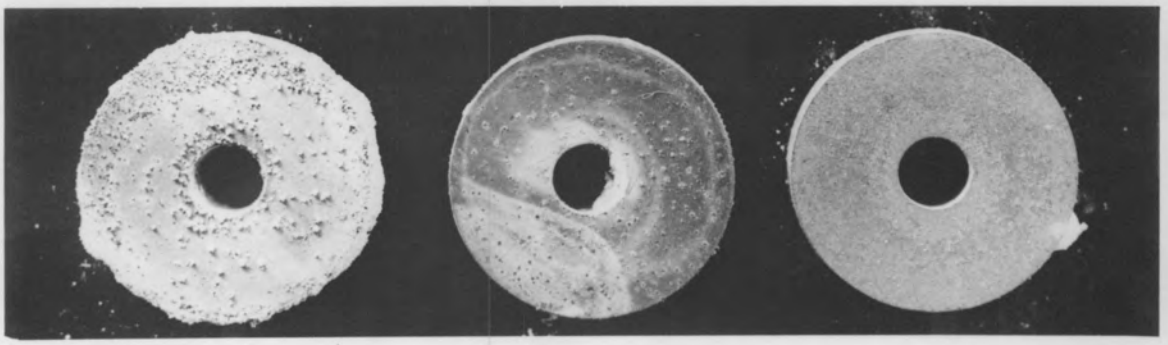
FIGURE 13. PHOTOGRAPHS OF EXTRUDED BERYLLIUM SAMPLES AS REMOVED, AFTER EXPOSURE TO 0.005 M H_2O_2 SOLUTIONS AT 85° C FOR 4 MONTHS. (MAG. 1.5x)

UNCLASSIFIED
PHOTO NO. 6-2861



A-11 C-11 D-11
AS MACHINED

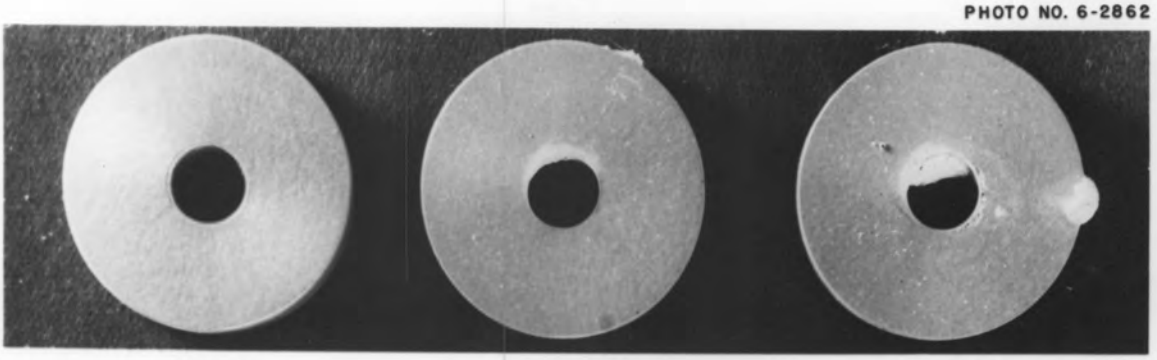
F-11 G-11 H-11
ANNEALED - MACHINED



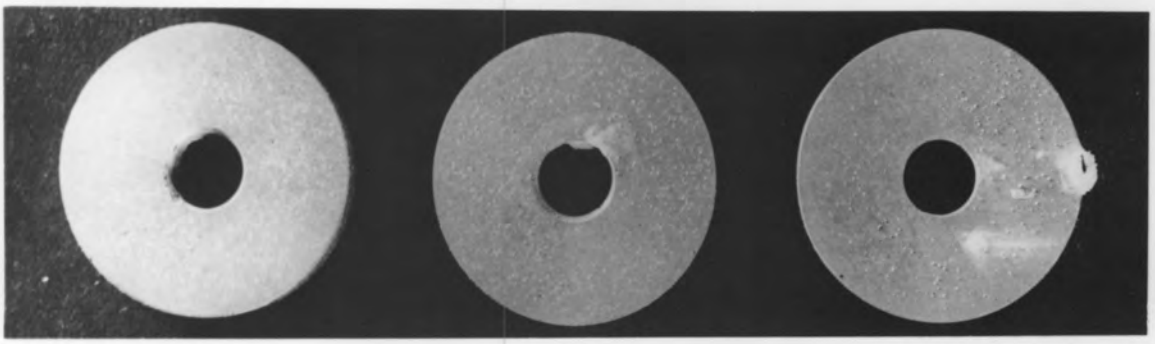
J-11 K-11 L-11
ANNEALED-MACHINED-ANNEALED

FIGURE 14. PHOTOGRAPHS OF EXTRUDED BERYLLIUM SAMPLES AS REMOVED, AFTER EXPOSURE TO 0.005 M H_2O_2 SOLUTIONS AT 85°C FOR 6 MONTHS. (MAG. 1.5x).

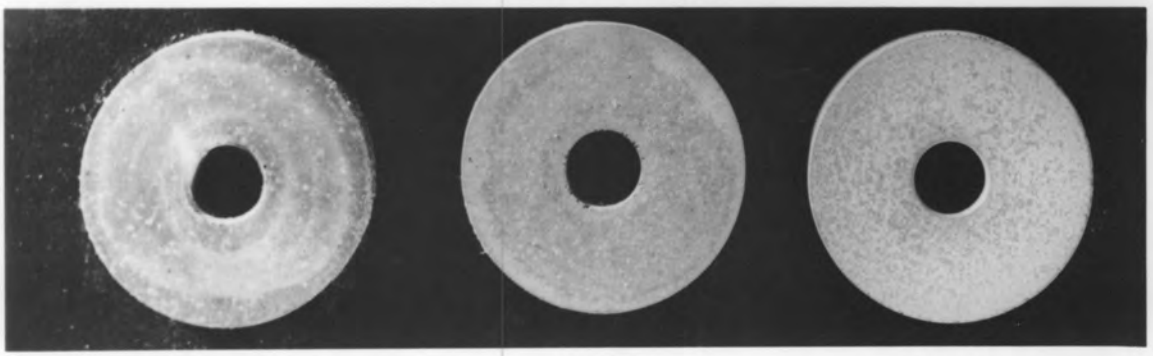
UNCLASSIFIED
PHOTO NO. 6-2862



A-12 C-12 D-12
AS MACHINED



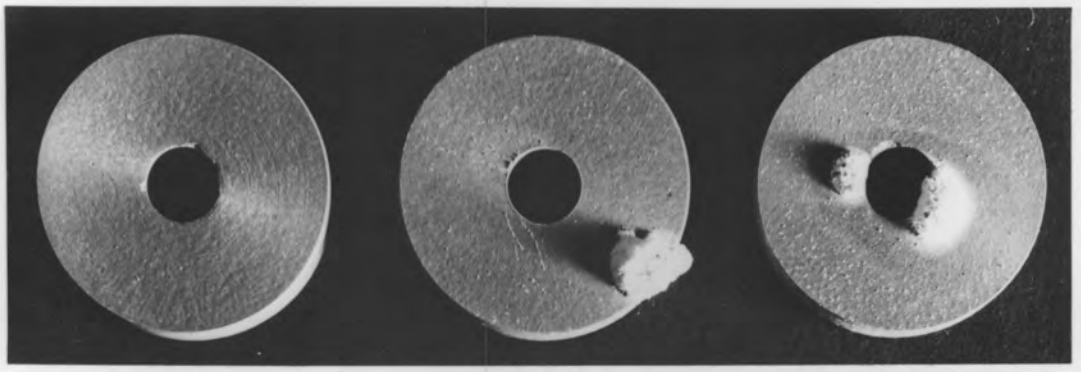
F-12 G-12 H-12
ANNEALED—MACHINED



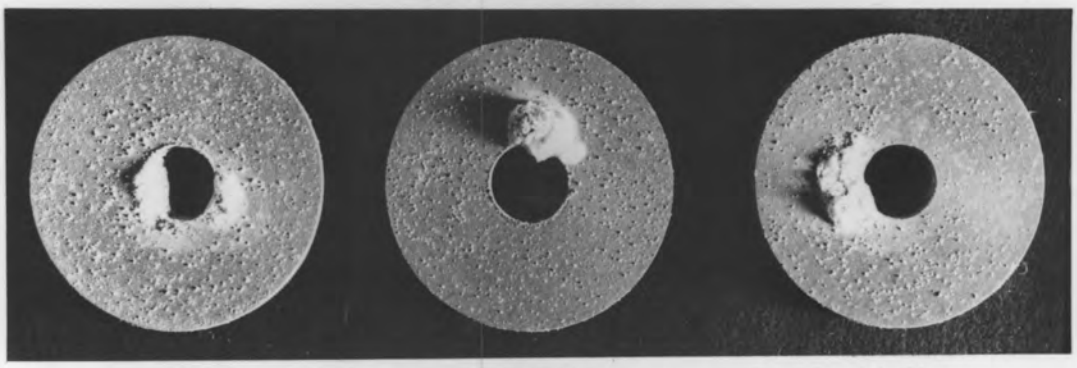
J-12 K-12 L-12
ANNEALED-MACHINED-ANNEALED

FIGURE 15. PHOTOGRAPHS OF EXTRUDED BERYLLIUM SAMPLES AS REMOVED, AFTER EXPOSURE TO 0.005 M H_2O_2 SOLUTIONS AT 85°C FOR 8 MONTHS (MAG 1.5x).

