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HIGH-TEMPERATURE BERYLLIUM
CORROSION PROTECTION

Phase Report
Evaluation of Coatings
Anodized by Brush Beryllium Corporation

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for

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North American Aviation, Inc.
8900 DeSoto Avenue
Canoga Park, California
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ABSTRACT

The use of anodizing as a means of protecting S8ER beryllium neutron reflectors from atmospheric corrosion during ground testing has been evaluated. Anodic coatings applied by the Brush Beryllium Company were oxidized at varying temperatures in dried air and reduced oxygen content atmospheres to determine coating life. The results indicate that a coating life of at least 3000-4000 hours at 1330°F can be anticipated in an inert atmosphere containing 100 ppm oxygen.

Subsequent work to improve anodic processing with respect to the elimination of defects at contact points and inside of holes was carried out. Finally, qualification specimens resembling the actual reflector parts were anodized and evaluated.

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HIGH-TEMPERATURE BERYLLIUM CORROSION PROTECTION

Phase Report

Evaluation of Coatings

Anodized by Brush Beryllium Corporation

I. INTRODUCTION

This report summarizes our work in evaluating coatings of anodized beryllium applied by the Brush Beryllium Corporation. These coatings have been applied to small test coupons for the purpose of evaluating the reliability of the process and the influence of processing modifications. The coatings have also been applied to simulated reflector parts and evaluated.

The decision to use the Brush Beryllium Corporation anodizing process as a means of protecting the S8ER beryllium neutron reflectors from oxidation during ground testing was made at a meeting at Atomics International on May 11, 1962, involving J. J. Rausch of ARF and Messrs. R. B. Hinze, T. S. Nakae, J. W. Ramsey, G. Erwin, Jr., and W. Netter of Atomics International. This decision was based upon the performance of this coating in previous tests at the Foundation, particularly those conducted during April, 1962, the results of which were reported in ARF-2229-11, dated June 18, 1962. These results indicated that the anodic coating was capable of providing extensive protection, based on our screening criteria; however, problems associated with reliability of the process, the ability to coat holes and recesses, and avoiding defects which occurred in contact regions during processing, remained to be overcome.

II. INITIAL OXIDATION TESTS

As an initial step, twelve samples which were cylinders 3/4-in. diameter x 1-in. long, having a 1/4-in. hole and a 1/2-in. recess along the cylindrical axis, were supplied to Brush for anodizing.

The anodized samples were received from Brush in batches of six. Oxidation tests were performed in laboratory air at $1452^{\circ} \pm 3^{\circ}\text{F}$ with the samples contained in preoxidized stainless steel trays. The initial batch of six specimens was examined after 70, 86, 103, 119 and 136.5 hours. Two had failed at the base of the recess between the 1/2 in. - and 1/4-in.

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diameter hole after 70 hours. Two others showed similar defects after 103 and 119 hours, respectively. The remaining two samples had failed in this region after 136.5 hours.

The second batch of six samples was identified with numbers 1 to 6. The test results appear in Table I. All of the specimens had a uniform dark color, as received; however, Specimens 4-6 were considerably more shiny than Specimens 1-3. Specimens 2-5, in which the outside contacts only were stripped, contained large bright areas of apparently bare metal at the points where contact was made during anodizing. This was on the outer cylindrical surface. In Specimens 1 and 6 these defects were also present but were smaller. None of the specimens were coated completely on the ledge between the 1/4-in. diameter and 1/2-in. diameter holes. Specimens 4 and 5 appeared to have the best coating in this area. During oxidation testing Specimen 6 failed very seriously after 46 hours, the first time at which the specimens were examined. Failure occurred in the 1/4-in. hole and on the ledge between this hole and the 1/2-in. hole. The failure in the small hole had progressed almost to the point of forming sufficient BeO to crack the specimen. Failures in the other samples were localized and were generally confined to the contact points either on the internal or external cylindrical surfaces. Color changes which occurred in the specimens during oxidation were from black originally, through dark green, to a light green. The light green color was observed initially at the ends of the specimens. The darker coloring was most pronounced on the external cylindrical surfaces of the specimens except in the contact regions. Weight change measurements were made during oxidation. However, these show no consistent variation and cannot be directly related to the inception of failure.

