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DEVELOPMENT OF ALUMINUM BOBBIN SEALS FOR SEPARABLE CONNECTORS FOR ROCKET FLUID SYSTEMS

B. Goobich, J. R. Thompson, T. M. Trainer

TECHNICAL DOCUMENTARY REPORT NO. AFRPL-TR-67-191

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DEVELOPMENT OF ALUMINUM BOBBIN SEALS FOR SEPARABLE CONNECTORS FOR ROCKET FLUID SYSTEMS

by

B. Goobich, J. R. Thompson, T. M. Trainer

FOREWORD

This report summarizes research conducted under Phase I of USAF Contract No. 04(611)-11204 from January, 1966, to May, 1967. The research was performed by the Columbus Laboratories of Battelle Memorial Institute under the auspices of the Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, with Capt. John L. Feldman serving as project monitor. The principal investigators were J. R. Thompson, Research Engineer; B. Goobich, Associate Fellow; and T. M. Trainer, Project Manager.

This technical documentary report has been reviewed and is approved.

JOHN L. FELDMAN

Captain, USAF
Project Engineer

ABSTRACT

During earlier research under Contract No. AF 04(611)-9578, it was shown that damage to the flanges of the 6061-T6 aluminum AFRPL connector could be prevented if the seal material was softer than the flange material by 20 points Brinell. Under Phase I of follow-on Contract AF 04(611)-11204, five methods of accomplishing this were studied: (1) soft coatings, (2) skin annealing, (3) softer aluminum alloys, (4) anodizing the flange, and (5) special aging of 6061-T6. The overaged 6061 aluminum for the seal was selected because of economic and technical reasons. Repeated-assembly, thermal-gradient, and stress-reversal-bending tests were conducted with helium-leakage rates below the limit of 7×10^{-7} atm cc/sec. Tentative specifications and standards were revised.

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DEVELOPMENT OF ALUMINUM BOBBIN SEALS FOR SEPARABLE CONNECTORS FOR ROCKET FLUID SYSTEMS

by

B. Goobich, J. R. Thompson, and T. M. Trainer

Families of threaded connectors were developed for 6061-T6 aluminum tubing systems during earlier research under Contract No. AF 04(611)-9578. Although helium-leakage rates measured for typical connectors were well below the allowable leakage rate of 7×10^{-7} atm cc/sec for all testing modes, the 6061-T6 aluminum Bobbin seals caused considerable galling and radial distortion of the 6061-T6 aluminum-connector flange-cavity sealing surfaces. The condition limited reuse of the connector to a relatively few times, whereas it was desirable that each connector be reusable 25 times.

A view of a typical seal-flange interface after the fifteenth reassembly is shown in Figure 1. Lines indicate the original location of the flange-sealing face. Investigation of this limitation showed that the damage could be eliminated if the seal were softer than the flange by approximately 20 to 25 points Brinell. Details concerning the earlier research can be found in Report No. AFRPL-TR-65-162, "Development of AFRPL Threaded Fittings for Rocket Fluid Systems", November, 1965.

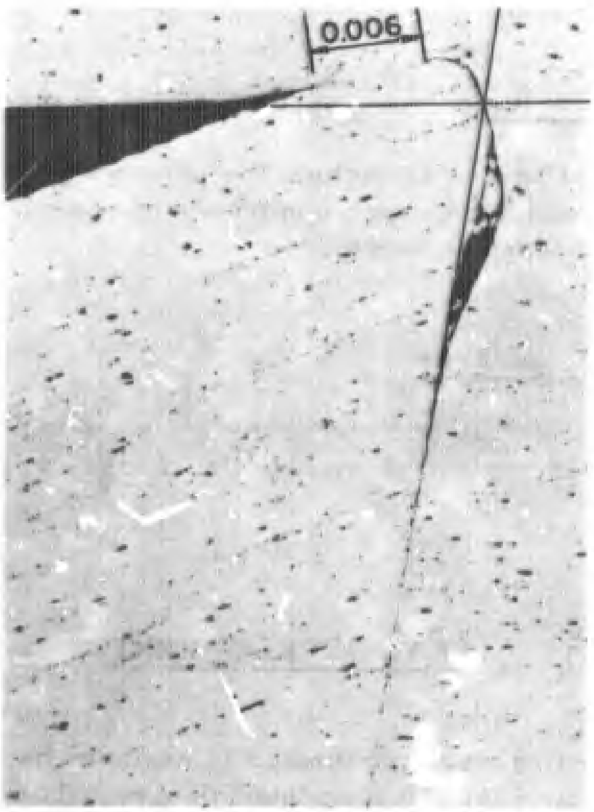

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FIGURE 1. SEAL INTERFACE DAMAGE OF 6061-T6 SEAL IN 6061-T6 FLANGE AFTER FIFTEENTH REASSEMBLY

This report describes Phase I of Contract No. AF 04(611)-11204, which was conducted from January, 1966, to May, 1967, to solve the flange-deformation problem of the aluminum threaded connectors. It was expected that the solution would be applicable to the aluminum flanged connectors to be designed under Phase II of Contract No. AF 04(611)-11204.

SCOPE AND OBJECTIVES OF PHASE I PROGRAM

The Phase I research was to include three types of activity: seal development, limited qualification testing, and revision of MS specifications and standards.

Seal Development

Research was to include an evaluation of five possible methods of eliminating the flange damage: (1) coating the aluminum seal with a softer material, (2) annealing the outer skin of the 6061-T6 aluminum seal, (3) using a softer aluminum alloy for the seal, (4) hardening the sealing surface of the flange, and (5) heat treating 6061 aluminum to a softer temper than that of the T6 condition.

Limited Qualification Testing

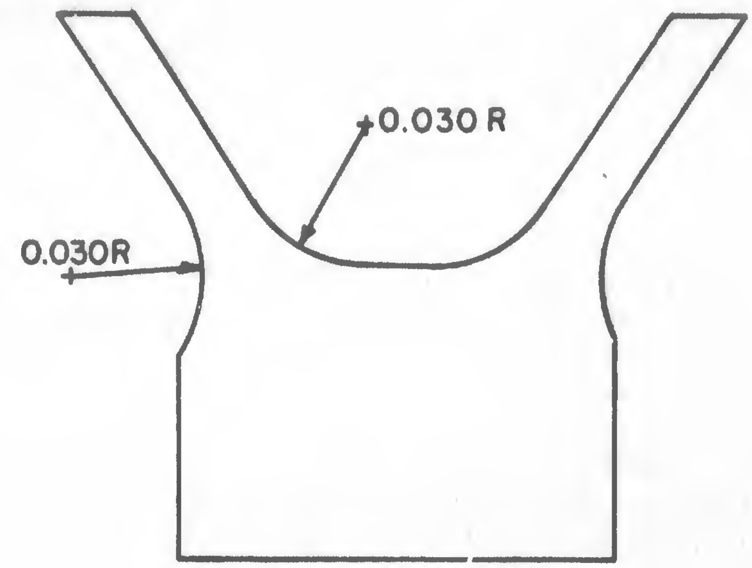
Qualification testing was to include the performance evaluation of 6061-T6 aluminum threaded connectors under conditions of repeated assembly, stress-reversal bending, and thermal shock.

MS Specifications and Standards

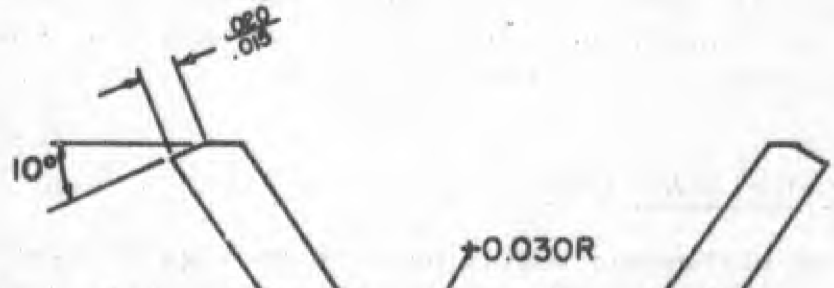
Tentative specifications and standards prepared under Contract No. AF 04(611)-9578 were to be revised to include the changes resulting from the research effort.

SEAL DEVELOPMENT

Preliminary studies were performed to evaluate the five methods of eliminating the damage to the 6061-T6 aluminum flanges. Tests showed that damage could be eliminated and helium-leakage rates less than 7×10^{-7} atm cc/sec could be achieved by use of three methods. Because of lower cost, greater inherent reliability, and process simplicity, it was decided that the use of overaged 6061 aluminum seals was the best solution.



a. Standard



b. 10-Degree Reverse Angle

FIGURE 2. REVISED BOBBIN SEAL DESIGNS

During the course of the preliminary studies a revised seal design was evolved and evaluated. This design is shown in Figures 2a and 2b. The configuration in Figure 2a was chosen for small-diameter seals (-16 connectors and smaller) and the configuration in Figure 2b was chosen for larger diameter seals (tubing diameters greater than 1 inch and up to 16 inches).

Coating

Possible coatings for 6061-T6 aluminum Bobbin seals and methods of coating application were investigated. These included electroplating, vapor and vacuum deposition, and metal spraying of aluminum, nickel, and the noble metals. Also studied were organic coatings such as Teflon and Kel-F.

In selecting a coating material, consideration was given to the operating temperature (-423 to 200 F), the expected corrosion rate, the effects of galvanic corrosion, and the possibility of fluid decomposition. A perusual of the available literature led to the selection of aluminum as the best coating within the present technology, primarily due to the adverse effects of galvanic corrosion normally encountered with the other metallic coatings in contact with aluminum. Experimental results indicated that electroplated seals eliminated deformation and provided low leakage rates. However, electroplating was not selected as the final solution because the chemicals presently used are explosive and the process is too complex for economical commercial operations.

Methods of Application of Aluminum

Four methods of aluminum application were investigated: electroplating, vacuum or vapor deposition, metal spraying, and a new process developed by Engelhard Industries.

Electroplating. A process for electroplating aluminum coatings as thick as 0.006 inch with a Knoop hardness of 42 to 50 had been developed during prior research at Battelle's Columbus Laboratories. Although the process was operational, it was necessary to establish the exact plating parameters to meet the requirements of the Bobbin seal. Detailed descriptions of the equipment used and of the process are given below.

A second method considered for electroplating was the Dalic process. In this process deposits are made directly onto the conductive surfaces from highly concentrated electrolyte solutions that are held in absorbent materials attached to portable electrodes. The advantage of the Dalic process is that an immersion bath is not required, and, therefore, it is possible to plate only the critical sealing surface, quickly and economically, without special masking operations and with relatively simple tools. However, chemical solutions are not now available for plating aluminum.

Vacuum and Vapor Deposition. In vacuum deposition the material to be deposited is introduced as a vapor and coats the specimen as it contacts the cold surface being coated. Because the vaporized material flows in a line-of-sight fashion, it would be necessary to rotate the Bobbin seal to achieve an even coating. This requirement complicates the process. However, the major problem is the deposition of heavy coatings. The metal is deposited in tension, and as the coating becomes thicker there is a tendency for the plating to peel.

In the vapor-deposition process the metal to be deposited is introduced as a gaseous chemical compound and the specimen is heated. Because the gas diffuses and fills the available volume, it is not necessary to rotate the specimen. When the gas contacts the heated specimen a chemical reaction occurs, and the metal is deposited on the surface. It is possible to achieve even coatings on complex shapes. However, to deposit thick coatings the specimen must be heated to elevated temperatures for long periods.

Metal Spraying. In the metal-spraying process, melted metal is sprayed on a rotating part by a stationary metalizing gun. Although it is claimed that the thickness of the coating can be controlled and that thicknesses greater than 0.0025 inch can be deposited easily, porosity is a major problem. It is also necessary to remachine the seal if the deposits are uneven or rough. For these reasons metal spraying was not considered.

Engelhard Industries. The Engelhard process involves the use of a "true solution" composed of the metal to be deposited and certain organic compounds. This solution is "painted" onto the surface, and the specimen is baked at temperatures ranging from 750 to 930 F. As the organic substances are driven off, the metal deposit is bonded to the surface. Three reasons for not choosing this process are: (1) the bonding temperature is too high, (2) the thickness of the deposit is generally 1×10^{-5} inch, and considerable effort is required to attain thicker coatings, and (3) aluminum deposition is not one of the processes now available.

Electroplating Procedure and Equipment

A laboratory setup was assembled to plate a limited number of seals for experimental evaluation.

Plating Solution. A 4 to 4-1/2-liter plating solution consisting of the following was prepared:

Aluminum chloride[a], $AlCl_3$:	400 to 450 g/liter
Lithium hydride[b], LiH:	6 to 7 g/liter
Ethyl ether[c], $(C_2H_4)_2O$:	to make a 1-liter solution.

(a) Anhydrous, No. A-575, Fisher Scientific Company.

(b) Metals Hydrides Company.

(c) Anhydrous, No. 9244, J. T. Baker Chemical Company.

The solution was prepared in a three-neck 5-liter distillation flask. The flask was provided with a dry nitrogen atmosphere, a stirrer, and a cooling bath (<15 F). The aluminum chloride, followed by the lithium hydride, was slowly added to the ether while maintaining the solution temperature at 15 ± 10 F.

The plating bath was prepared in a fume hood under conditions so that no open flame was present and no electric sparks could occur. At no time was the plating bath permitted to come in contact with water. For disposal, the solution was permitted to dilute itself slowly while open to air in a vented hood. When well diluted, it was disposed of with a surplus of water. It is imperative that these precautions be followed to prevent explosive hazards.

Equipment. The plating solution was syphoned (nitrogen pressure in flask) into the aluminum plating cell, shown in Figure 3, which was blanketed with a flowing dry nitrogen atmosphere. High-purity-aluminum strip anodes (1 by 11 inches) were positioned with about 4 inches of immersed length on opposing sides and about 1-1/2 inches from the Bobbin seal surfaces. Cathode agitation was accomplished by reciprocating-rotational movement (slightly over 1 revolution) at 33 cycles/minute thus providing a surface movement of about 16 ft/minute on the outer surfaces of the racked seals.