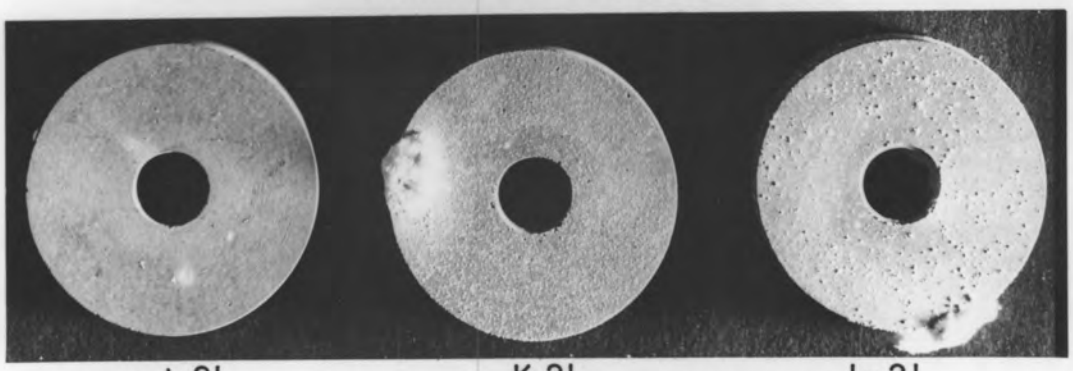
UNCLASSIFIED
PHOTO NO. 6-2863



A-21 C-21 D-21
AS MACHINED



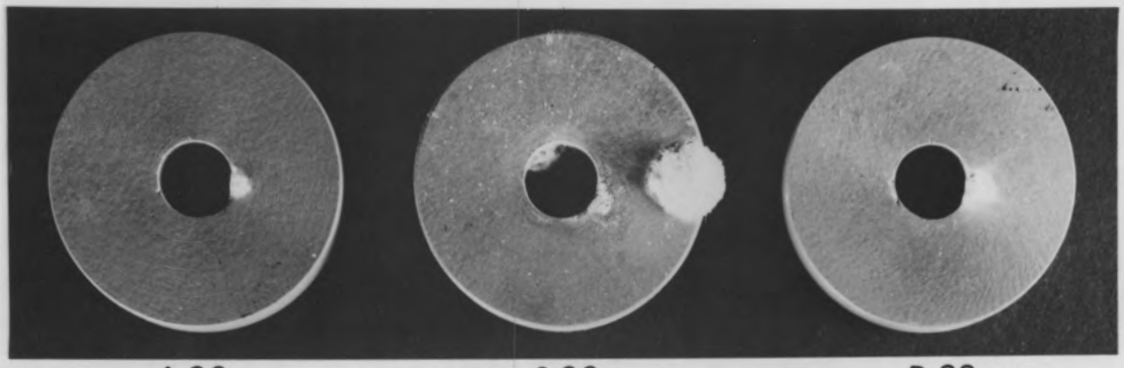
F-21 G-21 H-21
ANNEALED-MACHINED



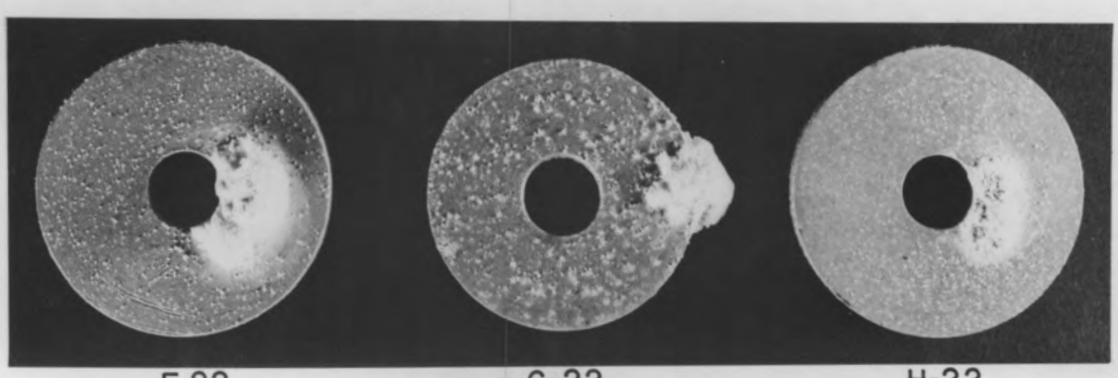
J-21 K-21 L-21
ANNEALED-MACHINED-ANNEALED

FIGURE 16. PHOTOGRAPHS OF EXTRUDED BERYLLIUM SAMPLES AS REMOVED, AFTER EXPOSURE TO 0.005 M H_2O_2 SOLUTIONS AT 85°C FOR 10 MONTHS (MAG. 1.5 x).

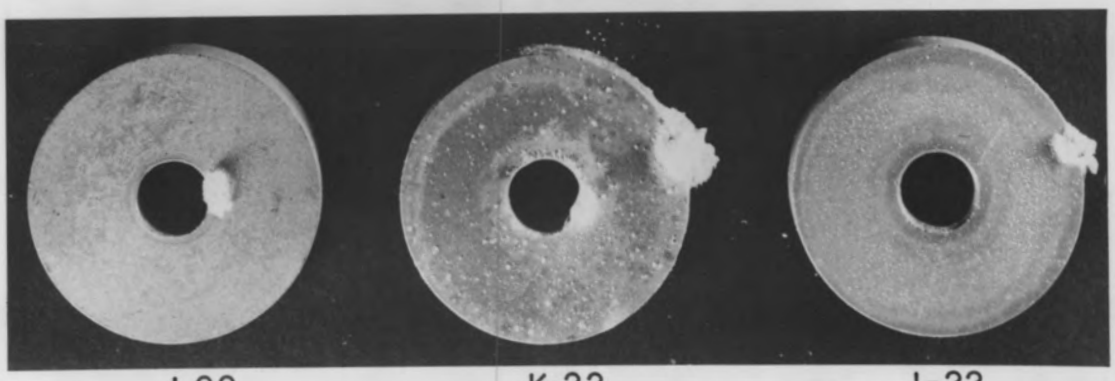
UNCLASSIFIED
PHOTO NO. 6-2864



A-22 C-22 D-22
AS MACHINED



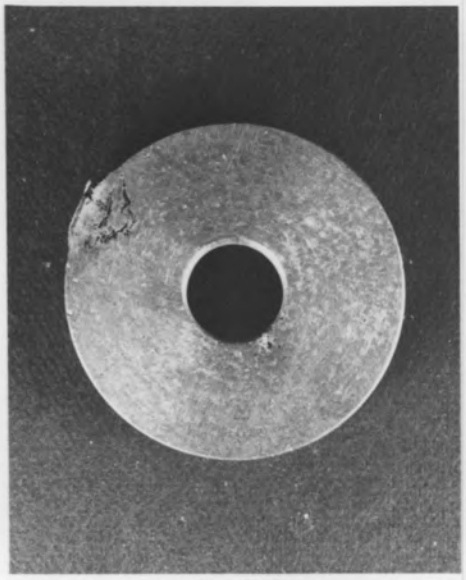
F-22 G-22 H-22
ANNEALED-MACHINED



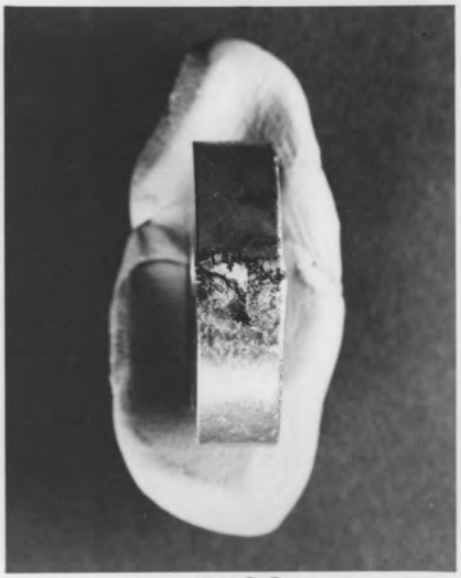
J-22 K-22 L-22
ANNEALED-MACHINED-ANNEALED

FIGURE 17. PHOTOGRAPHS OF EXTRUDED BERYLLIUM SAMPLES AS REMOVED, AFTER EXPOSURE TO 0.005M H_2O_2 SOLUTIONS AT 85°C FOR 12 MONTHS (MAG. 1.5 x).

UNCLASSIFIED
PHOTO NO. 6-2865

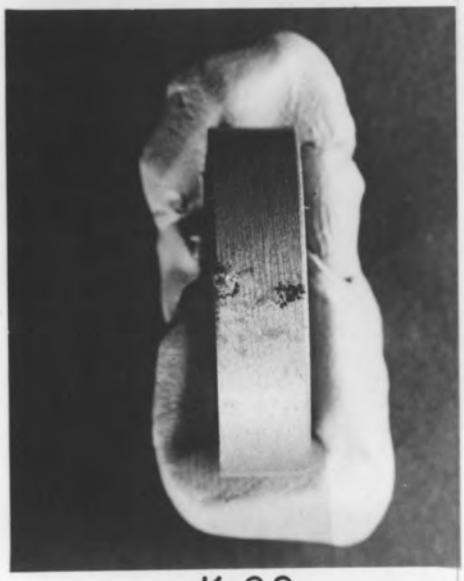


G-22



G-22

ANNEALED-MACHINED



K-22

L-22

ANNEALED-MACHINED-ANNEALED

FIGURE 18. BLISTER FORMATION ON EXTRUDED BERYLLIUM SAMPLES AFTER 12 MONTHS EXPOSURE IN 0.005M H_2O_2 SOLUTIONS AT 85°C. ALL SAMPLES HAVE BEEN DEFILMED.