Six anodized samples that were previously tested and stopped after the first sign of coating failure were retested to determine the total life of the remaining coating. The specimens selected, along with their prior oxidation history, are given in Table II. Five of the specimens were from the twelve described above. The other was a solid cylindrical sample which had been received previously and had been oxidized for 34.5 hours at 1450°F. In addition one sample that had been oxidized in dried air for 141.5 hours was also included in this evaluation. Continued exposure of this

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TABLE I

OXIDATION TEST EVALUATION

OF DOUBLE-HOLED CYLINDERS

RECEIVED FROM BRUSH BERYLLIUM CORPORATION

Specimen No.	Additional Comments	Time of First Appearance of Failure, hr	Remarks
1	Inside contacts only were stripped	120	Very small defect started to develop at outside contact point.
2	Outside contacts only were stripped	64	Failed at inside contact point.
3	Outside contacts only were stripped	46	Same as 2
4	Outside contacts only were stripped	64	Same as 2
5	Outside contacts only were stripped	104	Failed at outside contact point.
6	Inside and outside contacts not stripped	46	Failed at outside contact point. The most serious failure, however, occurred at the small-diameter end of the specimen. Oxidation within the hole in this region had progressed nearly to the point of cracking the specimen.

TABLE II
OXIDATION TEST HISTORY OF ANODIZED SPECIMENS
BEFORE RETEST AT 1450°F IN LABORATORY AIR

Specimen No.	Specimen Type	Prior Oxidation Time at 1450°F in Laboratory Air, hr	Condition of Specimens Prior to Retesting
A	Solid cylinder	34.5	Small defect as one end in contact region
B	Double-hole cylinder, No. 4 in Table I	64	Small defect at inside contact point
C	Double-hole cylinder, No. 3 in Table I	46	Same as B
D	Double-hole cylinder, No. 1 in Table I	120	Very small defect at outside contact point
E	Double-hole cylinder, one of the first 6 received from Brush	136.5	Small defect at inside contact point
F	Same as E	136.5	Same as E
G	Solid cylinder	141.5	Slight oxidation at one end in contact region; this sample was tested in dried air

sample was also in dried air. The results of retesting are presented in Table III.

The sample tested in dried air was still in good condition after a total of 367 hours of exposure. One sample of the original twelve double-holed cylinders received from Brush remained in good condition, except for some oxidation of the ledge between the 1/4-in. diameter and 1/2-in. diameter holes, after 372 hours of exposure. The remaining samples failed by general oxidation after total times varying between 89.5 and 301.5 hours, except for Specimens B and C. General failure occurred at some time considerably longer than that at which the first evidence of failure appeared.

III. DEVELOPMENT OF IMPROVED ANODIZED COATINGS

These test results indicated that the anodized coating, if properly applied, was capable of affording more than adequate oxidation protection for beryllium. Although there was some variation in the general protective capability of the coating, the most serious problem appeared to be that of protecting the areas in which electrical contact was made during anodizing. In an attempt to overcome this problem, specimens were made in which electrical contact was made in a threaded hole which was subsequently drilled out and closed with a "shrink-fit" anodized beryllium plug. The samples were 1 1/4-in. cubes having hole configurations similar to those in the twelve samples previously tested. A photograph showing three of these samples prior to testing appears in Figure 1.

The anodized cubes were tested at 1450°F in laboratory air. Preliminary test results appear in Table IV. All of the samples were very seriously oxidized after exposure for 16-39 hours. Photographs showing the condition of some of these specimens after oxidation appear in Figures 2-4. It may be noted that failure of the coating occurred in all regions. The initial failure in some cases occurred in the recess area or in the region where the shrink-fit plug was inserted.

The very poor performance of this batch of six specimens presented an additional problem which Brush would have to overcome--namely, the coating of large and complicated shapes--if their anodizing process were

TABLE III
OXIDATION RETESTING
OF SPECIMENS LISTED IN TABLE II

Specimen No.	Total Oxidation Time, hr	Remarks
A	216.5	General oxidation has occurred on all faces.
B	107.5	Seriously oxidized on inside diameter. The oxidation apparently started at the inside contact points. Also has oxidized in a general manner on outside diameter.
C	89.5	Same as B.
D	301.5	General oxidation has occurred on all faces.
E	235.5	Seriously oxidized on inside diameter causing cylinder to crack.
F	372	Specimen shows slight oxidation on ledge between 1/4-in. diameter and 1/2-in. diameter holes.
G	367	Slight increase in the amount of oxidation which has occurred at one end of the cylinder. This sample was tested in dried air.