Procedure. The plating procedure is described in the following 14 steps.

(A) Solvent degrease: tissue wipe with mineral spirits, follow by tissue wipe with acetone.

(B) Alkaline soak clean: immerse in "Sprex AC" (15 g/liter) non-etching proprietary cleaner (Du Bois) at 170 ±10 F for 5 minutes, follow by tap-water rinse.

(C) Nitric acid pickle: immerse in 8N HNO_3 at 80 ±5 F for 3 minutes, follow by tap-water rinse.

(D) Zincate treatment: immerse in Enthone's "Alumon D" (130 g/liter) solution at 75 ±5 F for 1 minute, follow by tap-water rinse.

(E) Zinc strip treatment: same as (C) but separate nitric acid solution.

(F) Zincate treatment: same as (D).

(G) Alcohol rinse: immerse in ethyl alcohol.

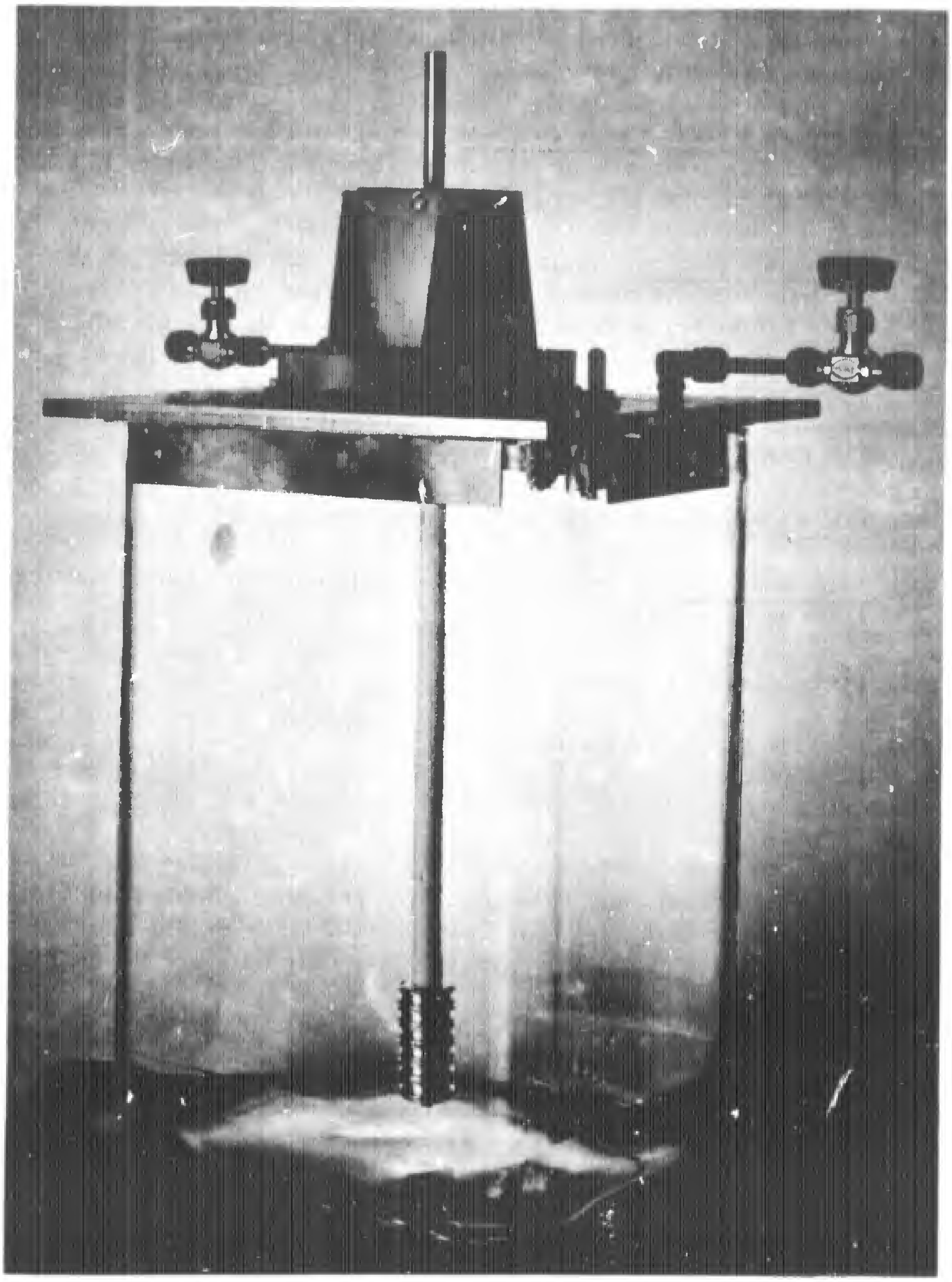


FIGURE 3. LABORATORY EQUIPMENT USED TO PLATE SOFT ALUMINUM ON 6061-T6 BOBBIN SEALS

(H) Rack: position Bobbin seals on cathode rod alternating with two spacers (one spacer at top end). Transfer the seals and spacers from the alcohol (Step G) with bone-tipped tweezers, not touching areas to be aluminum plated.

(I) Oleic acid activation treatment: immerse in 100 percent acid at 75 ±5 F for 1 minute and drain for about 1 minute prior to transfer to aluminum-plating cell.

(J) Aluminum plating: position in aluminum-plating cell, start reciprocating-rotational agitation, and apply plating current after 1/2 to 1 minute of agitation. (With a cell voltage of 2.2 ±0.2 volt and plating current of 0.55 ±0.05 ampere, the range of current density on the six Bobbin seal edges was 20 ±5 amp/sq ft with plating rates of 1 ±0.25 mil/hour). Plate for 3 ±1/2 hours.

(K) Ether rinse: immerse in ethyl ether to dilute.

(L) Tap water rinse: immerse in tank with overflow rinse, unrack seals and spacers.

(M) Distilled-water rinse.

(N) Acetone rinse and air dry.

Evaluation of Plating

Regular Bobbin seals and 10-degree reverse-angle Bobbin seals plated with high-purity aluminum were assembled with 6061-T6 connectors and leak tested with 2000 psi helium. Table 1 presents the assembly data and the leakage rates.

TABLE 1. ASSEMBLY DATA AND LEAKAGE RATES FOR ALUMINUM-PLATED SEALS

Specimen	Seal Type	Plating Thickness, in.	Assembly Load, lb Peak 1	Assembly Load, lb Peak 2	Change in ID, in.	Leakage at 2000 psi
1	Regular	0.0020	1165	1350	0.012	(b)
2	10 deg	0.0025	1300	1320	0.016	(b)
3	Regular	0.0020	1260	1400	(a)	(b)
4	10 deg	0.0030	1230	1260	(a)	(b)

(a) Not measured; specimens prepared as microsections.

(b) Leaks were less than mass-spectrometer sensing capability.

Microsections were prepared from Specimens 3 and 4. Figure 4 shows the two sealing interfaces of Specimen 3. Seal contact length ranged from 0.008 to 0.013 inch with negligible flange deformation. The average thickness of the aluminum plating was 0.002 inch. Figure 5 shows the two sealing interfaces of Specimen 4. Seal contact length in this case ranged from 0.008 to 0.014 inch. Again deformation in the flange was negligible. Plating thickness averaged 0.003 inch.

Although Specimen 4 successfully sealed helium, Figure 5 shows that the aluminum plating separated approximately 0.001 inch from the basis metal on one sealing leg. Although the area in which this separation existed was not in contact with the fluid in this case and did not appear to affect the sealing capabilities of aluminum-plated seals, it could be a potential source of trouble. A suggested method of enhancing the adherence of the aluminum plating on the basis metal is to heat treat the plated seals at 250 F for 3 to 4 hours. This would cause the basis metal, the zinc strike, and the electrodeposited aluminum to diffuse, at the interface, into a thin intermetallic bond with a considerably greater adherence.

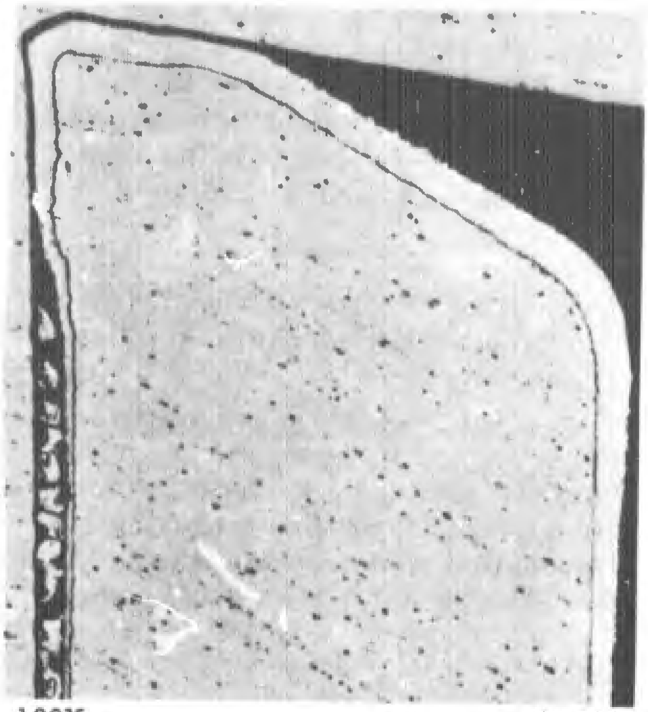
Skin Anneal of 6061-T6

The objective of this investigation was to determine the feasibility of annealing the skin of 6061-T6 seals to a depth of 0.030 inch. Inquiries at Battelle's Columbus Laboratories and at Alcoa indicated that similar work had not been attempted with aluminum alloys. However, the feasibility of the approach was not ruled out by the metallurgists. Experiments were devised, based on the theory that if the surface of the specimen were heated slightly above the annealing temperature for short periods of time while the core was kept as a substantially lower temperature, the resulting effect would be a hardness gradient in which the core would maintain a comparatively harder condition than the surface.

Although experimental results were inconclusive, there was evidence that "skin annealing" of 6061-T6 is possible. However, the methods that seem most applicable are either too complex or too difficult to control to be practical at present.

Experimental Method A

Under Method A, 12 test specimens approximating the 3/4-inch seal were fabricated from 6061-T6 material. In addition, heat-absorbing copper chills that contacted all surfaces of the aluminum seals, except the outside diameter, were made. A typical assembly for Method A is illustrated in Figure 6. This assembly was inserted into a nitride/nitrate salt bath (preheated and stabilized at 800 F) for 10 seconds and was then quenched in water at room temperature. This procedure was repeated with additional assemblies immersed in the salt bath for 20- and 30-second periods and with three assemblies that were precooled to -320 F prior to the insertion in the salt bath.



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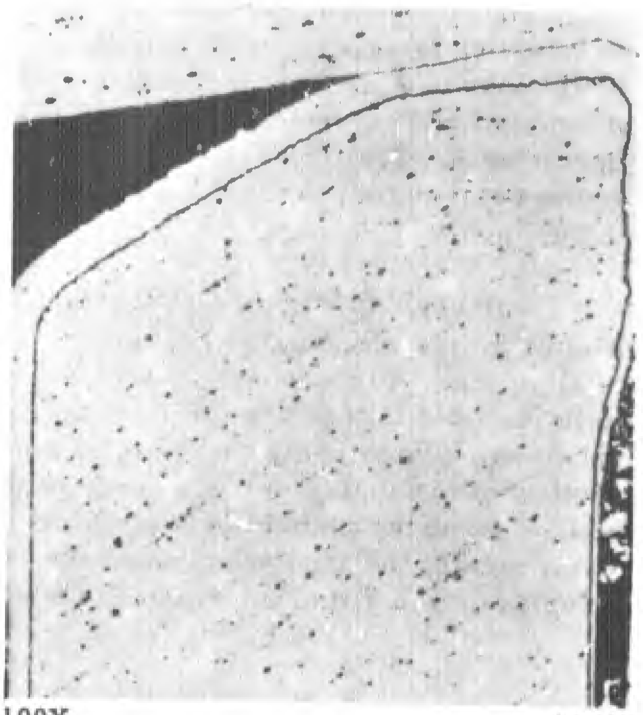

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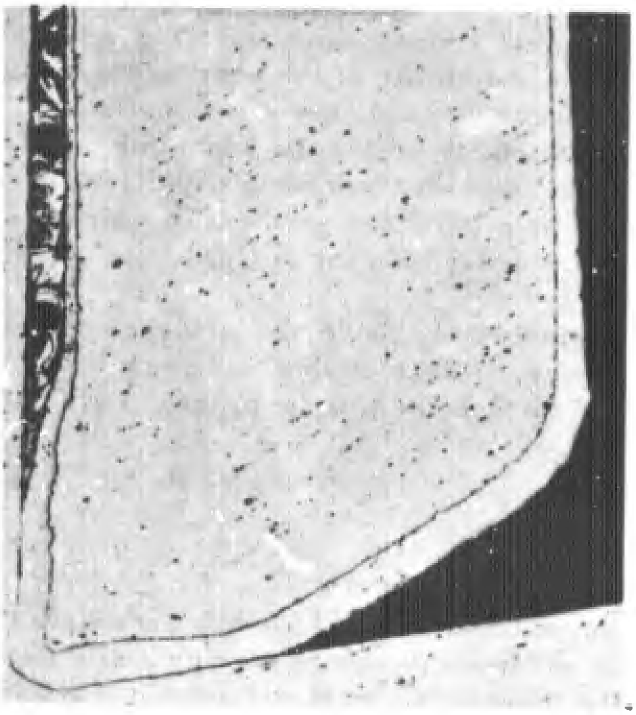