HIGH TEMPERATURE CORROSION OF EXTRUDED BERYLLIUM AT 515°F AND 840 PSI FOR SEVEN DAYS IN DISTILLED WATER

In hopes of reducing the testing time on all beryllium, the group used the large autoclave shown in Figure 19. The results of duplicate samples of all forms are included in Table 7. While Figures 20, 21, and 22 are photographs of these specimens as removed from the test.

The high rates of weight loss and wide variation in samples clearly indicate that this procedure is not a satisfactory substitute for the standard tests. However such autoclaving is useful in pointing out gross defects in the metal. Although this is not apparent here, it has been shown by other tests and previously reported.

TABLE 7

CORROSION OF EXTRUDED BERYLLIUM IN DISTILLED WATER AT 525°F AND 840 PSI FOR 7 DAYS

Sample	Condition	Weight Loss (MDM)
A-8	I	-717
A-18	I	-152
C-8	I	-827
C-18	I	-552
D-8	I	-439
D-18	I	-357
F-9	II	- 23
F-18	II	-198
G-8	II	-371
G-18	II	-108
H-8	II	- 72
H-18	II	- 53
J-8	III	-171
J-18	III	-254
K-8	III	-306
K-18	III	-367
L-8	III	-290
L-18	III	-533

UNCLASSIFIED
PHOTO NO. 6-2866

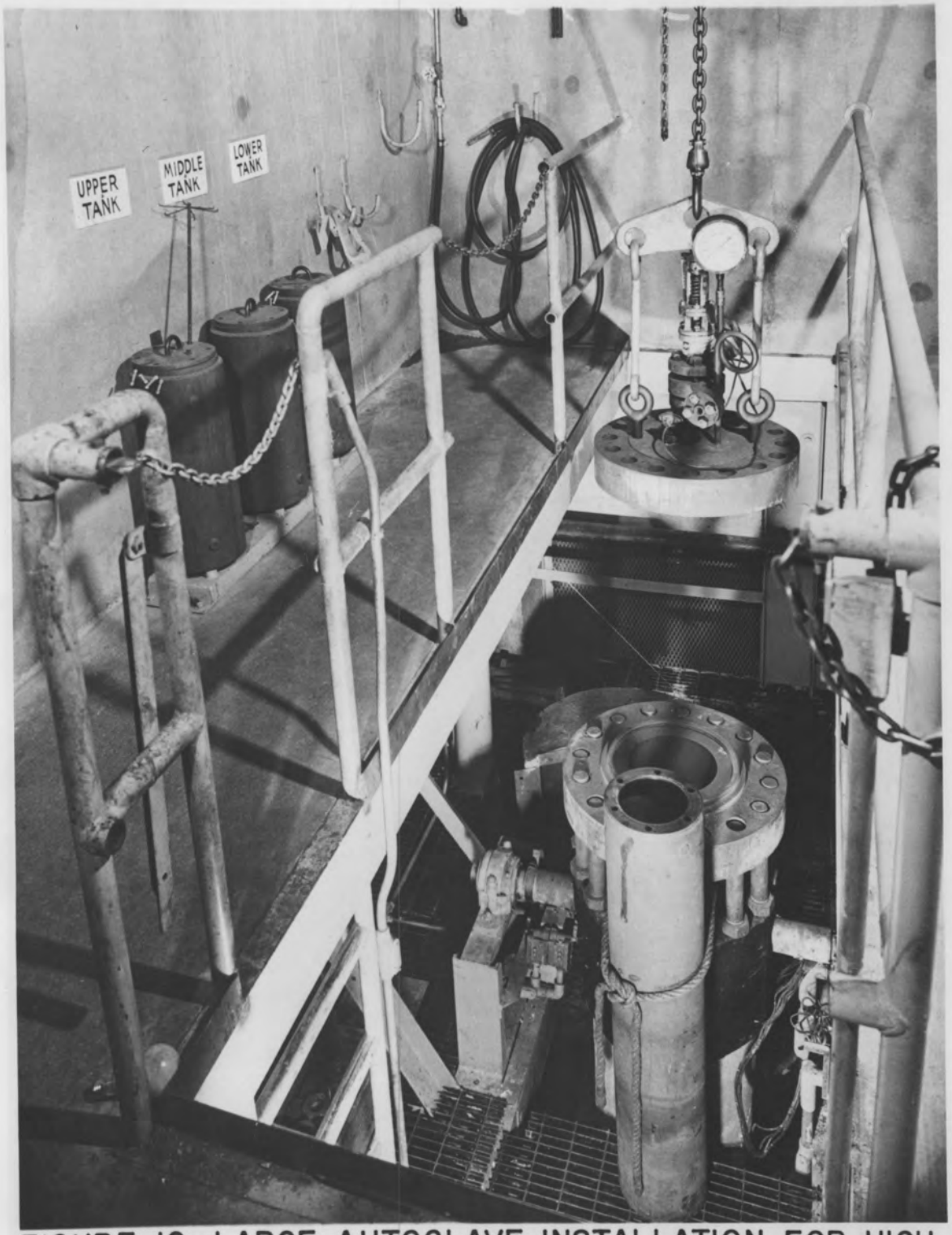
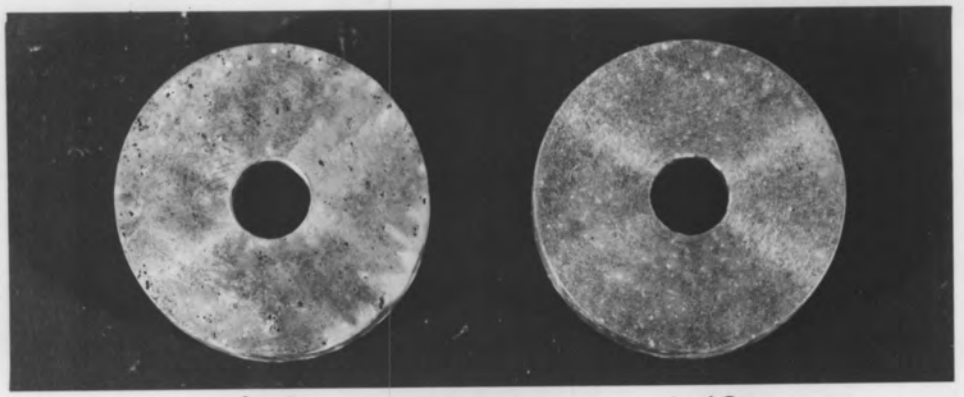


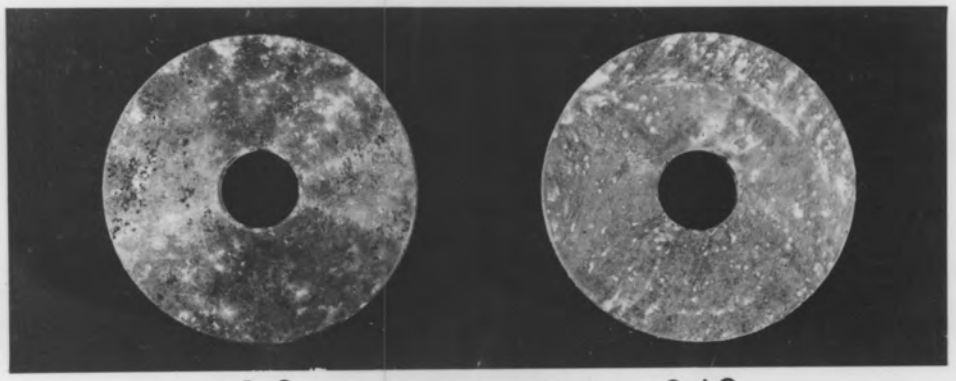
FIGURE 19. LARGE AUTOCLAVE INSTALLATION FOR HIGH TEMPERATURE CORROSION STUDIES. SHOWING AUTOCLAVE AND TWO TYPES OF STAINLESS STEEL LINERS FOR VARIOUS SIZE SAMPLES.