TABLE IV

TEST RESULTS FOR ANODIZED BERYLLIUM CUBES
OXIDIZED IN LABORATORY AIR AT 1450°F

Specimen No.	Time, hr.	Remarks
1 C	16	Slight oxidation in recess.
2 C	16	Completely oxidized.
5 C	16	Slight oxidation in recess.
A C	34	Completely oxidized. After 18.5 hours there was slight oxidation apparent in recess.
B C	18.5	Completely oxidized.
C C	18.5	Slight oxidation in recess.

to be used for coating of reflectors. Furthermore, the poor performance of these specimens compared to others which had been previously tested indicated that simple evaluation of the coating based upon visual inspection would not be valid since these latest coatings appeared uniformly black and shiny and there was no evidence of "burned" areas. As a consequence, several small cylindrical and disk-type specimens were ordered to see if a correlation could be developed between electrical discontinuity in the coating and the occurrence of premature failure. No valid correlation could be developed. In the meantime, Brush was further developing their anodizing process with the objective of improving performance, coating into holes and recesses, eliminating contact defects, and overcoming the problems associated with scale-up.

On July 19, 1962, T. Nakae of Atomics International and J. Rausch of ARF visited Brush to review the results of their development work and to witness anodizing of the first qualification specimen (Type 2). Brush was confident that, based on their development work, they could successfully anodize the bulk of this specimen and eliminate contact defects. The procedure to be used consisted of a tank anodizing treatment in a chromic acid solution after prior electrolytic cleaning in an alkaline solution. Anodizing was carried out for 30 minutes using a fixture which contained auxiliary stainless steel cathodes adjacent to each face of the specimen. In addition, there were looped wire cathodes in the specimen holes. After tank anodizing the anode contact points were coated using a chromic acid saturated cotton swab. The specimen was then sealed in boiling water. Photographs showing the five faces of the specimen after anodizing appear in Figures 5-7. The face containing two blind holes is shown in Figure 5. The bottoms of these holes are shiny, indicating the absence of a coating or the presence of a transparent coating. In the blind hole at the bottom of the photograph there is an additional shiny area near the top of the tapered part of the hole. This is barely visible in the photograph. A shiny area is also evident on the rear face of the specimen shown in Figure 6. This area appears as a band which spans two or more machining grooves. The views of the side faces, in Figure 7, show various light and dark areas; the light areas appear as

scratches. Face C shows the area where anodic contact was made during tank processing. The square region was subsequently coated by the portable method described. The dark areas adjacent to the square denote the region in which anodizing occurred in both processes.

The specimen was oxidized for 64 hours at temperatures which varied between 1398° and 1609°F in air having a dew point of -18°F. The condition of the faces after this exposure may be seen in Figures 8-11. In Figures 8 and 9 the condition of the blind holes may be noted. The specimen was washed in water and rinsed with alcohol prior to photographing. After oxidation the holes contained an extensive growth of BeO around the edges. During washing, this growth was removed and the region of attacked metal within the holes is clearly visible. The attack which occurred exists as a band which extends somewhat irregularly partially around the diameter of the straight portion of the hole. The bottoms of the holes, particularly the regions which appeared shiny prior to testing, appear to be relatively unattacked.

The other faces of the specimen show no evidence of failure. Surface discolorations are apparent. Some areas are transparent and resemble fingerprints which apparently resulted from handling during processing. In Figure 11, face C, there is a small dark area which appears to be a defect; however, there was no evidence of BeO at this spot and no evidence of corrosion after the specimen was washed.

The shiny area evident in Figure 6 is still apparent after oxidation, as may be seen in Figure 10. There is no evidence of attack in this region.

The specimen was tested for an additional 40 hours at 1450°F, and after that time slight evidence of oxidation in the form of small nodular growths of BeO through the coating was apparent on the side faces of the coating.

During the meeting at Brush on July 19, 1962, it was also decided to further evaluate anodizing processing variables while simultaneously studying the coatability of snap ring grooves. Six specimens were made which were cubes approximately 1 x 1 x 1 3/4 in. having two 1/4-in. diameter

scratches. Face C shows the area where anodic contact was made during tank processing. The square region was subsequently coated by the portable method described. The dark areas adjacent to the square denote the region in which anodizing occurred in both processes.

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During the meeting at Brush on July 19, 1962, it was also decided to further evaluate anodizing processing variables while simultaneously studying the coatability of snap ring grooves. Six specimens were made which were cubes approximately $1 \times 1 \times 1 \frac{3}{4}$ in. having two $\frac{1}{4}$ -in. diameter

blind holes drilled into one of the large faces. One of these holes had a snap-ring groove similar to that contained in the reflector parts. The specimens were anodized in pairs according to the following schedule:

Specimens 1 and 2

- (a) Tank-anodize for 30 min.
- (b) Portable-anodize contact points.
- (c) Seal in boiling water.