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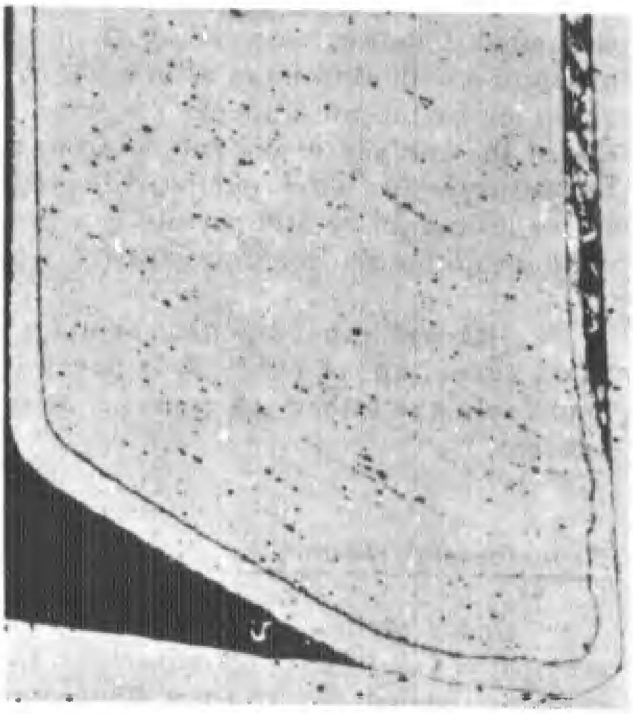

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FIGURE 4. SEAL INTERFACE – ALUMINUM-PLATED SEAL, SPECIMEN 3

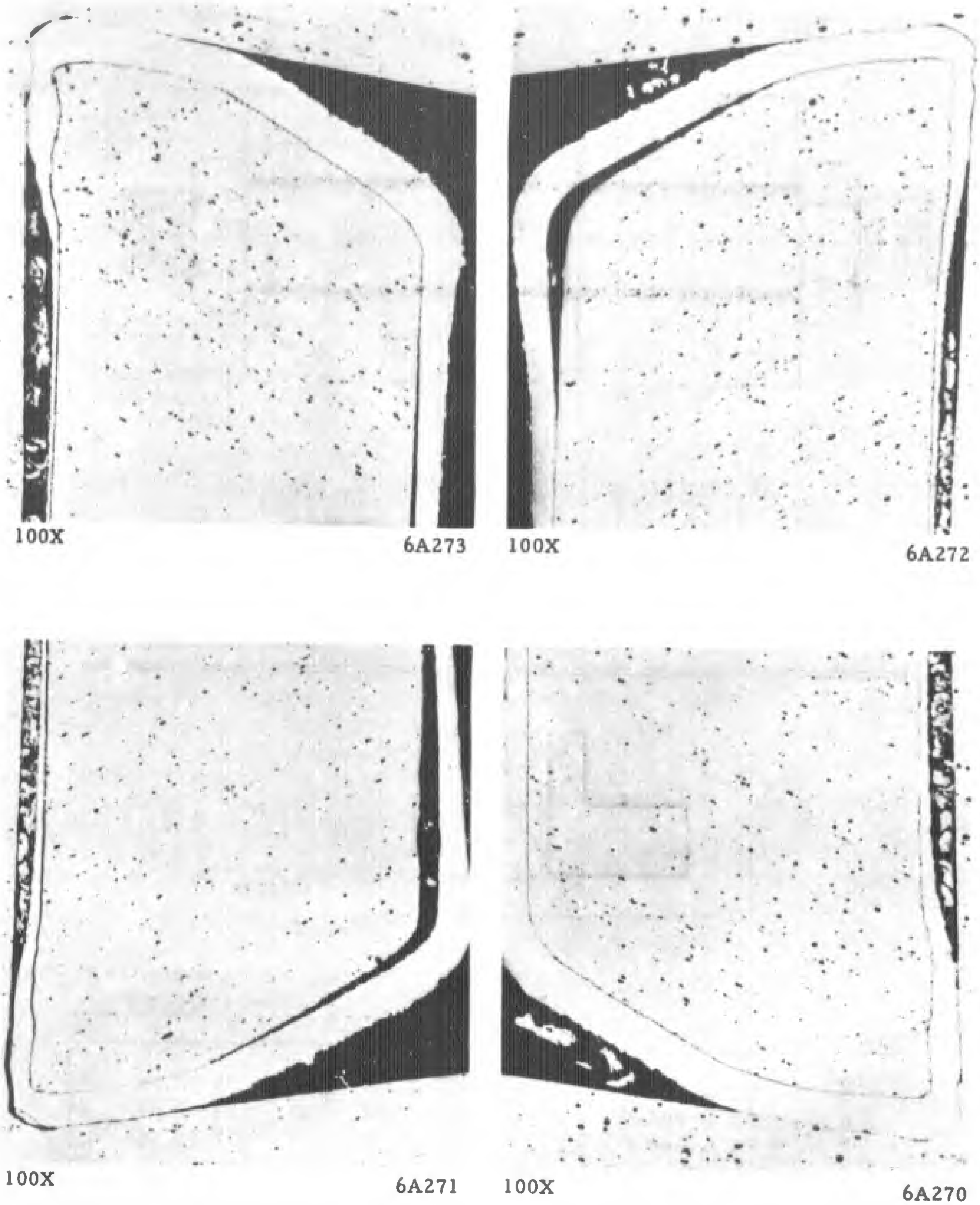

FIGURE 5. SEAL INTERFACE – ALUMINUM-PLATED SEAL, SPECIMEN 4

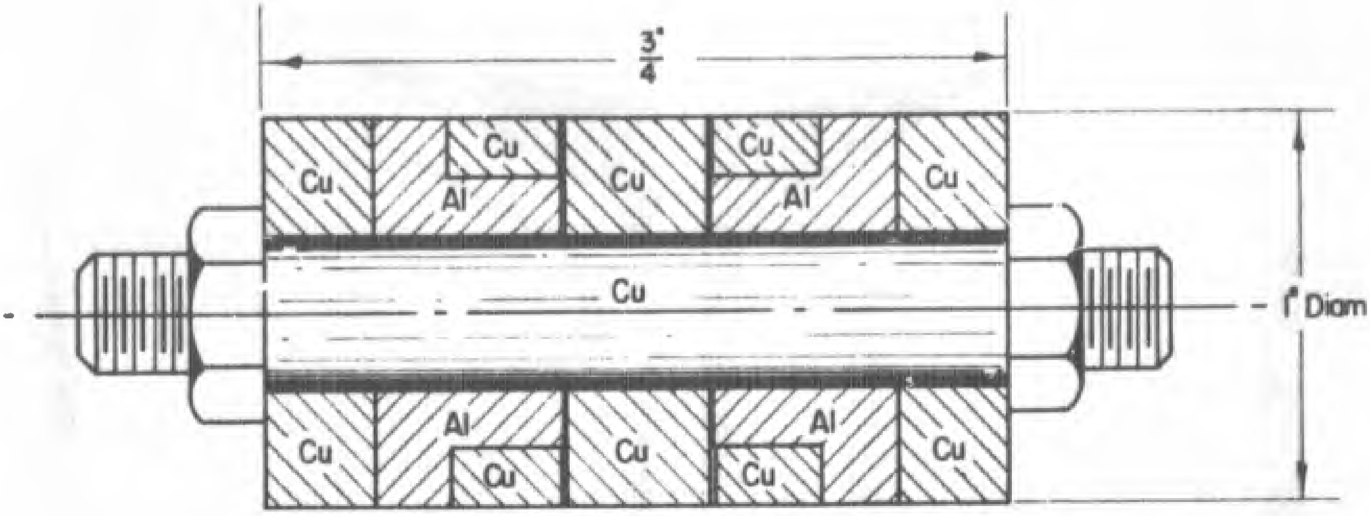

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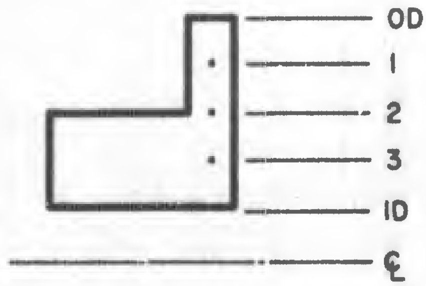
FIGURE 6. EXPERIMENTAL ASSEMBLY FOR SKIN ANNEAL – METHOD A

After heat treatment, the specimens were mounted and sectioned, and the hardness gradient was measured. Table 2 presents these data.

TABLE 2. KNOOP-HARDNESS DATA FOR SPECIMENS 5 THROUGH 10



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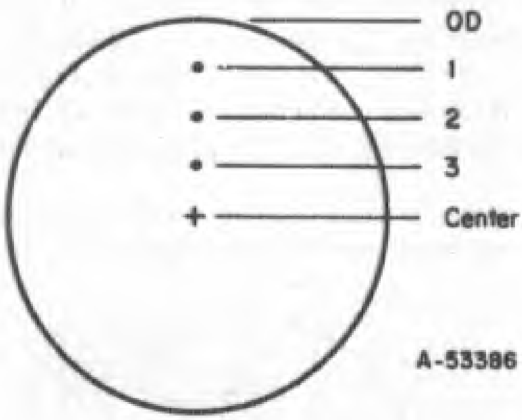
Specimen	Condition	Time Exposed at 800 F, sec	Average Knoop Hardness at Locations Indicated				
			OD	1	2	3	ID
5	RT to 800 F	10	99	101	95	95	96
6	RT to 800 F	20	81	83	79	87	83
7	RT to 800 F	30	77	74	77	80	82
8	-320 F to 800 F	10	97	103	106	99	103
9	-320 F to 800 F	20	102	100	100	100	107
10	-320 F to 800 F	30	88	91	93	94	101

From an examination of the data it appeared that moderate success was achieved with only Specimen 10. Although the hardness at the outside surface was less than the hardness at the inside surface in Specimens 6, 7, 8, and 9, some areas of the core were softer than the outside surface. However, in the case of Specimen 10 the hardness gradient across the section followed the desired pattern. The fact that no noticeable alteration in grain structure occurred was another favorable indication that the material's strength was not significantly changed, although the surface was made softer.

Experimental Method B

The procedure used for Method B was the same as that described for Method A except that the specimens were solid 1-inch-diameter bars of 6061-T6 aluminum. Table 3 presents the average Knoop-hardness gradient from the outside diameter to the center at 0.3-centimeter intervals.

TABLE 3. KNOOP-HARDNESS DATA FOR SPECIMENS 11 THROUGH 16



Specimen	Condition	Time Exposed at 800 F, sec	Average Knoop Hardness at Locations Indicated				
			OD	1	2	3	Center
11	RT to 800 F	10	107	111	107	102	104
12	RT to 800 F	20	105	109	102	101	95
13	RT to 800 F	30	83	80	83	83	78
14	-320 F to 800 F	20	101	107	105	105	101
15	-320 F to 800 F	10	103	112	105	105	99
16	-320 F to 800 F	30	102	100	102	106	96

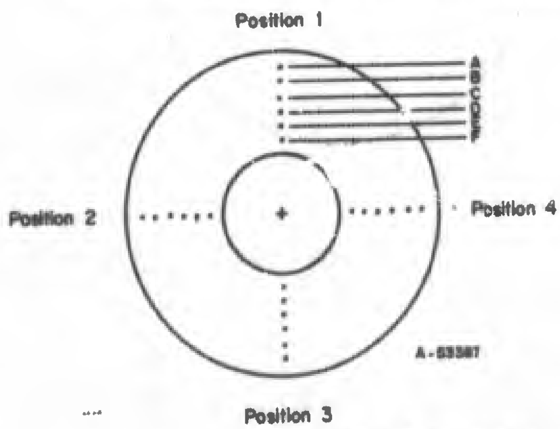
The hardness readings at the outside surface were higher than those obtained at the same location under Method A, and the core was consistently softer than the outside surface. This indicated that the copper chills used in Method A had the beneficial effect of maintaining a substantially lower temperature at the core.

Experimental Method C

Method C entailed the fabrication of a 6061-T6 heavy-walled tube, 1-inch outside diameter and 6 inches long with an 11/16-inch hole through the center. This specimen was solution heat treated for 1 hour, and immediately upon removal from the furnace, the outside diameter was wrapped with insulation, and the entire assembly was quenched in cold water, thus causing the cylinder to be cooled from within. This delayed quench was expected to result in a T4 temper at the inside surface and a softer condition at the outside surface. The specimen was then heated to 350 F for 6 to 8 hours and air cooled to artificially age harden the inside surface to approximately the T6 temper.

Knoop-hardness measurements of a coupon taken from the center of the bar are presented in Table 4. Preliminary analysis of the hardness readings indicated that the delay quench was not entirely successful; probably because Transite was used as the insulating material. Transite, being hard, apparently did not seal the outside diameter of the tube from the quenching water, thereby allowing the cooling to take place from the outside surface as well as from the inside surface. In any case, the change in hardness was insignificant at either surface.

TABLE 4. KNOOP-HARDNESS DATA FOR METHOD C



Location	Distance From OD to ID, cm	Knoop Hardness for Positions Indicated 1	2	3	4	Average
A	0.025	110	105	109	109	108+
B	0.075	107	108	108	113	109
C	0.150	114	114	111	113	113
D	0.225	114	111	114	108	112+
E	0.300	113	113	114	111	113-
F	0.350	113	109	110	108	110

Softer Aluminum Alloy

The use of softer aluminum alloys in standard tempers, whose yield strengths range from 30,000 to 35,000 psi, was also considered. Materials in this category are listed in Table 5. The mechanical properties were obtained from standard references such as the Alcoa Aluminum Handbook and the Metals Handbook. Those alloys with the desired mechanical properties are marked with an asterisk. However, as indicated in Table 5, none of the candidate alloys are readily available in a form usable for manufacturing small-diameter Bobbin seals. Therefore, it was concluded that no advantage would be gained by using any of these alloys instead of 6061.