UNCLASSIFIED
PHOTO NO. 6-2867



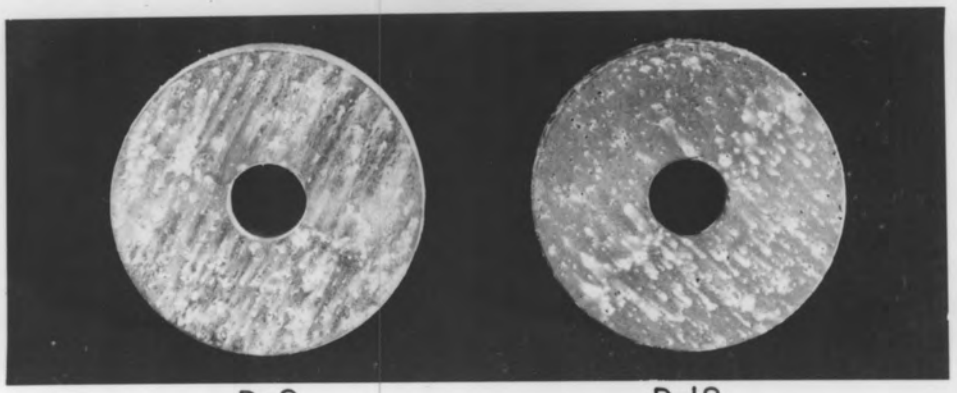
A-8 A-18

LEADING



C-8 C-18

CENTER

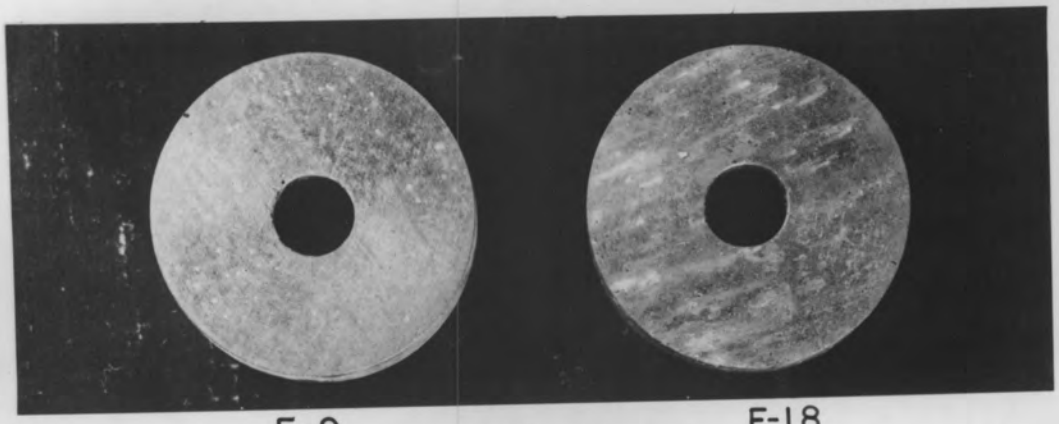


D-8 D-18

TRAILING

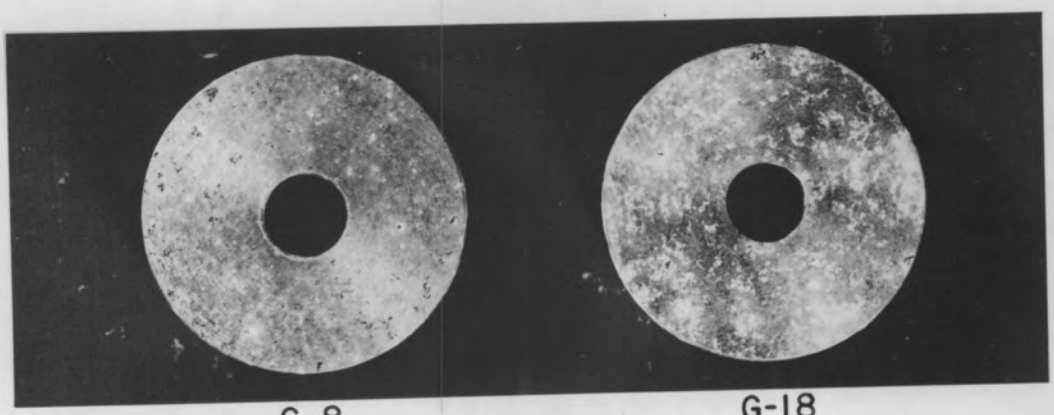
FIGURE 20. PHOTOGRAPHS OF EXTRUDED BERYLLIUM IN THE AS MACHINED CONDITION AFTER SEVEN DAYS EXPOSURE TO DISTILLED WATER AT 525°F AND 840 P.S.I.

UNCLASSIFIED
PHOTO NO. 6-2868



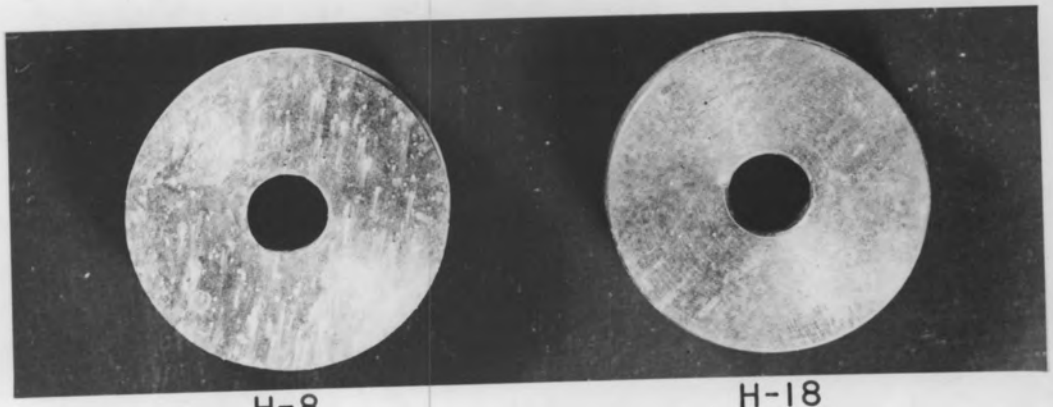
F-9 F-18

LEADING



G-8 G-18

CENTER

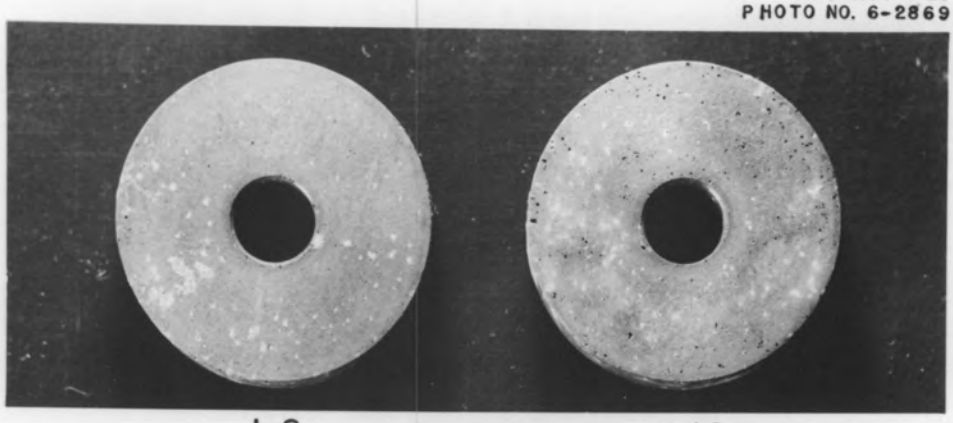


H-8 H-18

TRAILING

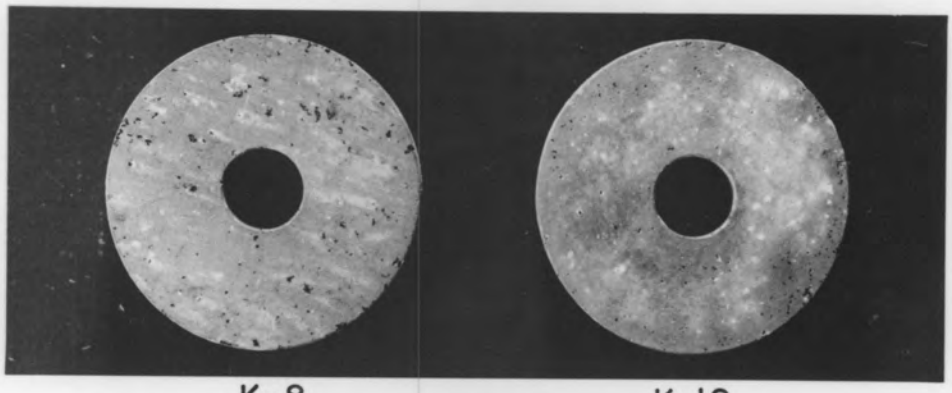
FIGURE 21. PHOTOGRAPHS OF EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED CONDITION AFTER SEVEN DAY EXPOSURE TO DISTILLED WATER AT 525°F AND 840 P.S.I.

UNCLASSIFIED
PHOTO NO. 6-2869



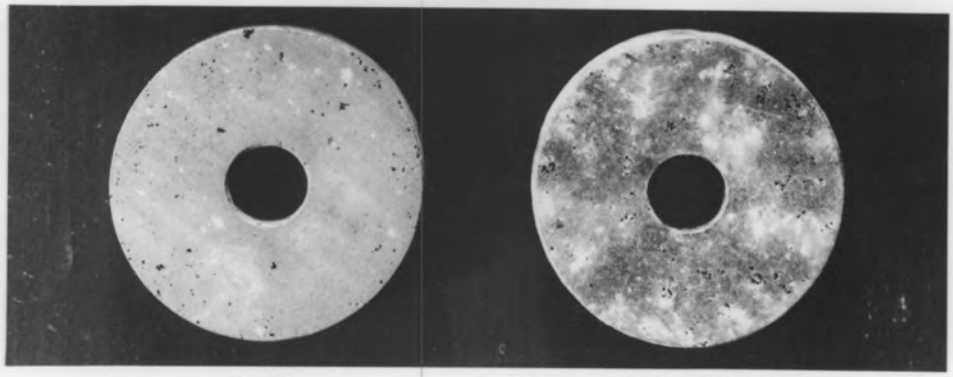
J-8 J-18

LEADING



K-8 K-18

CENTER



L-8 L-18

TRAILING

FIGURE 22. PHOTOGRAPHS OF EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED-ANNEALED CONDITION AFTER SEVEN DAYS EXPOSURE TO DISTILLED WATER AT 525°F AND 840 P.S.I.