Specimens 3 and 4

- (a) Tank-anodize for 30 min.
- (b) Seal in boiling water.
- (c) Tank-anodize for 30 min.
- (d) Portable-anodize contact points.
- (e) Seal in boiling water.

Specimens 5 and 6

- (a) Tank-anodize for 30 min.
- (b) Portable-anodize contact points and holes.
- (c) Seal in boiling water.
- (d) Re-portable-anodize contact points and holes.
- (e) Seal in boiling water for 1/3 normal time.

These treatments were designed to compare normal processing (Specimens 1 and 2) with double anodizing procedures. All of the specimens were to be tested at 1450°F in dry air. However, the furnace containing Specimens 1, 3, and 5 overheated to 1600°F during the first 28 hours of exposure. Specimen 3 showed no failure after this exposure. Specimens 1 and 5 both failed in the hole which did not contain a snap-ring groove. These specimens also showed evidence of failure on the faces. Specimens 2, 4, and 6 were oxidized at 1450°F in dry air for 128.5 hours. The results were similar to those obtained with the first three specimens. Specimen 4 was the only one that did not fail.

Based on these results, it appears that the double tank anodizing process offers a distinct advantage; however, our evidence is certainly not statistically valid.

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While this work was being performed, Brush Beryllium Corporation processed a Type 1 qualification coupon using the same procedure originally used for the Type 2 specimen but with a modification in cathode wire configuration intended to improve coating within the holes. After the initial anodizing treatment Brush was not pleased with the appearance of the holes; therefore, the specimen was subjected to a second anodizing treatment. It is not clear whether a sealing treatment was employed between these operations. This specimen was tested at 1450°F for 103 hours in air at an average dew point of -40°F. There was no evidence of failure. The test had been interrupted and the specimen examined after 40 hours. Photographs showing the appearance of this specimen after 103 hours of exposure appear in Figures 12-16.

In Figure 12 one of the anodic contact points may be seen as a large light-colored circle on the lower part of the specimen. Several discolorations appear on this surface, particularly near the corners; however, there is no evidence of coating failure in these regions.

A second Type 2 qualification specimen was processed by Brush using the same procedure as for the original but incorporating the change in cathode wire configuration for coating holes that were used on the Type 1 qualification coupon. There was also, apparently, a relocation in the position of the auxiliary cathodes adjacent to the faces to permit more uniform coverage. This specimen was oxidized at 1450°F for 100 hours in air having a dew point of -40°F and showed no evidence of failure. Photographs showing the condition of the specimen after exposure appear in Figures 17-21.

A second Type 1 specimen was coated by Brush and tested at 1416°-1450°F with the dew point of the air varying between -40° and -60°F. After 39 hours there was no evidence of failure; however, a number of small defects were evident, as may be seen in Figures 22 and 23. All of the four major surfaces contained at least one defect. Several of the holes also contained defects. The specimen was oxidized for an additional 90 hours and, as may be seen in Figures 24-27, there is no significant increase in the extent of oxidation.

After reviewing our experimental conditions, we could find no variation to account for the difference in performance of the two Type 1 qualification specimens. Accordingly, this difference must be ascribed to a lack of consistency in the coating process. Fortunately, failure of the second specimen was not catastrophic; however, it would certainly be advantageous to have better reliability in the coating process.

In an attempt to find a more reliable coating process, nine samples were requested from Brush. These samples were anodized in triplicate by three different processes. Samples 1, 4, and 7 received the standard tank anodizing treatment followed by portable anodizing of the contact points. This is the procedure that was used on all but the second qualification specimen. Samples 3, 6, and 9 received a double tank anodizing treatment, and Specimens 2, 5, and 8 were partially re-anodized. The specimens were exposed at 1450°F for 115 hours. No failures occurred except in Specimens 4, 5, and 6. The failures in these specimens were in holes that were very roughly machined and contained minor cracks. The extent of failure was slight. Based on these test results there is no clearly demonstrated superiority of any of these coating techniques. The samples were exposed for an additional 64 hours, making a total of 179 hours. After this time period, Specimens 4, 5, and 6 showed continued growth of oxide in the cracked regions.

Since most of our experimental data have been obtained on coatings produced by single anodic processing for 30 minutes, it has been our recommendation that this technique be employed to coat the actual reflector parts. There is strong evidence to indicate that poor machined surface quality could be very detrimental to coating performance, and this factor should be considered before the parts are coated. All of the evidence that we have indicates that coatings produced by double anodic processing are at least as good or better than those produced in the single process. However, since this process has never really been used to coat one of the qualification specimens, recommendation of its use for the final parts could be a risk.