TABLE 5. POSSIBLE ALLOYS FOR USE AS BOBBIN-SEAL MATERIAL

Material	Yield Strength, ksi	Elongation, percent	Brinell Hardness	Remarks
6061-T6	40	17	95	
2218-T72	37	11	95	(a)
3004 H36	33	9	70	(b)
3004 H38	36	6	77	(b)
5052 H36*	35	10	73	Mill run only
5052 H38	37	8	77	(b)
5086 H32	30	12	77	(c)
5086 H34	37	10	86	(a)
5154 H34*	33	13	73	Mill run only
5154 H36*	36	12	78	Mill run only
5254 H34*	33	13	73	Mill run only
5254 H36*	36	12	78	Mill run only
5357 H38	30	6	55	(b)
5454 H34*	35	10	81	1/4-inch plate only – other forms, mill run only
5456 H311	33	18	86	(a)
5456 H321	37	16	90	(a)
5652 H36*	35	10	73	Relatively new, possibly in bar
5652 H38	37	8	77	(b)
6053 T6*	33	13	80	Rivet stock
6063 T6*	31	12	73	Extrusions
6063 T83	35	9	82	(b)
6463 T6	31	12	[illegible]	Extrusions

(a) Too hard.
(b) Insufficient elongation.
(c) Yield strength too low.

Harden Flange

Various methods of hardening the sealing surface of the flange were investigated. These included the Martin hardcoat, the Sanford process, and the Alumilite process.

Some of the disadvantages which are known to be associated with any anodizing process and which could cause a premature failure in a connector of this type are:

(1) The fatigue life of 6061-T6 aluminum is reduced 60 percent by anodizing

(2) The film is susceptible to corrosion from certain strong alkalis and acids

(3) Porosity is inherent in the anodizing process

(4) Fine hairlike cracks, called crazing, occur in the surface of the anodic film.

Since the anodized surfaces are small in comparison with the size of the connector and are not located where fatigue stresses are severe, fatigue failure was not considered to be a problem.

Although hardening by the Sanford process prevented deformation of the connector flange, this process could not be used because cracks in the film caused leakage. Anodizing by the Alumilite was acceptable because deformation was prevented and low leakage rates were obtained.

Sanford Process

Information about the Sanford and the Martin hard-coating processes was obtained. In both processes the thickness of the anodic film and its hardness, porosity, and corrosion resistance are reportedly similar. Basic differences in the two processes are found in the sealing methods and in the electrolyte compositions. Those who employ the Sanford process recommend sealing the anodic coating in deionized cold water to reduce porosity in the film. In the Martin process, sealing is not recommended if maximum hardness is desired. In the Sanford process, the electrolyte is a mixture of sulphuric and oxalic acids in solution, whereas in the Martin process, only sulphuric acid in solution is used. Electrolyte temperature and current density are approximately the same for both processes.

Though both processes are similar and reportedly produce equivalent results, it was decided to conduct experiments with specimens treated by the Sanford process. Leakage tests were performed with three sets of flanges hard coated by the Sanford process. Films of 0.001-, 0.0015-, and 0.002-inch thicknesses were prepared. Two 6061-T6 seals were tested with each set of flanges. None of the specimens sealed helium, even at low pressures, although it was possible to seal hydraulic fluid at 8000 psi. These data are presented in Table 6.

TABLE 6. ASSEMBLY DATA AND LEAKAGE RATES FOR FLANGES TREATED BY THE SANFORD PROCESS

Seal Specimen	Seal Type	Anodic Film Thickness, in.	Assembly Load, lb		Change in ID, in.	Leakage Rate
			Peak 1	Peak 2		
17	10-deg.	0.001	1290	1320	0.015	(b)
18	Regular	0.0015	1410	1400	0.015	(b)
19	10-deg.	0.002	1200	1325	0.014	(b)
20	Regular	0.001	1230	1335	(a)	(b)
21	10-deg.	0.0015	1140	1275	Not measured	(b)
22	Regular	0.002	1300	1440	Not measured	(b), (c)

(a) Not measured; specimen prepared as microsection.
(b) Leakage was much greater than mass-spectrometer sensing capability, i.e., $>10^{-5}$ atm cc/sec.
(c) Hydraulically tested at 8000 psi, no leaks.

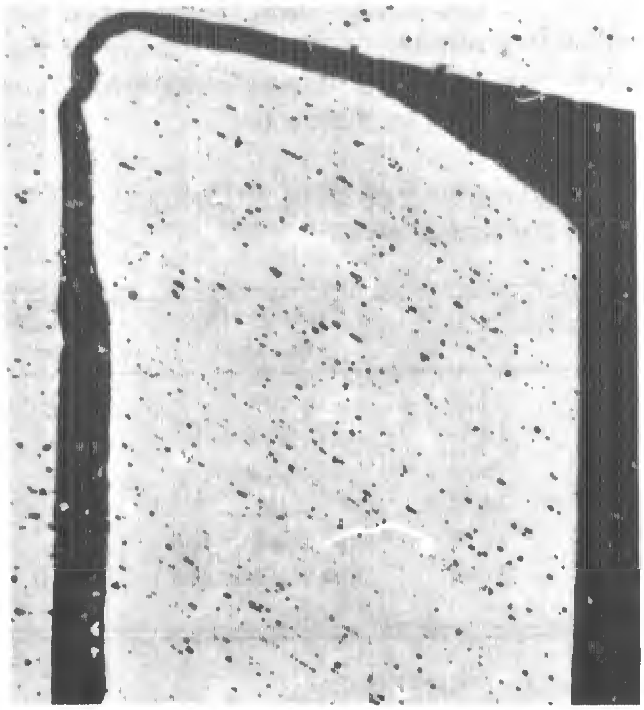
Figure 7 shows the two interfaces of Specimen 20. Because both the anodic film and the background are dark, it is difficult to distinguish between them in the photographs. Examination of the specimen with a high-power microscope, however, revealed an excellent contact, ranging from 0.010 to 0.013 inch in length, at the interface. However, evident cracks and porosity within the film were the probable causes of leakage. A section was taken on a plane normal to the connector axis through the flange corner as indicated in Figure 8a. Figure 8b is a view of a typical area at 500X showing several cracks of varying widths within the anodic film (dark area). These cracks probably criss-cross with each other and with cracks running at right angles to create a leakage path.

Alumilite Process

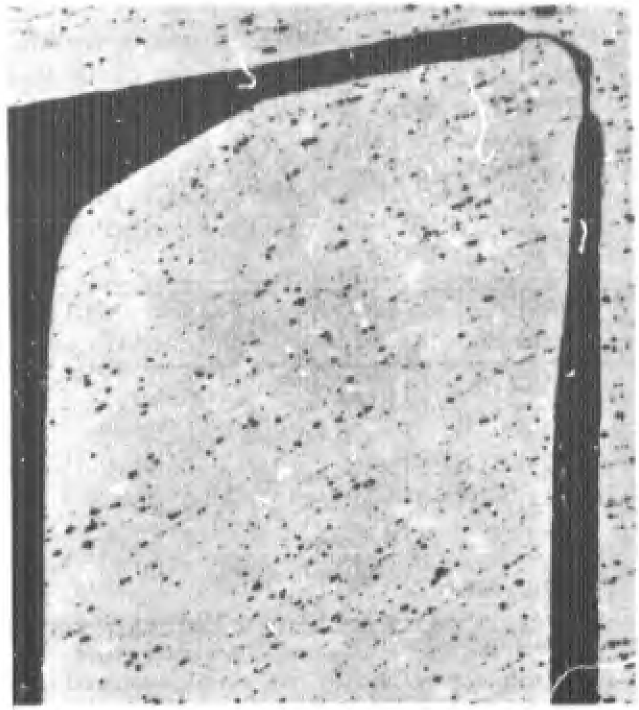
The Alumilite process is the standard sulphuric acid anodizing process developed by Alcoa. An anodic film (aluminum oxide) is formed and then is sealed in a solution of nickel acetate and water.

Initially, a threaded flange and a plain flange were anodized with an 0.0005-inch anodic film. Each anodized flange was assembled with a nonanodized flange and a 6061-T6 seal. One seal was of the standard Bobbin configuration and the other had a 10-degree reverse angle. The two connectors were assembled in a universal testing machine and microsections were prepared.

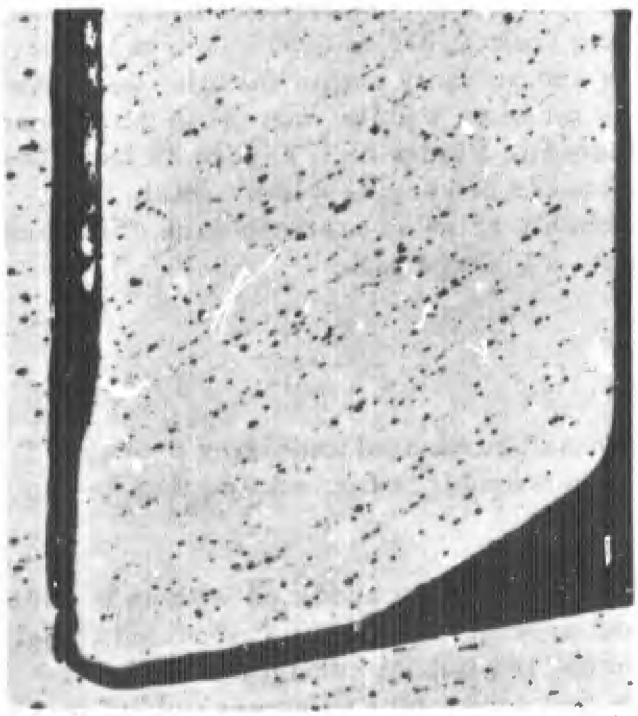
Photomicrographs of the anodized seal interfaces, at 100X are presented in Figure 9. Figure 9a shows the seal with a 10-degree angle, and Figure 9b shows the standard seal. Figures 10a and 10b are photomicrographs of the nonanodized flanges with the 10-degree seal and the standard seal, respectively.