CORROSION DATA ON BERYLLIUM SAMPLES COUPLED WITH 356 ALUMINUM FOR 60 DAYS IN 0.0005M H_2O_2 AND 0.005M H_2O_2 SOLUTIONS AT 85°C

The couple tests of the extruded beryllium in various conditions were conducted in Type B apparatus. That is, nine couples were placed in a 16 liter battery jar. The method of testing is important in this case because the results given in Tables 8 and 9 and the photographs in Figures 23, 24, 25 and 26 show clearly the decided effect that environment changes have on the rate of attack of beryllium. Only the 0.005M H_2O_2 tests are shown because of the duplication in appearance of the samples in both peroxide concentrations. Figures 27, 28, and 29 are graphical representations of the data.

The limitation of nine samples per jar led to one sample from each portion of the billet in the annealed-machined condition being in test together with the as-machined samples and the other in test with the annealed-machined-annealed samples. In the latter case, it was necessary to make more additions of nitric acid to maintain the proper pH range with the higher rate of corrosion. This effect was magnified in the case of the 0.005M H_2O_2 solutions as was the overall attack on all samples. The surfaces of annealed samples in the higher peroxide solution were so attacked that no definite pitting frequency could be assigned.

The tests indicate that the corrosion rates for beryllium coupled with 356 aluminum will be much higher than for insulated beryllium and again stress the fact that annealed samples are more susceptible to attack.

[redacted]

TABLE 8

CORROSION OF EXTRUDED BERYLLIUM-356 ALUMINUM COUPLES IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

Sample No.	Material	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
A-3	Be	I	1	4.4	24.3
	Al		0.6	5.4	1.7
A-13	Be	I	0.3	3.5	22.5
	Al		3.0	2.7	1.0
C-3	Be	I	0.3	4.6	22.2
	Al		1.0	9.7	1.3
C-13	Be	I	0.3	3.5	30.7
	Al		1 pit	15.3	5.3
D-3	Be	I	0.3	3.1	19.6
	Al		1 pit	4.3	+0.3
D-13	Be	I	1	2.7	24.0
	Al		.7	14.2	0.33
F-3	Be	II	1	3.0	12.2
	Al		--	-	2.7
F-13	Be	II	1	5.0	20.6
	Al		1.7	5.7	5.3
G-3	Be	II	1	2.8	23.3
	Al		1.3	4.6	0.99
G-13	Be	II	1.6	8.6	98.0
	Al		2.6	20.0	41.2
H-3	Be	II	2.6	6.7	94.9
	Al		0.6	60.0	87.0
H-13	Be	II	2.3	6.8	136.6
	Al		1.2	60.0	85.8
J-3	Be	III	2.0	7.4	157.6
	Al		6.6	60.0	63.1
J-13	Be	III	0.6	9.0	96.1
	Al		8.2	40.0	65.0

[redacted]

TABLE 8 (CONT'D)

CORROSION OF EXTRUDED BERYLLIUM-356 ALUMINUM COUPLES IN 0.005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

Sample No.	Material	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
K-3	Be	III	0.8	8.7	197.0
	Al		4.6	4.5	51.3
K-13	Be	III	0.4	5.7	204.0
	Al		4.4	5.1	29.7
L-3	Be	III	1.3	9.2	171.0
	Al		1.5	40.0	66.8
L-13	Be	III	1	10.5	143.0
	Al		6.7	60.0	89.3

[1] Entire contact area pitted

UNCLASSIFIED
PHOTO NO. 6-2870

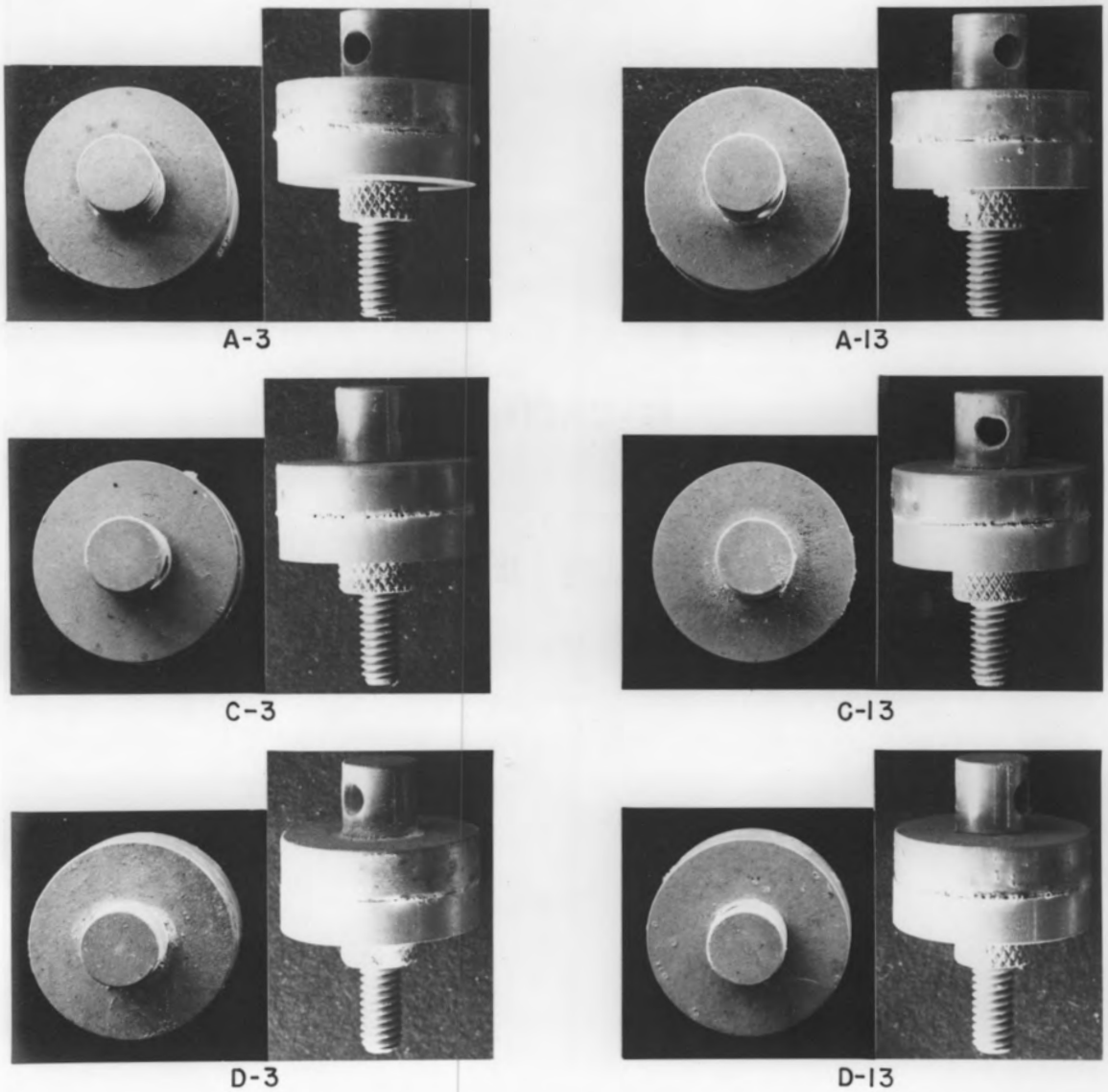


FIGURE 23. CORROSION OF EXTRUDED BERYLLIUM IN THE AS-MACHINED CONDITION COUPLED WITH 356 ALUMINUM EXPOSED 60 DAYS TO 0.005M H_2O_2 SOLUTION AT 85°C.