IV. LOGBOOKS AND PERSONNEL

The following personnel have contributed to this work:

John J. Rausch, Project Leader
Ray J. VanThyne, Project Engineer
Oswald Sanders, Technician
Andrew Williams, Technician

Data are contained in ARF Logbook Nos. C12058 and 12219.

Respectfully submitted,

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J. J. Rausch
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Neg. No. 23228

Fig. 1 - Anodized beryllium cubes containing recessed hole and shrink-fit plug. Photograph taken prior to oxidation testing.

Neg. No. 23296

Fig. 2

Specimen 1C after oxidation at 1450°F for 39 hours in laboratory air. After 16 hours there was only slight evidence of oxidation in the recess area.

Neg. No. 23297

Fig. 3

Specimen 5C after oxidation at 1450°F for 39 hours in laboratory air. After 16 hours there was only slight evidence of oxidation in the recess area.

Neg. No. 23286

Fig. 4

Specimen AC after oxidation at 1450°F for 34 hours in laboratory air.

Face A

Face B

Neg. No. 23371

Face C

Fig. 5 - Front view of anodized Type 2 Specimen prior to oxidation.

Face B

Face A

Face C

Fig. 6 - Rear view of anodized Type 2 Specimen prior to Oxidation.

Face A

Neg. No. 23368

Face B

Neg. No. 23370

Face C

Neg. No. 23367

Fig. 7 - Anodized faces of Type 2 qualification test coupon prior to oxidation.

Face A

Face B

Neg. No. 23381

Face C

Fig. 8 - Front face of Type 2 qualification test coupon after oxidation exposure for 64 hours.

Neg. No. 23382

Fig. 9 - Same as Figure 8 showing more detail within the holes.

Face B

Face A

Neg. No. 23377

Face C

Fig. 10 - Rear view of Type 2 qualification test specimen after oxidation at 1450°F for 64 hours in dry air.

Face A

Neg. No. 23379

Face B

Neg. No. 23378

Face C

Neg. No. 23380

Fig. 11 - Anodized faces of Type 2 qualification test specimen after oxidation exposure at 1450°F for 64 hours in dry air.

Neg. No. 23538

Fig. 12 - Front view of original Type 1 qualification specimen after oxidation for 103 hours.

Neg. No. 23539

Fig. 13 - Rear view of original Type 1 qualification specimen after oxidation for 103 hours.

Neg. No. 23542

Fig. 14 - Side view of original Type 1 qualification specimen after oxidation for 103 hours.

Neg. No. 23540

Fig. 15 - Top view of original Type 1 qualification specimen after oxidation for 103 hours.

Neg. No. 23541

Fig. 16 - Bottom view of original Type 1 qualification specimen after oxidation for 103 hours.

Face A

Face B

Neg. No. 23706

Face C

Fig. 17 - Front view of second Type 2 qualification specimen after oxidation for 100 hours at 1450°F.

Face B

Face A

Neg. No. 23706

Face C

Fig. 18 - Rear view of second Type 2 qualification specimen after oxidation for 100 hours at 1450°F.

Neg. No. 23709

Fig. 19 - Face A of second Type 2 qualification specimen after oxidation for 100 hours at 1450°F.

Neg. No. 23708

Fig. 20 - Face B of second Type 2 qualification specimen after oxidation for 100 hours at 1450°F.

Neg. No. 23710

Fig. 21 - Face C of second Type 2 qualification
specimen after oxidation for 100 hours
at 1450°F.

Neg. No. 23593

Fig. 22 - Beryllium specimen (Type 1, sample 4)
anodized and exposed to dry air at 1450°F
for 102 hours, showing white defect spots.

Neg. No. 23595

Neg. No. 23594

Fig. 23 - Defects in holes of beryllium specimen (Type 1, sample 4) after oxidation evaluation.

1 2 1 5 6

Neg. No. 23612

Fig. 24 - Second Type 1 qualification specimen after oxidation for 192 hours.

Neg. No. 23615

Fig. 25 - Rear view of second Type 1 qualification specimen after oxidation for 192 hours.

1 2 3 4 5 6
S E L L Z U 11 91 6 8 2 9 9 4 1 7 100

Neg. No. 23611

Fig. 26 - Top view of second Type 1 qualification specimen after oxidation for 192 hours.

1 2 3 4 5 6
S E L L Z U 11 91 6 8 2 9 9 4 1 7 100

Neg. No. 23613

Fig. 27 - Bottom view of second Type 1 qualification specimen after oxidation for 192 hours.