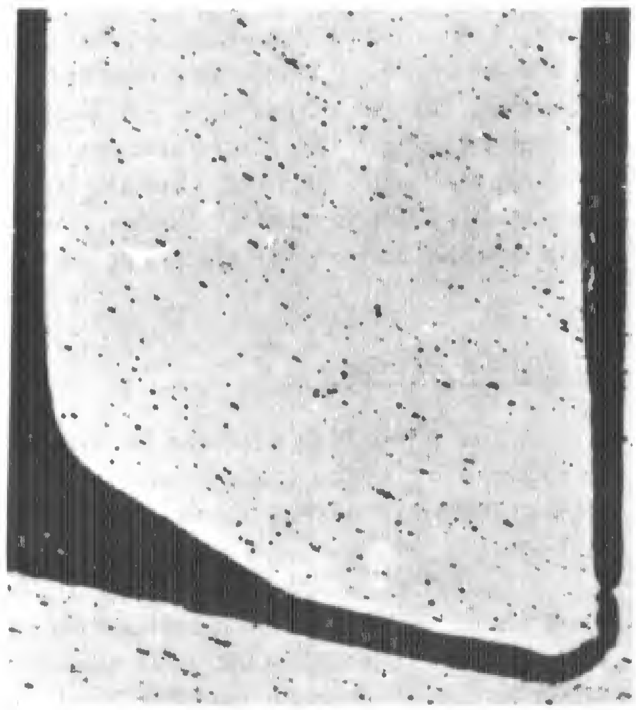
100X 6A274



100X 6A275

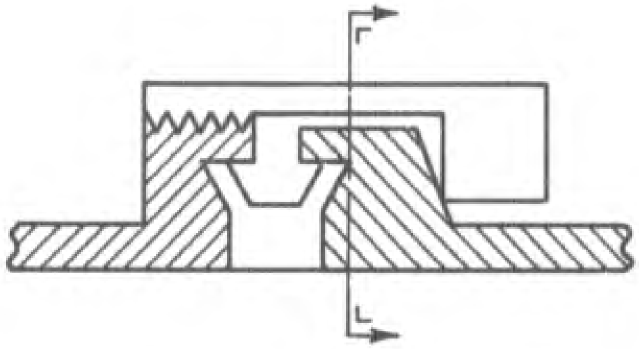


100X 6A276

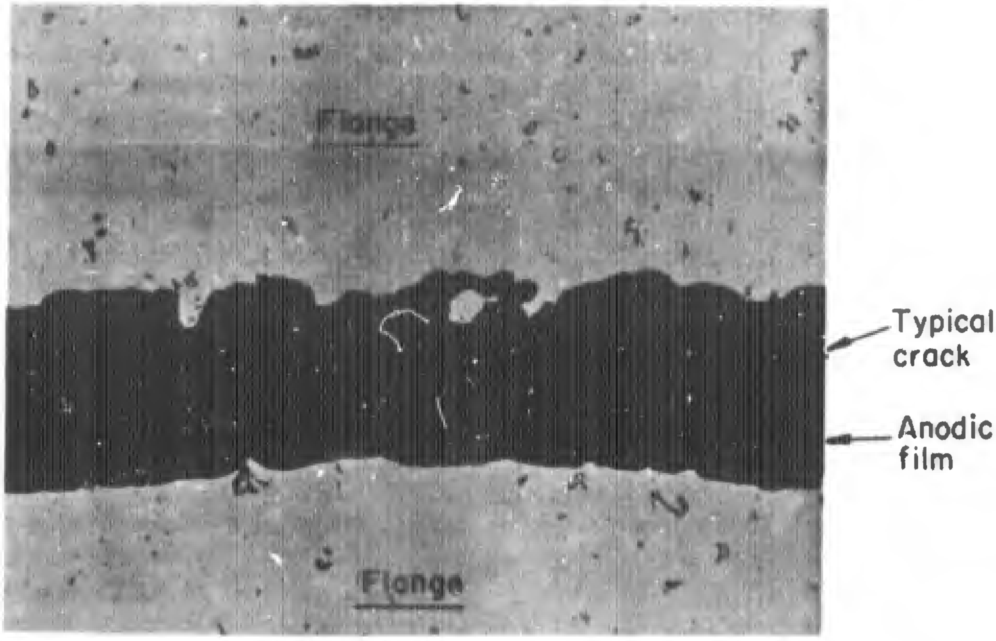


100X 6A277

FIGURE 7. ANODIZED SEAL INTERFACE - SPECIMEN 20



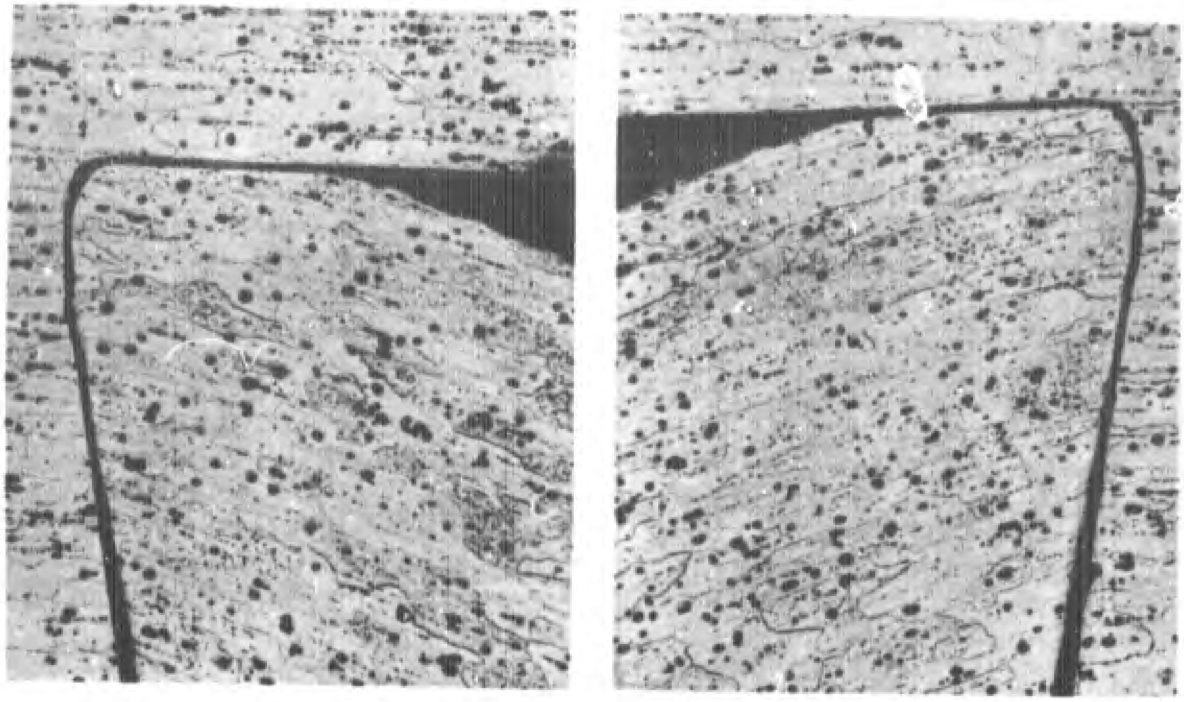
a. Location of Cross-Sectional Plane



500X 6A729

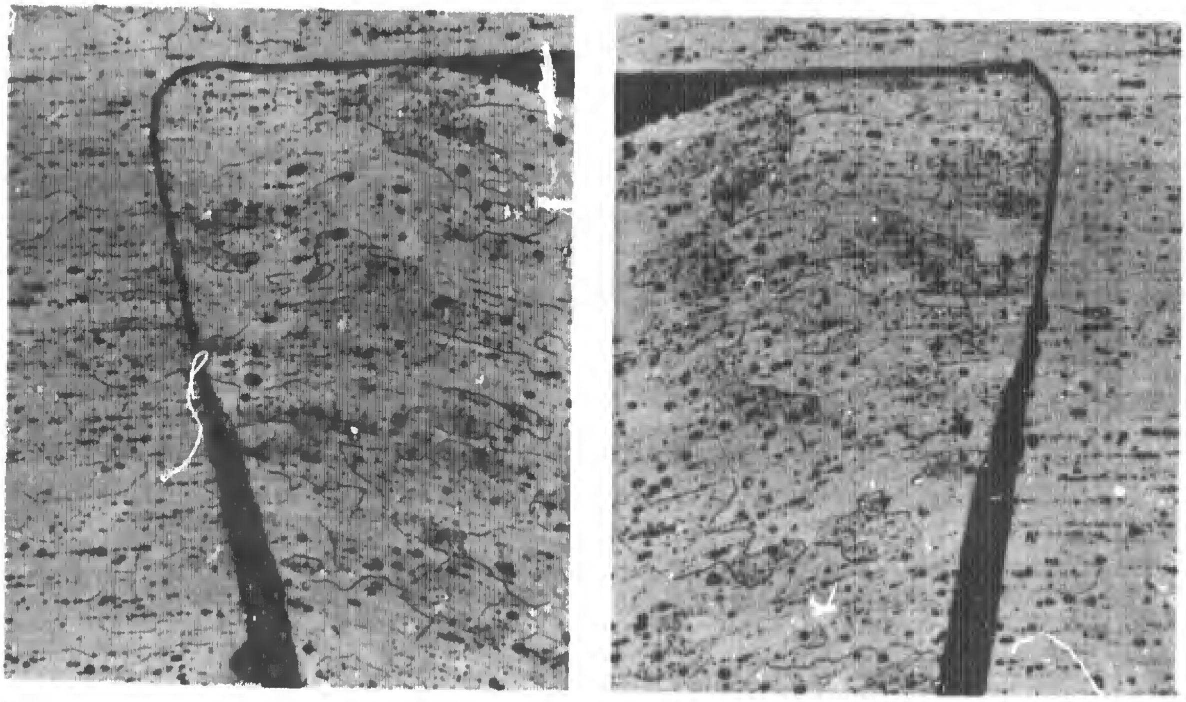
b. Enlarged View of Cross Section

FIGURE 8. SEAL INTERFACE – SANFORD HARD COAT



100X 3A608 100X 3A606

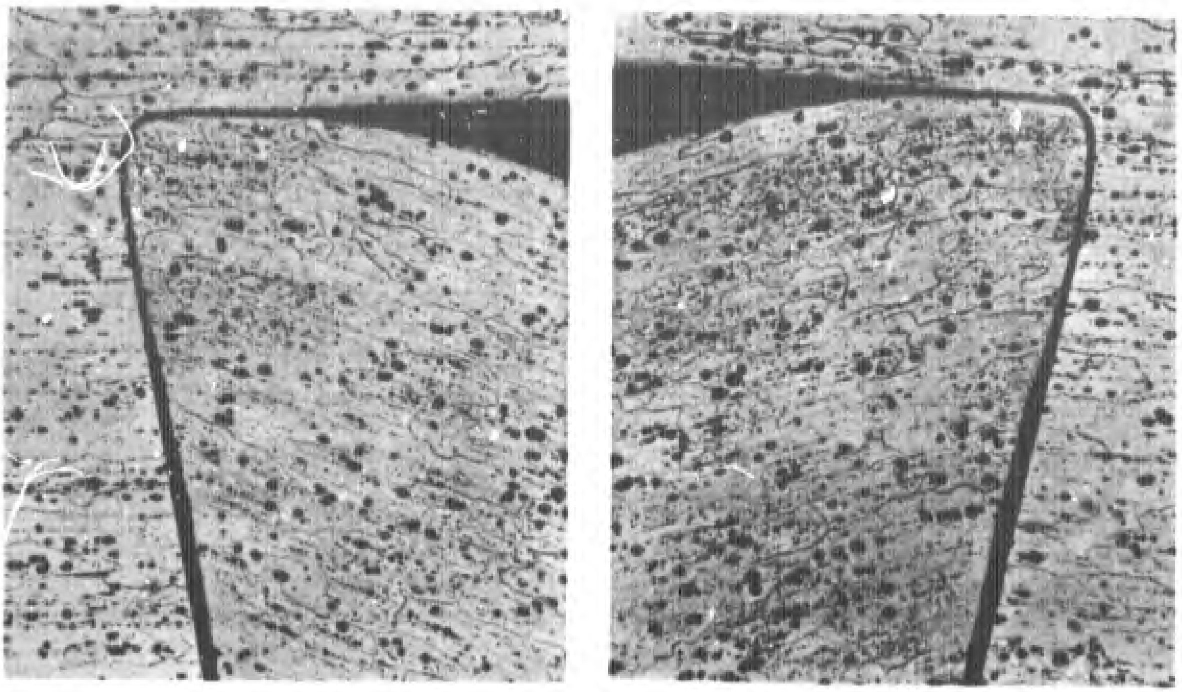
a. Seal With 10° Reverse Angle



100X 3A613 100X 3A611

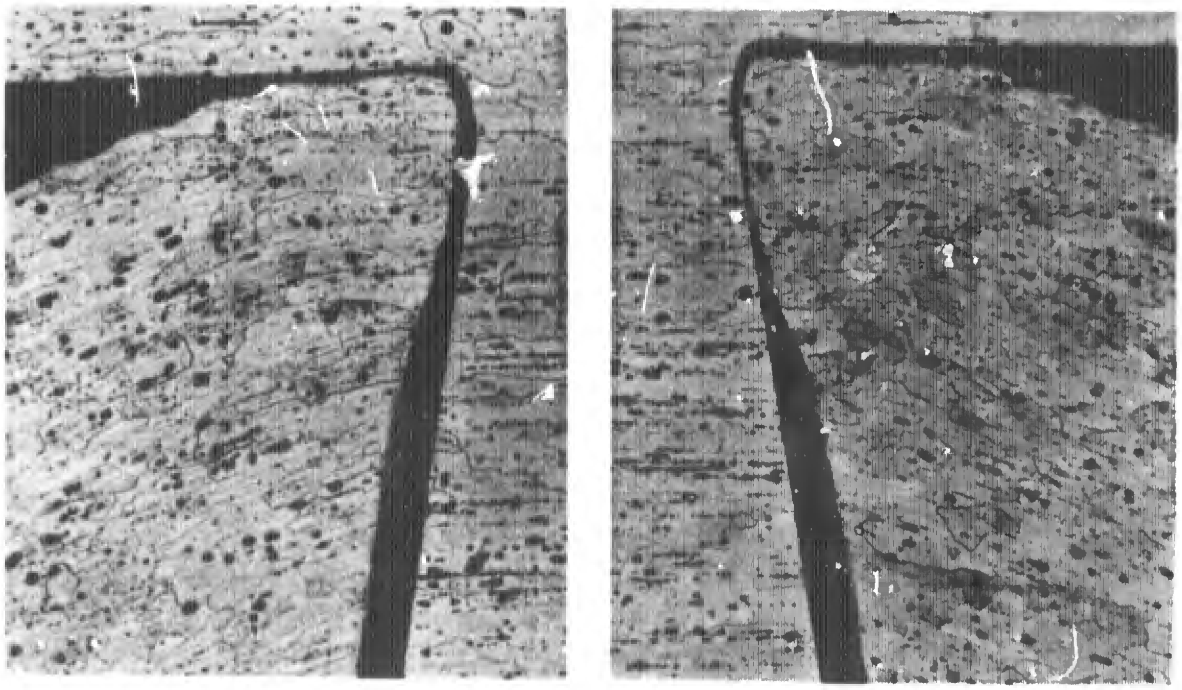
b. Seal Without 10° Reverse Angle

FIGURE 9. PHOTOMICROGRAPHS OF SEAL INTERFACE, ANODIZED FLANGES



100X 3A607 100X 3A609

a. Seal With 10° Reverse Angle



100X 3A612 100X 3A610

b. Seal Without 10° Reverse Angle

FIGURE 10. PHOTOMICROGRAPHS OF SEAL INTERFACE, NONANODIZED FLANGES

Because neither the anodized nor the nonanodized flanges were deformed, it was still uncertain whether anodizing by the Alumilite process could adequately prevent deformation at the flange interface.

Two more sets of flanges were anodized by the Alumilite process. One set of flanges was assembled with a regular 6061-T6 Bobbin seal and one set of flanges was assembled with a 10-degree reverse-angle seal. In both cases the resulting leak rate at 2000 psi was less than 5.6×10^{-9} atm cc/sec. These data are presented in Table 7.

TABLE 7. ASSEMBLY DATA AND LEAKAGE RATES FOR ANODIZED FLANGES

Seal Specimen	Seal Type	Assembly Load, lb Peak 1	Assembly Load, lb Peak 2	Leakage Rate, 10^{-9} atm cc/sec
23	Regular	1080	1365	5.6
24	10-deg.	1185	1245	3.8

A microsection was prepared of Specimen 23 to permit examination of the seal interface. Figure 11 shows the two interfaces. An excellent seal with a contact length ranging from 0.010 to 0.013 inch was created. However, the anodic film was not visible when the specimen was examined under high magnification.

Since the anodic film was supposed to be as hard as Rockwell C55, an attempt was made to verify the presence of this film by measuring the difference in hardness values between the film and the basis metal. Knoop-hardness readings were taken with a 100-g load at the locations shown in the lower left photograph of Figure 11. Two additional readings were taken normal to the flange face. A Knoop-hardness reading of 139 was recorded. Although the hardness values did not indicate the existence of a thick anodic coating, visual inspection of the flanges revealed a cloudy film, and the Knoop-hardness readings indicated that the surface was harder than the core.