UNCLASSIFIED
PHOTO NO.6-2871

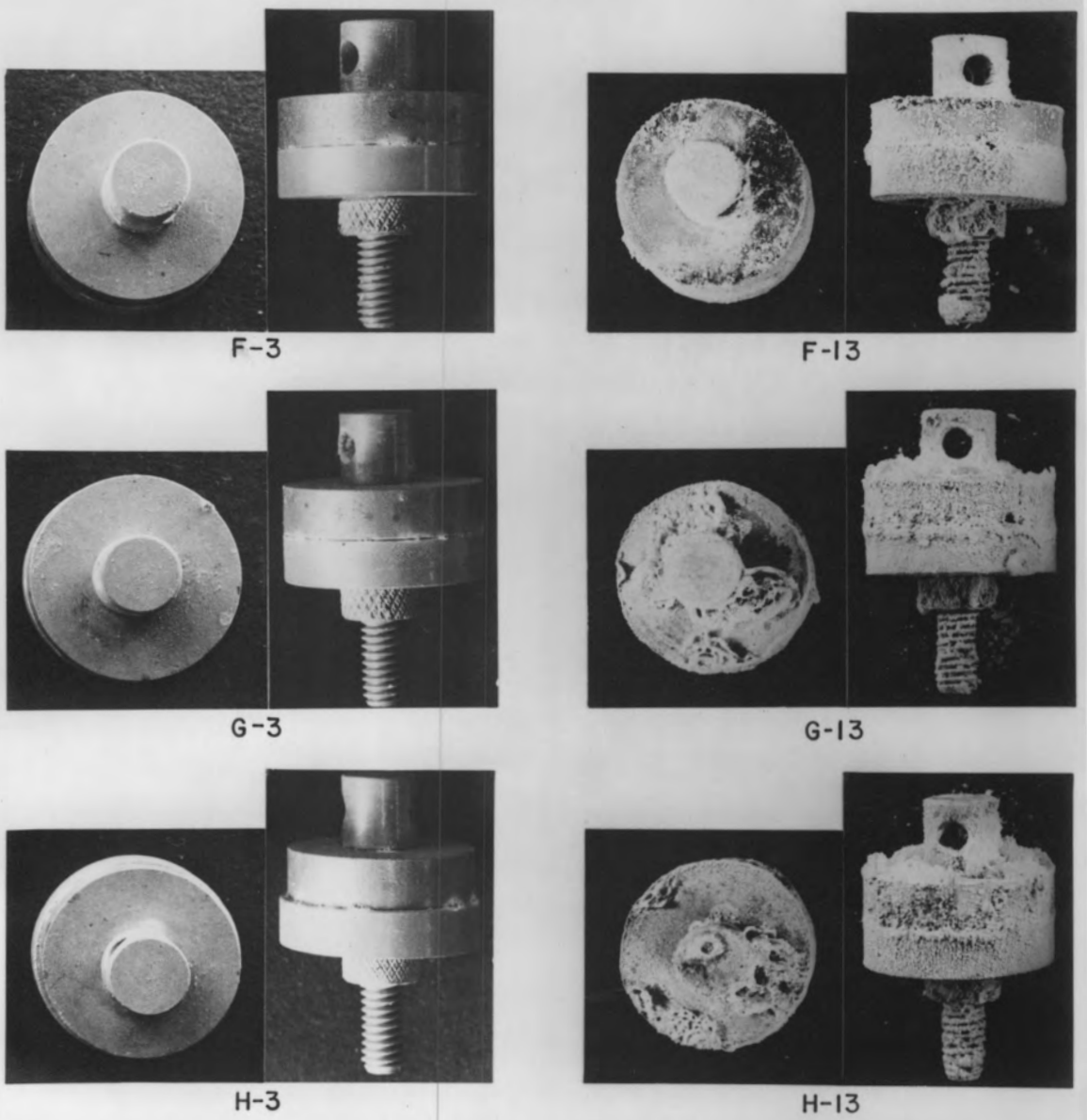


FIGURE 24. CORROSION OF EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED CONDITION COUPLED WITH 356 ALUMINUM EXPOSED 60 DAYS TO 0.005M H_2O_2 SOLUTIONS AT 85°C.

UNCLASSIFIED
PHOTO NO. 6-2872

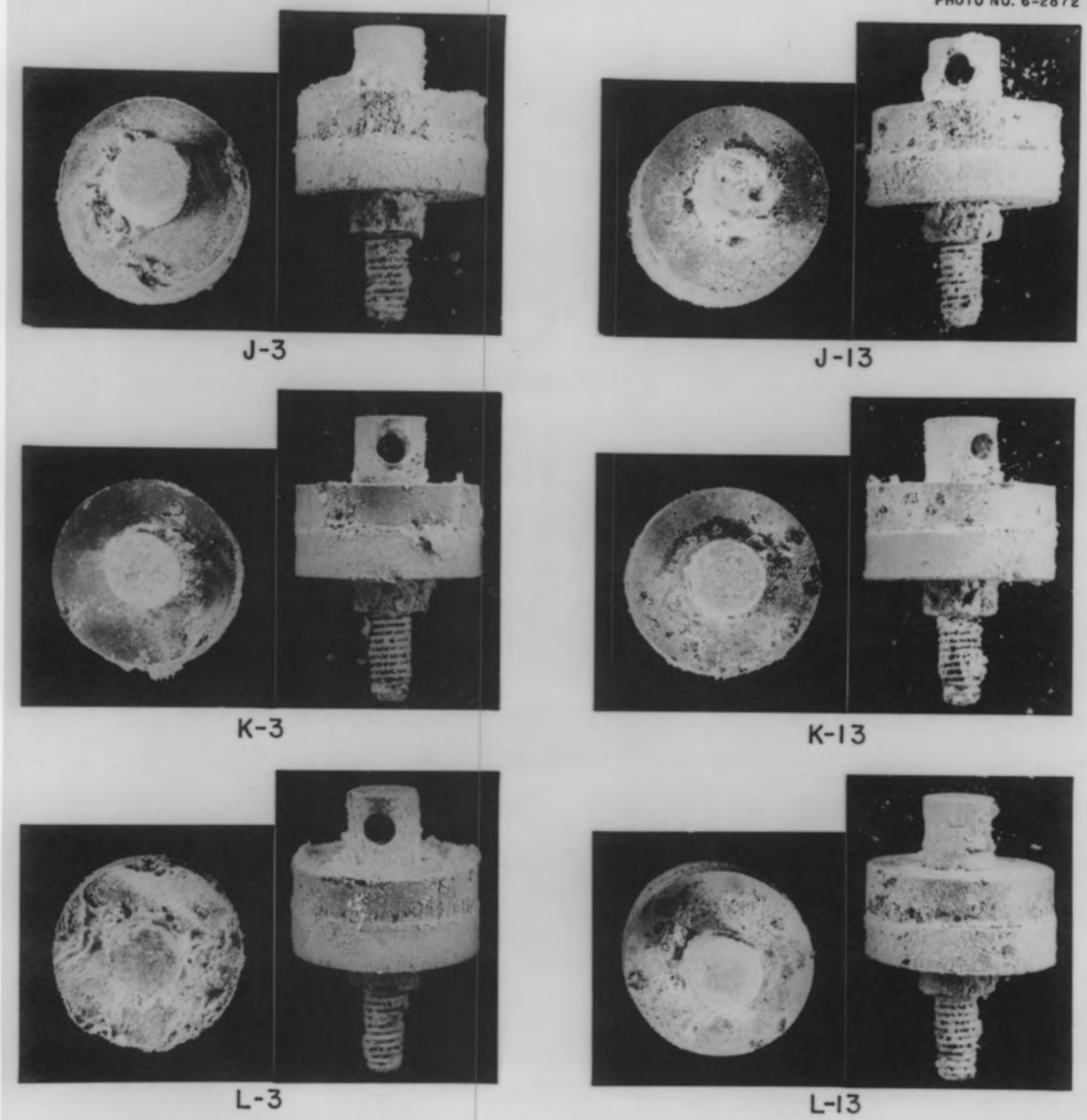


FIGURE 25. CORROSION OF EXTRUDED BERYLLIUM IN THE ANNEALED-MACHINED-ANNEALED CONDITION COUPLED WITH 356 ALUMINUM EXPOSED 60 DAYS TO 0.005M H_2O_2 SOLUTIONS AT 85°C.

UNCLASSIFIED
PHOTO NO. 6-2873

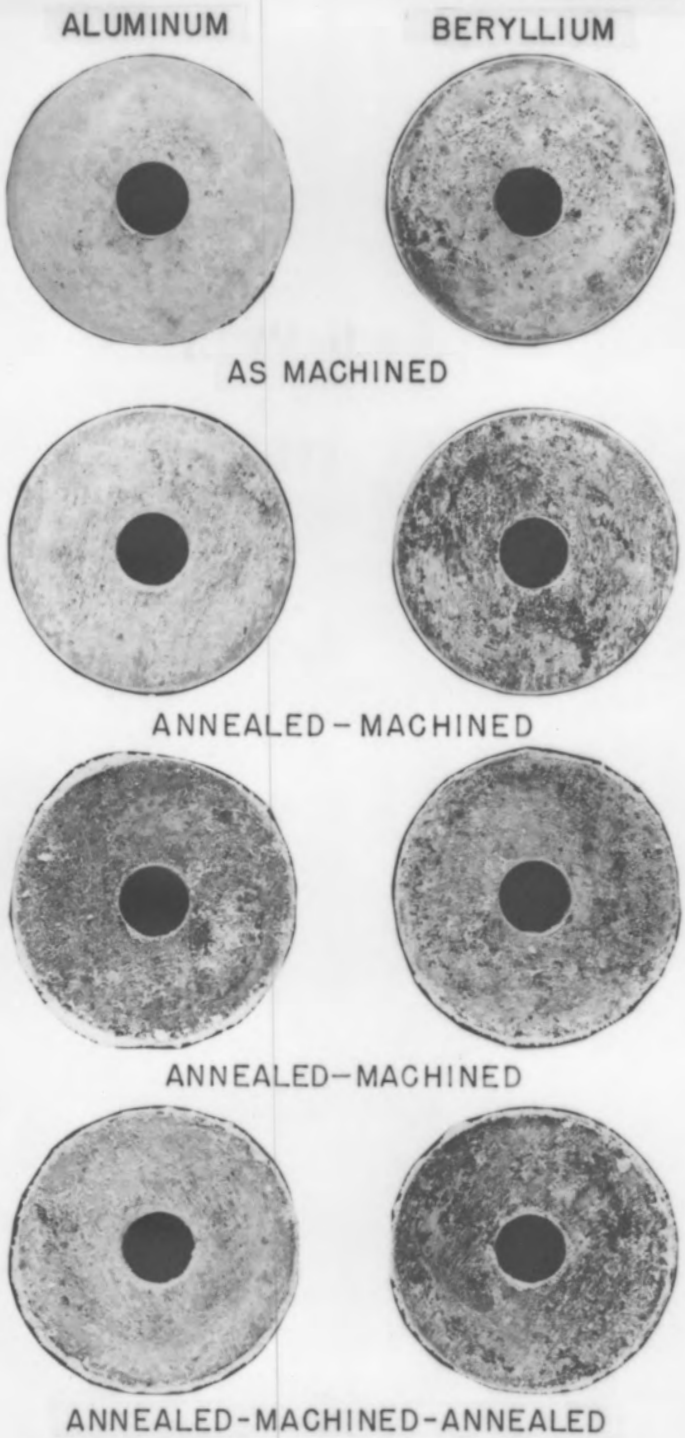


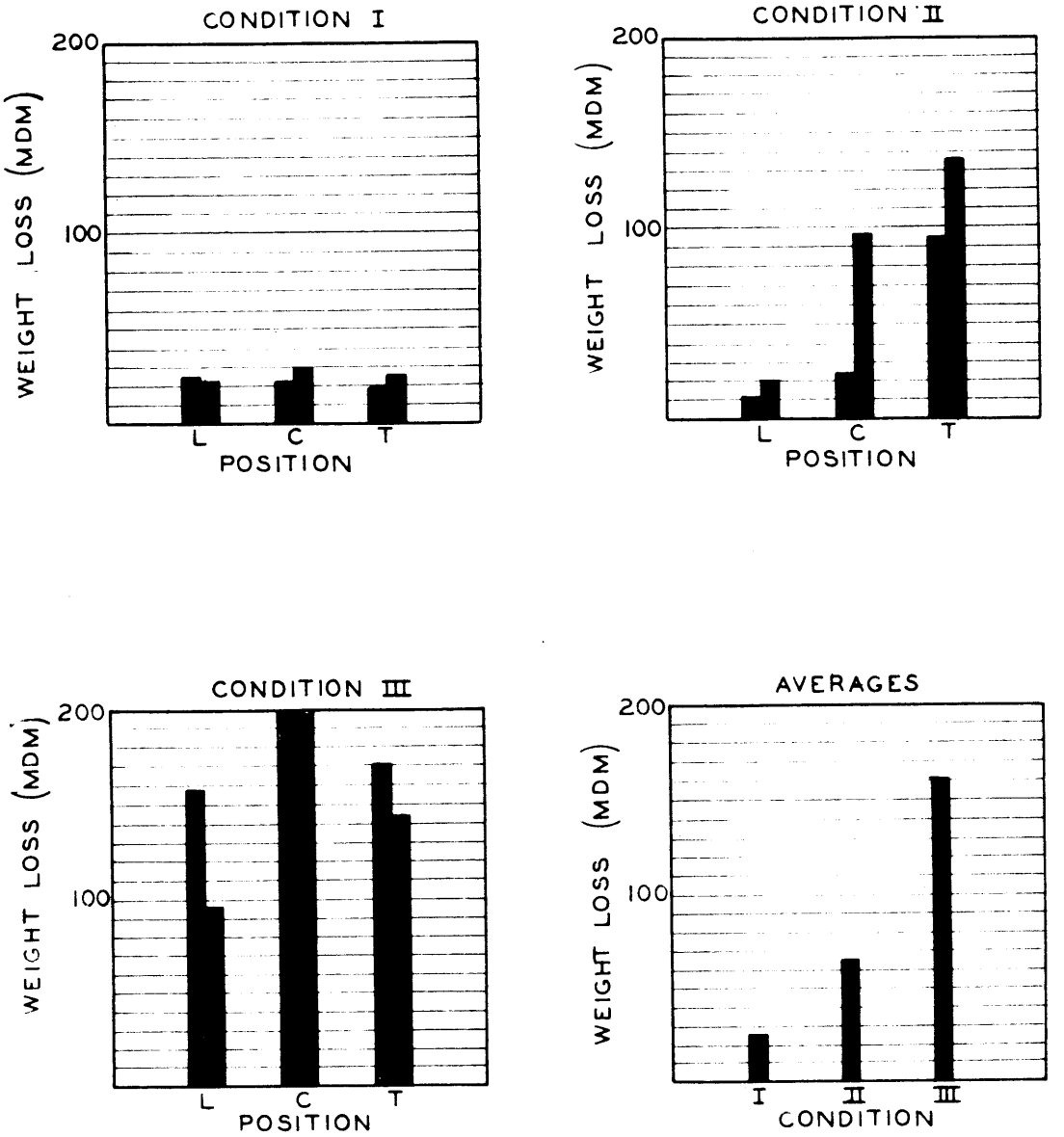
FIGURE 26. EXTRUDED BERYLLIUM-356 ALUMINUM COUPLE INTERFACES AFTER 60 DAYS EXPOSURE TO 0.005 M H_2O_2 SOLUTIONS AT 85° C.

FIGURE 27

[redacted] Dwg. 13607

COMPARATIVE CORROSION RATES OF EXTRUDED BERYLLIUM -356 ALUMINUM COUPLES IN 0.005 M H_2O_2 SOLUTIONS

TEMPERATURE -- 85°C
TIME -------- 1538 HOURS
DUPLICATE SAMPLES



LEGEND
L - LEADING END
C - CENTER
T - TRAILING END
I - AS MACHINED
II - ANNEALED - MACHINED
III - ANNEALED - MACHINED - ANNEALED

TABLE 9

CORROSION OF EXTRUDED BERYLLIUM-356 ALUMINUM COUPLES IN 0.0005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

Sample No.	Material	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
A-5	Be	I	15	1.2	12.2
	Al		20	2.6	14.9
A-15	Be	I	4.5	2.2	10.1
	Al		10.1	2.4	+5.9
C-5	Be	I	3.7	1.6	10.3
	Al		6.5	3.7	14.2
C-15	Be	I	4.5	1.1	9.3
	Al		4.5	1.2	11.0
D-5	Be	I	0.8	1.4	10.4
	Al		5.1	2.9	13.9
D-15	Be	I	2.6	1.0	+6.0
	Al		6.4	3.2	17.2
F-5	Be	II	8.2	2.7	11.1
	Al		6.4	3.3	18.2
F-15	Be	II	5.7	3.2	14.7
	Al		5.5	0.8	13.6
G-5	Be	II	7.5	3.4	21.4
	Al		6.7	1.8	17.2
G-15	Be	II	24.0	2.6	26.5
	Al		14	1.2	4.9
H-5	Be	II	13.6	3.7	28.5
	Al		1	4.8	11.9
H-15	Be	II	14.2	3.3	34.7
	Al		2.5	1.7	9.6
J-5	Be	III	5.0	3.5	18.2
	Al		14.0	3.4	11.2
J-15	Be	III	6.8	4.2	19.0
	Al		7.6	3.0	13.5

TABLE 9 (CONT'D)