Heat-Treated 6061

The use of specially heat-treated 6061 was the fifth method studied. Two approaches were tried -- the use of 6061-T4 and the use of overaged 6061. Although seals made from 6061-T4 sealed at low leakage rates, use of this material was rejected because its mechanical properties are unstable. Seals made from overaged 6061 also sealed at low leakage rates and did not require a redesign of the connector. As the preparation of overaged aluminum proved to be a simple, easily controlled process, and as the mechanical properties of the material were stable, overaged 6061 aluminum was chosen as the final solution to the material selection problem.

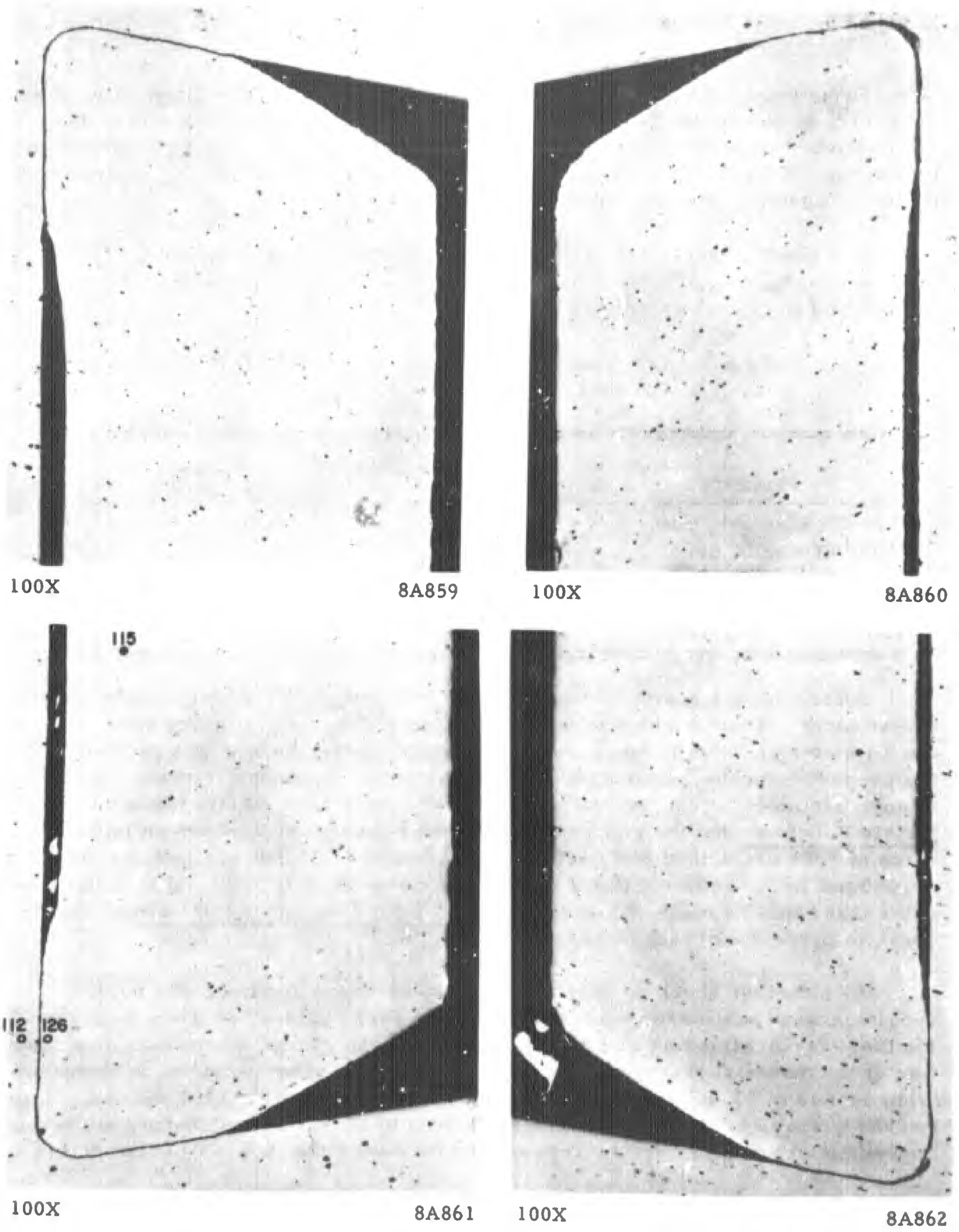


FIGURE 11. SEAL INTERFACE OF CONNECTOR ANODIZED BY ALUMILITE PROCESS

6061-T4

Properties. Since 6061-T4 aluminum is not available commercially, it was necessary to obtain the T4 temper by reheat treatment of 6061-T6. This was accomplished by a full anneal of the 6061-T6 specimen, followed by a solution heat treatment. A hardness of Brinell 58 to 62 resulted. This value approximates the hardness specified for 6061-T4 in the Alcoa Aluminum Handbook.

The tensile properties of the 6061-T4 specimen were measured. The results are compared in Table 8 to the minimum properties specified in Fed. QQ-A-325b and to typical properties of 6061-T6.

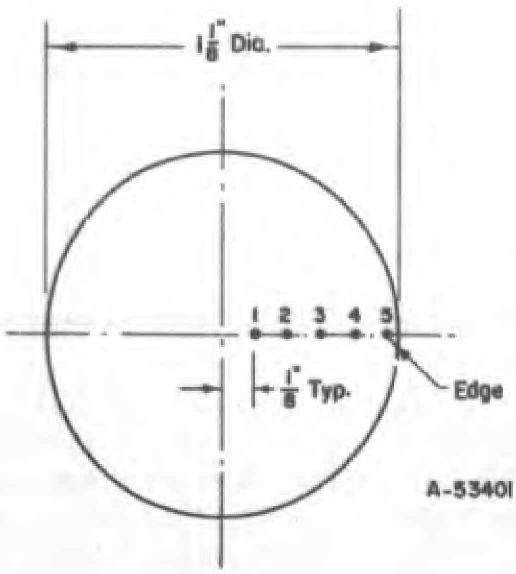
TABLE 8. COMPARISON OF MECHANICAL PROPERTIES OF 6061-T4 AND 6061-T6

Property	6061-T4 Test Results	6061-T4 Fed. QQ-A-325b	6061-T6
Tensile Strength, psi	34,600	30,000	45,000
Yield Strength, psi	16,900	16,000	40,000
Reduction in Area, percent	59.6	--	--
Elongation, percent	32.0	--	12
Hardness (Brinell)	58-62	--	95

After consulting with metallurgists at the Battelle's Columbus Laboratories and at Alcoa, it was decided to further investigate the natural aging properties of the T4 material. Due to age hardening at room temperature it was presumed that the properties of the T4 material would approach asymptotically those of 6061-T6 in approximately 10,000 hours. The American Society of Metals indicates in the Metals Handbook that the yield strength of 6061-T4 increases from an initial value of 9000 psi immediately after heat treatment to 15,000 psi in 1 day and to 20,000 psi in 2 months. After 2 months the material is fairly stable. This indicates that 6061-T4 seals should be stored at least 2 months before use to assure constant properties in all seals.

As a further check on shelf life, Knoop-hardness readings of a 6061-T4 specimen were taken over a 12-week period. These values are given in Figure 12. Further clarification was obtained by measuring the change in mechanical properties of the material over the same time period. The ultimate strength increased from 34,600 to 37,400 psi, the yield strength increased from 16,900 to 19,100 psi, and the average hardness increased from 70.3 to 77.5. These changes represent approximately a 10 percent increase, thus indicating that 6061-T4 is not stable.

Experimental Studies. In addition to the determination of mechanical properties, microsection examinations were made. Five 6061-T4 seals were assembled with 6061-T6 connectors and cross sectioned. Their microsections were



Location	Knoop Hardness (500G) at Indicated Date											
	2-22	3-1	3-8	3-15	3-22	3-29	4-5	4-12	4-19	4-26	5-3	5-10
1	68.9	75.8	72.9	76.7	73.5/79.1	74.1/76.5	79.6	76.0	76.9	77.9	78.0	78.3
2	70.5	75.8	74.1	76.7	70.4/75.0	77.0/70.8	72.2	75.5	74.8	75.4	76.9	80.5
3	71.3	71.9	73.9	72.9	75.0/75.5	70.4/72.2	76.0	75.5	74.8	73.5	74.0	74.9
4	71.9	71.5	72.2	70.2	71.7/76.0	71.3/70.4	72.6	78.0	73.2	75.4	76.5	77.9
5	68.8	71.5	73.7	73.8	71.7/79.1	71.7/73.1	74.5	76.0	75.4	77.4	78.4	76.0
Average	70.3	73.3	73.4	74.1	72.9/76.9	72.9/72.6	75.0	76.2	75.2	75.9	76.8	77.5

FIGURE 12. KNOOP HARDNESS VALUES FOR 6061-T4 ALUMINUM

compared with respect to estimated contact area, quality of the seal, and presence of structural defects. These data are summarized in Table 9.

TABLE 9. RESULTS OF MICROSECTION EXAMINATION OF 6061-T4

Specimen	Tang Thickness, in.	Estimated Seal Width, in. Surface 1	Surface 2	Cracks at Leg Root	Quality of Seal
25	0.115	0.014	>0.015	Yes	Excellent
26	0.105	0.014	0.014	Moderate	Good
27	0.094	0.010	>0.012	Moderate	Good
28	0.084	0.010	>0.010	Moderate	Good
29	0.075	>0.012	>0.008	Moderate	Good/Fair

In all cases the seal was deformed at the interface, whereas the flanges were unaffected, thus indicating that use of T4 material could eliminate the type of gouging caused by 6061-T6 seals. Although Specimen 25 had the best seal, it was not much better than Specimens 26, 27, and 28, which were approximately equal. However, the seal contact width was reduced with a decrease in tang thickness. The 0.008-inch-wide seal on Specimen 29 was only fair, but it appeared that poor machining was the contributing factor. Although rated good, the other sealing surface of Specimen 29 was not quite equivalent to that of the other three specimens, which were also rated as good. Bowing of the tang was evident in Specimen 29 as the inside diameter of the tang was reduced more at the midplane than at the preloading surfaces.

From these examinations it was concluded that, for a material with a yield strength of 17,000 psi, (1) the tang of the 3/4-inch seal should be no less than 0.080 inch thick and (2) the seal-leg thickness should be 0.040 inch.

From a comparison of the assembly data, the expected reduction in inside diameter for a seal with an 0.080-inch tang is about 0.022 inch. On this basis, the inside diameter of a 3/4-inch T4 seal should be 0.700 inch if a smooth flow passage is to result after assembly. The outside diameter would be 1.020 inches. This diameter is 0.036 inch greater than that of the 6061-T6 seal developed under Contract AF 04(611)-9578. As a result, the outside diameter of the plain flange would be increased, the thread size would be increased from 1-3/16 - 18 to 1-1/4 - 18, and the distance across flats on the nut would also increase by at least 1/16 inch.

Overaged 6061

Properties. Two procedures were followed to overage 6061 aluminum. The first procedure involved a two-step operation. Four 6061-T6 specimens were solution heat treated to the T4 temper and then each specimen was aged at a temperature and time as indicated in Table 10. Table 10 also includes the expected tensile yield strengths based on aging curves for 6061*, the actual tensile and yield strengths, and the Knoop hardness of each specimen.

TABLE 10. PROPERTIES OF OVERAGED 6061 ALUMINUM FROM T4 TEMPER

Specimen	Aging Condition Temp, F	Aging Condition Time, hr	Expected Ultimate Strength, psi	Actual Ultimate Strength, psi	Expected Yield Strength, psi	Actual Yield Strength, psi	Actual Knoop Hardness
30	400	8	39,000	42,500	35,000	38,700	112
31	400	16	38,000	41,150	32,000	37,200	102
32	450	8	35,000	33,200	29,000	26,900	81
33	450	16	34,000	33,600	27,000	26,700	80

In the second procedure, the solution heat treatment was eliminated and the four 6061-T6 specimens were overaged by heating them directly at elevated temperatures. Table 11 presents the aging conditions, the expected ultimate and yield strengths, the actual ultimate and yield strengths, and the actual Knoop hardness of each specimen.

TABLE 11. PROPERTIES OF OVERAGED 6061 ALUMINUM FROM T6 TEMPER

Specimen	Aging Condition Temp, F	Aging Condition Time, hr	Expected Ultimate Strength, psi	Actual Ultimate Strength, psi	Expected Yield Strength, psi	Actual Yield Strength, psi	Actual Knoop Hardness
34	425	10	38,000	40,000	33,000	35,200	88
35	425	16	37,000	38,750	32,000	34,000	84
36	450	8	36,000	37,400	30,000	32,000	83
37	450	10	35,500	36,800	29,000	31,400	78

In each case the actual values of strength obtained by the second procedure were only slightly higher than the expected values. Elongation ranged from 17.5 to 20 percent, and the decrease in Knoop hardness matched the decrease in strength. In contrast, the results obtained by the first procedure were less consistent. One possible explanation is the dependence of the aged properties on the solution-treated properties which in turn are highly sensitive to slight changes in temperature and cooling rate.

From these data it was concluded that the one-step procedure is more reliable and more easily controlled. Furthermore, the present fitting dimensions can be maintained if seals are fabricated from material similar to either Specimen 34 or 35 in Table 11.

*Metals Handbook, 8th Edition, American Society of Metals, Vol. 2, p 276, Fig. 11.

Experimental Studies. Regular Bobbin seals and 10-degree reverse-angle seals were fabricated from overaged 6061 aluminum. Two heat treatments were used, 425 F for 10 hours and 425 F for 16 hours. These seals were assembled with 6061-T6 connectors and were leak tested with helium. The results are given in Table 12.