CORROSION OF EXTRUDED BERYLLIUM-356 ALUMINUM COUPLES IN 0.0005M H_2O_2 SOLUTIONS AT 85°C FOR 60 DAYS

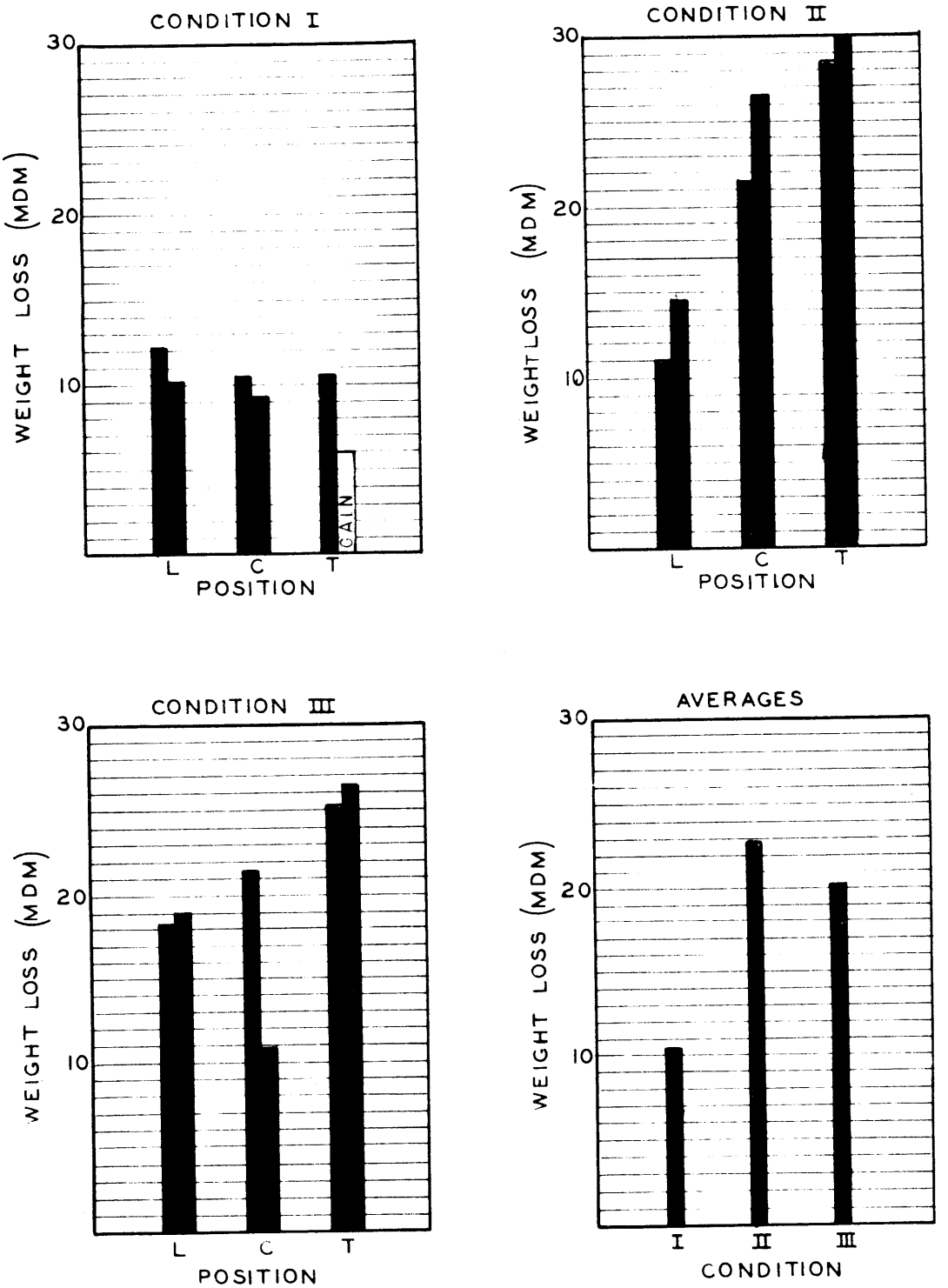
Sample No.	Material	Material Condition	Pit Count per sq cm	Max. Pit Depth (mils)	Weight Loss (MDM)
K-5	Be	III	10.6	4.0	21.4
	Al		7.5	5.2	12.6
K-15	Be	III	4.0	3.8	10.9
	Al		6.1	2.1	10.6
L-5	Be	III	7.6	6.4	25.1
	Al		6.8	2.9	13.3
L-15	Be	III	7.1	3.9	26.2
	Al		5.7	6.0	8.6

[redacted]

FIGURE 28

COMPARATIVE CORROSION RATES OF EXTRUDED BERYLLIUM-356 ALUMINUM COUPLES IN 0.0005 M H_2O_2 SOLUTIONS

TEMPERATURE --85°C
TIME ---------1440 HOURS
DUPLICATE SAMPLES



LEGEND
L-LEADING END
C-CENTER
T-TRAILING END
I-AS MACHINED
II-ANNEALED-MACHINED
III-ANNEALED-MACHINED-ANNEALED

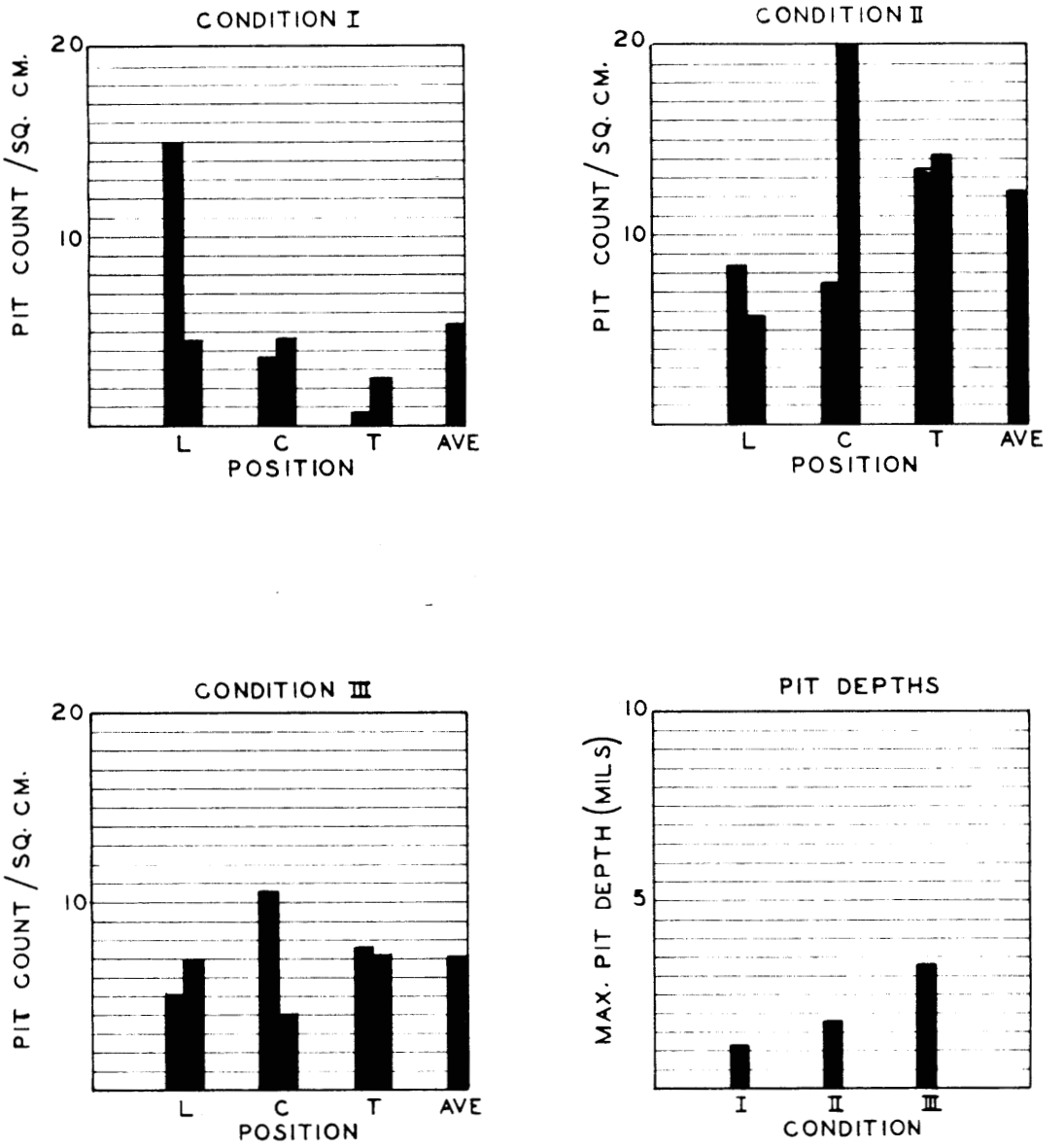
[redacted]

Dwg. [illegible]

FIGURE 29

COMPARATIVE PITTING RATES OF EXTRUDED BERYLLIUM - 356 ALUMINUM COUPLES IN 0.0005 M H_2O_2 SOLUTIONS

TEMPERATURE - 85 C
TIME - 1440 HOURS
DUPLICATE SAMPLES



LEDGEND
L - LEADING END
C - CENTER
T - TRAILING END
I - AS MACHINED
II - ANNEALED - MACHINED
III - ANNEALED - MACHINED - ANNEALED

DISCUSSION OF RESULTS

In general, it was found that for vacuum cast and extruded beryllium, subsequent annealing increased the susceptibility of the metal to corrosion by 85°C demineralized water containing 0.0005 and 0.005 molar hydrogen peroxide.

The results of these tests can be summarized in a few brief statements:

1) With vacuum cast and extruded beryllium there is no definite variation in corrosion characteristics from the leading to the trailing end of the extruded billet.

2) An increase in hydrogen peroxide concentration from 0.0005M to 0.005M will markedly decrease pit frequency and increase overall corrosion attack.

3) Removal of the surface layer of a sample, machined in the as-extruded condition by chemical etching, will activate this metal corrosion-wise. This is based on a 60-day exposure only.

4) Comparison of metal in the as-extruded condition and subsequently annealed material shows a much higher initial rate of corrosion for the latter. Tests extended to a 12-month period, however, show both rates to be comparable within the spread of attack on samples identically treated.

5) High temperature, short time autocalve tests (516°C at 840 psi for seven days) are not reliable in predicting the corrosion behavior of beryllium in demineralized water at 85°C for one year. The attack is so severe that it totally masks variations in the quality of the metal.

6) Susceptibility of beryllium to the formation of blisters in a year's exposure is increased in direct proportion to the number of anneals the metal receives.

7) Beryllium coupled directly with Type 356 aluminum in demineralized water containing hydrogen peroxide is more rapidly attacked than separately exposed metal. The rate of corrosion attack increase with increased H_2O_2 concentration. In addition, it was found that coupled samples which started to accumulate corrosion products or which were in the same solution as rapidly corroding beryllium samples, corroded at a rapid rate.

   Under the conditions of these tests, this may be explained as a combination effect. It has previously been shown that the rate of corrosion attack is higher in water with a pH above 6.5.

In these tests the accumulation of beryllium hydroxide tended to raise the pH above the specified limit, requiring the addition of more than the usual amount of dilute nitric acid. These additions resulted in a decrease in the specific resistance of the solution which, in turn, accentuated the couple action. Thus the poor material continued to corrode rapidly and the more resistant material, subject to an increased couple action, also corroded rapidly.

ACKNOWLEDGMENTS

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