Table 12 shows that seals fabricated from the overaged 6061 alloy sealed helium at leakage rates well below the performance specification. Microsections were prepared from Specimens 38, 40, 41, and 42. Figures 13 and 14 are photomicrographs of Specimens 38 and 42, respectively. For Specimen 38, the seal contact length ranged from 0.009 to 0.014 inch. Although deformation at the flange interface was negligible, a saw-tooth deformation was evident in the flange. This may have been caused during assembly, or it may have resulted from improper machining. Figure 14 indicates that this condition was not evident in Specimen 42. However, in both cases the length and the quality of the seal interface was equal.

TABLE 12. ASSEMBLY DATA AND LEAKAGE RATES FOR OVERAGED 6061 SEALS

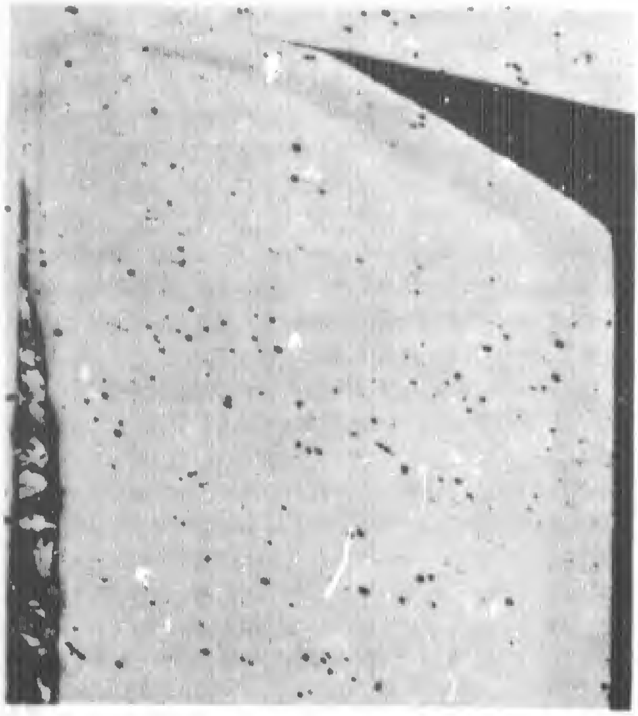
Seal Specimen	Seal Type	Overaged Condition Temp., F	Overaged Condition Hr	Assembly Load, lb Peak 1	Assembly Load, lb Peak 2	Change in ID, in.	Leakage at Designated Pressure, 10^{-9} atm cc/sec 1200 Psi	Leakage at Designated Pressure, 10^{-9} atm cc/sec 2000 Psi
34	Regular	425	10	1251	1374	0.016	(b)	--
35	Regular	425	16	1230	1479	0.018	(b)	--
36	10-deg.	425	10	1194	1266	0.016	0.49	--
37	10-deg.	425	16	1155	1260	0.016	0.41	--
38	Regular	425	10	1224	1350	(a)	--	(b)
39	Regular	425	16	1140	1400	0.016	--	16.0
40	10-deg.	425	10	1185	1250	(a)	--	0.41
41	10-deg.	425	16	1200	1260	(a)	--	(b)
42	Regular	425	16	1110	1400	(a)	--	0.16

(a) Not measured; specimens prepared as microsections.
(b) Leaks were less than mass-spectrometer sensing capability.

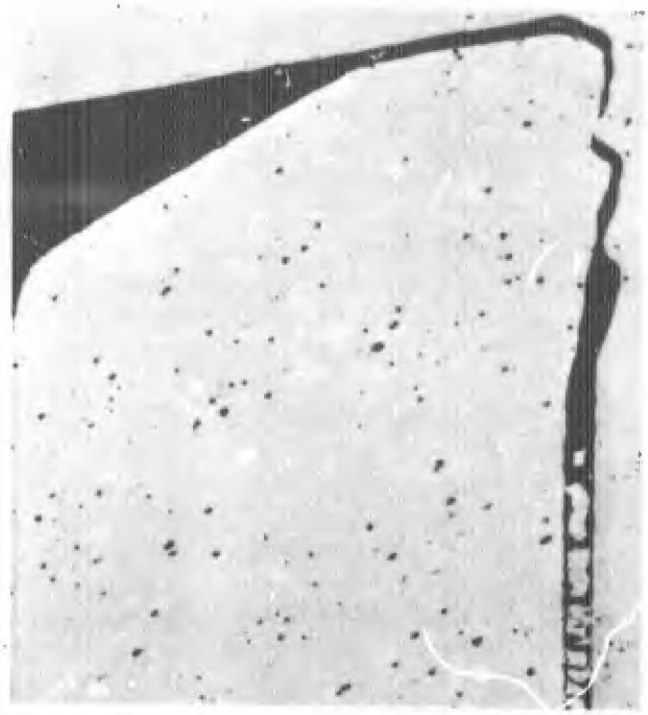
Figures 15 and 16 are photomicrographs of Specimens 40 and 41, respectively. The saw-tooth deformation was again evident in Specimen 40, and the seal interface and contact length in Specimen 41 appeared to be more uniform.

QUALIFICATION TESTS

Qualification tests included repeated assembly, stress-reversal bending, and determination of thermal gradient.



100X 5A840



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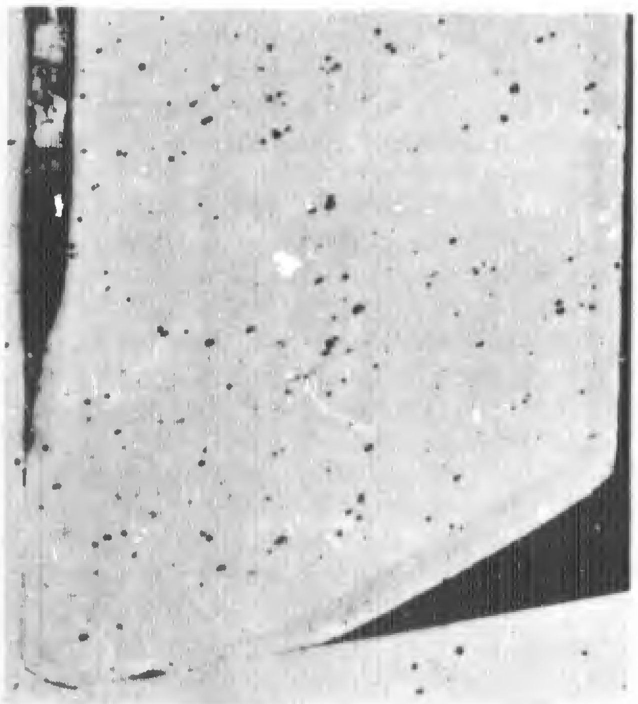
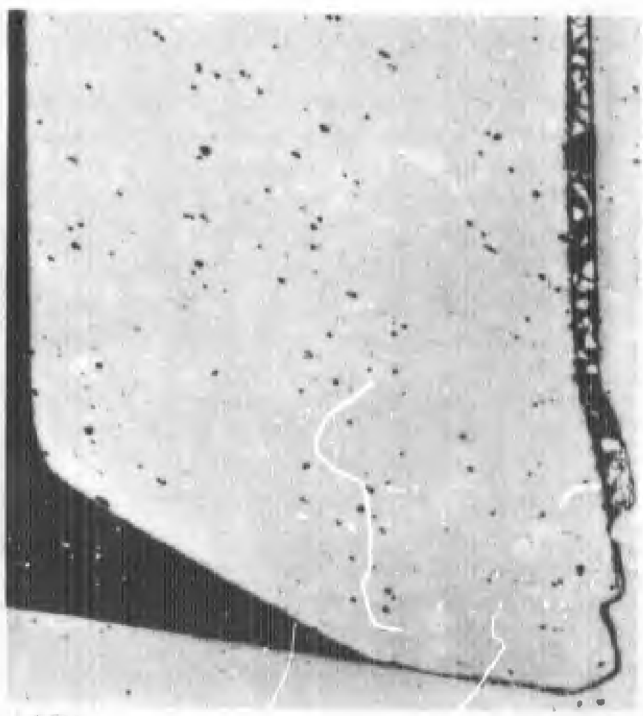


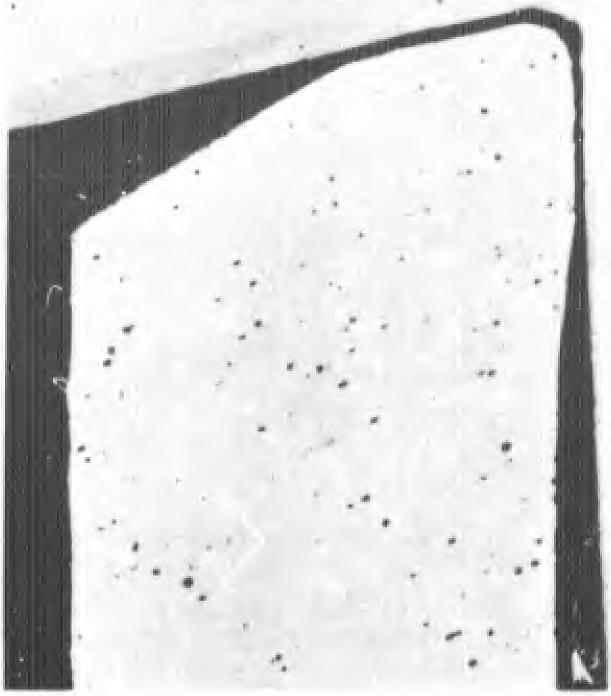
100X 5A838



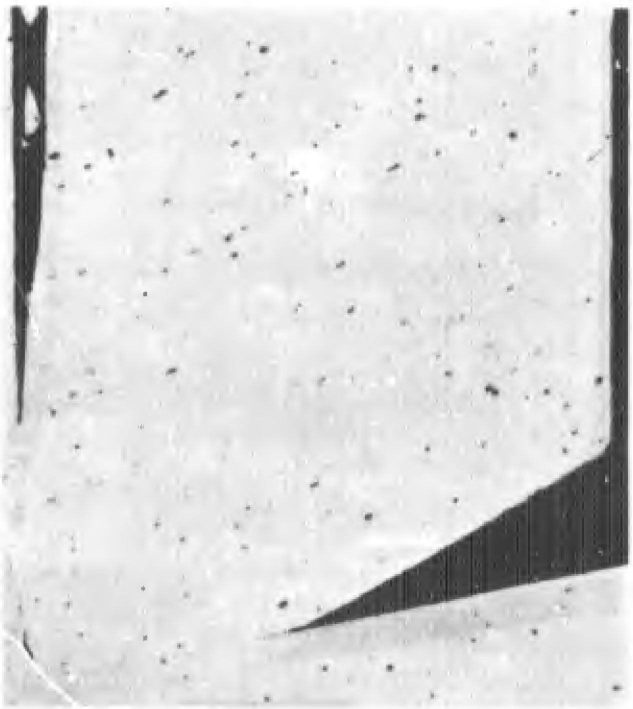
100X 5A839

FIGURE 13. SEAL INTERFACE - SPECIMEN 38

100X 5A848



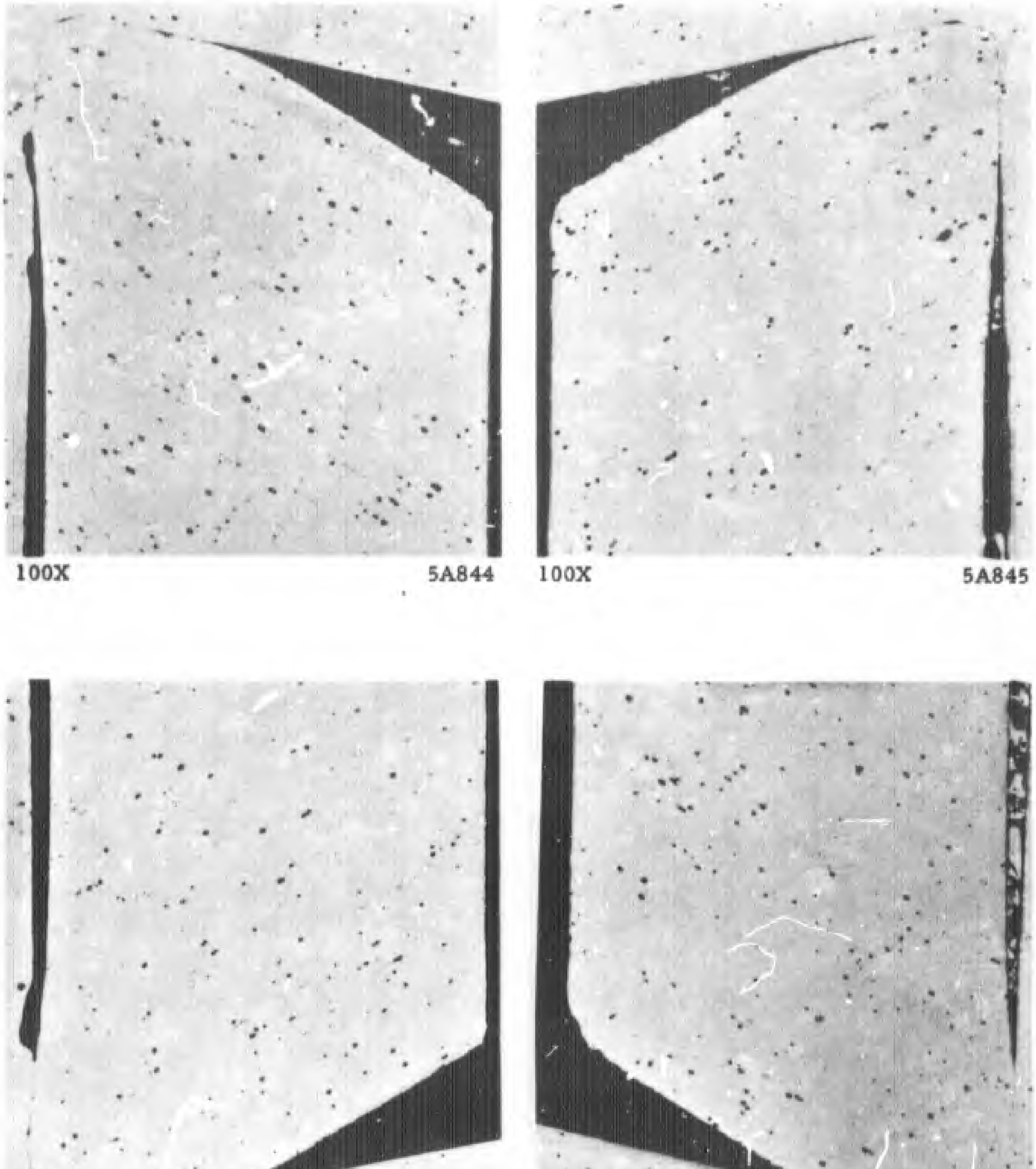
100X 5A849



100X 5A846

100X 5A853

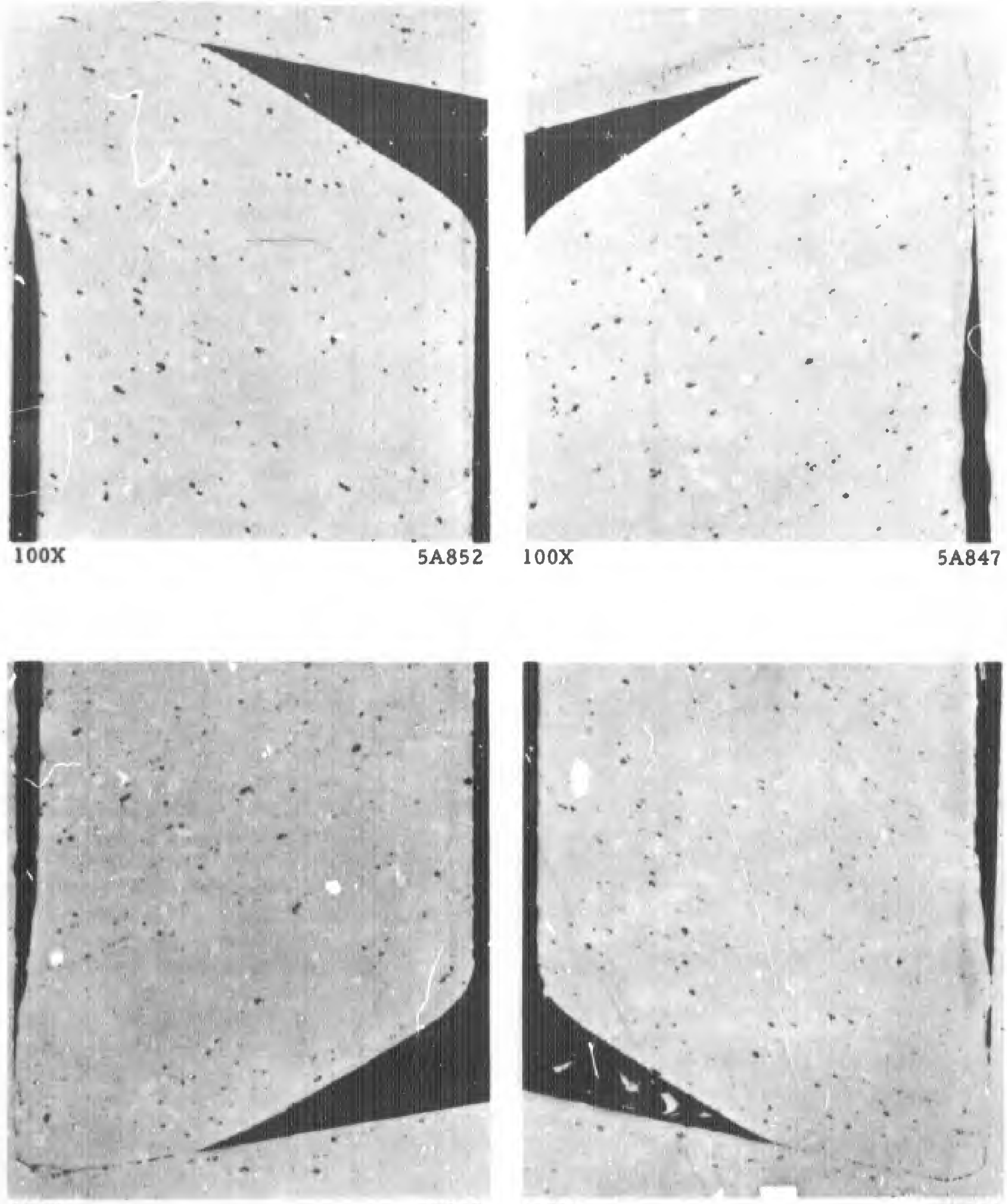
FIGURE 14. SEAL INTERFACE – SPECIMEN 42



100X 5A844 100X 5A845

100X 5A842 100X 5A843

FIGURE 15. SEAL INTERFACE - SPECIMEN 40



100X 5A852 100X 5A847

100X 5A850 100X 5A851

FIGURE 16. SEAL INTERFACE - SPECIMEN 41

Repeated Assembly

Repeated-assembly tests were conducted with four -6 and four -12 connectors. Each connector was reassembled 25 times with overaged aluminum seals A total of 120 seals were leak tested, and in only one did the leak rate exceed 7×10^{-7} atm cc/sec.

Leakage Measurements

Specimens 6RA1, 6RA2, 12RA1, and 12RA2* were leak tested with helium after each reassembly, whereas Specimens 6RA3, 6RA4, 12RA3, and 12RA4 were leak tested after the 5th, 10th, 15th, 20th, and 25th assembly. The assembly torques were 150 lb-in. and 450 lb-in. for the -6 and -12 connectors, respectively, and the test pressures were 1500 psi and 1125 psi, respectively. A lubricant consisting of a 50/50 mixture of Lubriplate and MoS_2 powder was applied periodically to the threads and to the back face of the plain flange.

The leakage measurements are presented in Tables 13 and 14. Specimens 6RA1, 6RA3, 12RA1, and 12RA3 were assembled with regular seals, whereas the other four specimens were assembled with 10-degree-angle seals. Of the 120 seals leak tested, 81.6 percent had leakage rates less than 10^{-8} atm cc/sec, and 17.5 percent had leakage rates between 10^{-8} and 7×10^{-7} atm cc/sec. Only one seal exceeded the allowable leak rate, but the leak rate of 1.9×10^{-6} atm cc/sec was less than 5 cc/month. This occurred during the tenth assembly of Specimen 6RA1.

Deformation

Deformation of the nuts and threaded flanges was insignificant even after 25 assemblies and would not have prevented continued use of the connector. Galling at the threads and at the back face of the plain flange was visible but even in the worst case it was possible to reassemble a lubricated connector without difficulty. The amount of galling generated was reduced when the bearing surfaces were lubricated more often. To determine the effect of repeated assembly on the flange sealing face, seven connectors were sectioned after the 25th assembly. Photomicrographs of Specimens 6RA1, 6RA2, 12RA1, and 12RA2 are given in Figures 17 and 18.

As shown in Figure 17, neither the regular nor 10-degree angle seal caused noticeable deformation of the flange. In both cases the sealing interface was equivalent in terms of length and degree of conformity. However, as shown in Figure 18, the resulting seals for the -12 connector were not equivalent nor were they of the same quality as those attained with the -6 connectors. Although the

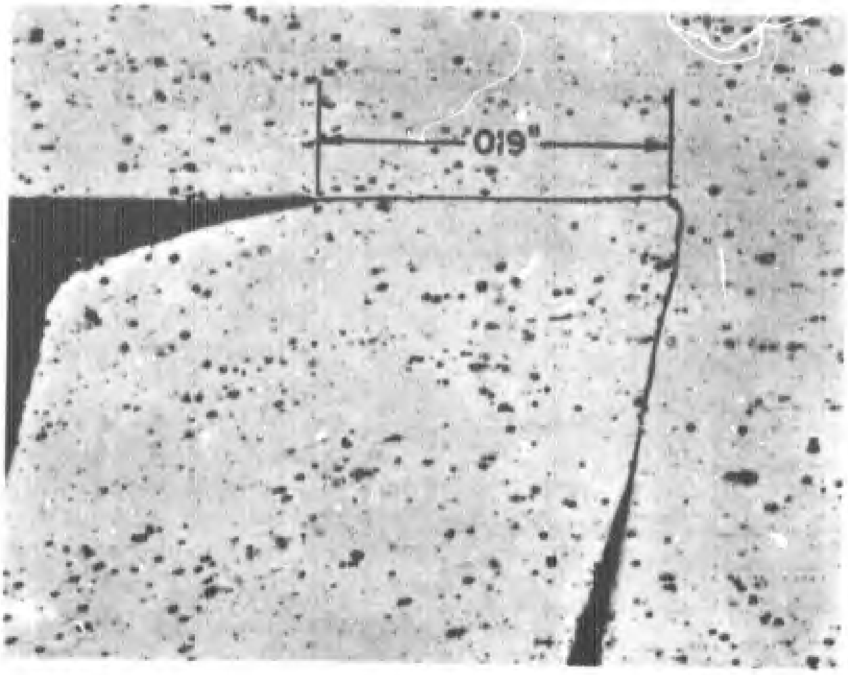
*The first number designates the tube size (6 = 3/8 inch and 12 = 3/4 inch). RA designates a repeated assembly test and the last number designates the specimen number. Thus, 6RA1 is the first 3/8-inch connector used in the repeated assembly test.

TABLE 13. RESULTS OF REPEATED ASSEMBLY TEST FOR -6 AND -12 CONNECTORS (RA1 AND RA2)

Assembly Number	Estimated Leakage Rates for Indicated Conditions, 10^{-7} atm cc/sec			
	-6 Connector		-12 Connector	
	6RA1 Regular Seal	6RA2 10-Degree Seal	12RA1 Regular Seal	12RA2 10-Degree Seal
1	0.003	0.005	0.101	0.060
2	0.012	0.005	0.069	0.035
3	0.291	0.006	0.028	0.023
4	0.003	0.130	0.018	0.015
5	0.015	2.035	0.014	0.041
6	0.020	0.006	0.035	0.017
7	0	0	0.020	0.017
8	0.023	0.008	0.015	0.015
9	0.125	0.009	0.018	0.015
10	19.100	0.008	0.015	0.026
11	0.029	0.015	0.012	0.015
12	0.011	0.425	0.012	0.012
13	0.029	0.263	0.008	0.009
14	0.064	1.867	0.008	0.009
15	0.038	0.336	0.008	0.008
16	0.032	0.150	0.006	0.006
17	0.226	0.101	0.006	0.006
18	0.035	0.067	0.006	0.089
19	0.699	0.024	0.012	0.020
20	0.225	0.032	0.052	0.018
21	0.136	0.051	0.014	0.015
22	0.226	0.041	0.018	0.015
23	0.090	0.024	0.017	0.015
24	0.034	0.021	0.012	0.014
25	0.023	0.018	0.015	0.015

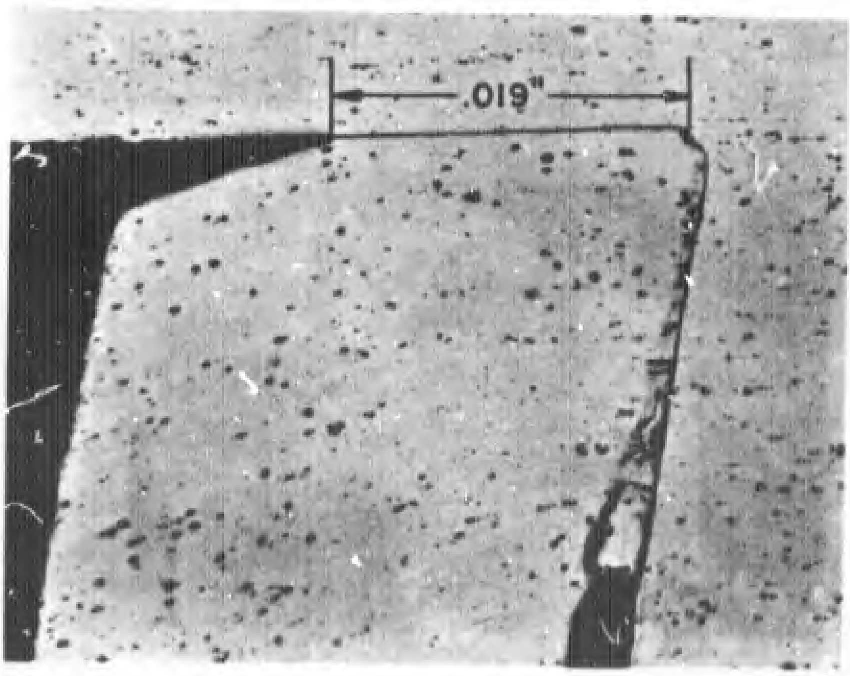
TABLE 14. RESULTS OF REPEATED ASSEMBLY TEST FOR -6 AND -12 CONNECTORS (RA3 AND RA4)

Assembly Number	Estimated Leakage Rates for Indicated Conditions, 10^{-7} atm cc/sec			
	-6 Connector		-12 Connector	
	6RA3 Regular Seal	6RA4 10-Degree Seal	12RA3 Regular Seal	12RA4 10-Degree Seal
5	0.028	0.040	0.157	0.263
10	0.028	0.121	0.110	0.161
15	0.023	0.018	0.080	0.088
20	0.028	0.077	0.090	0.053
25	0.032	0.051	0.027	0.039



100X 1B632

a. Specimen 6RA2



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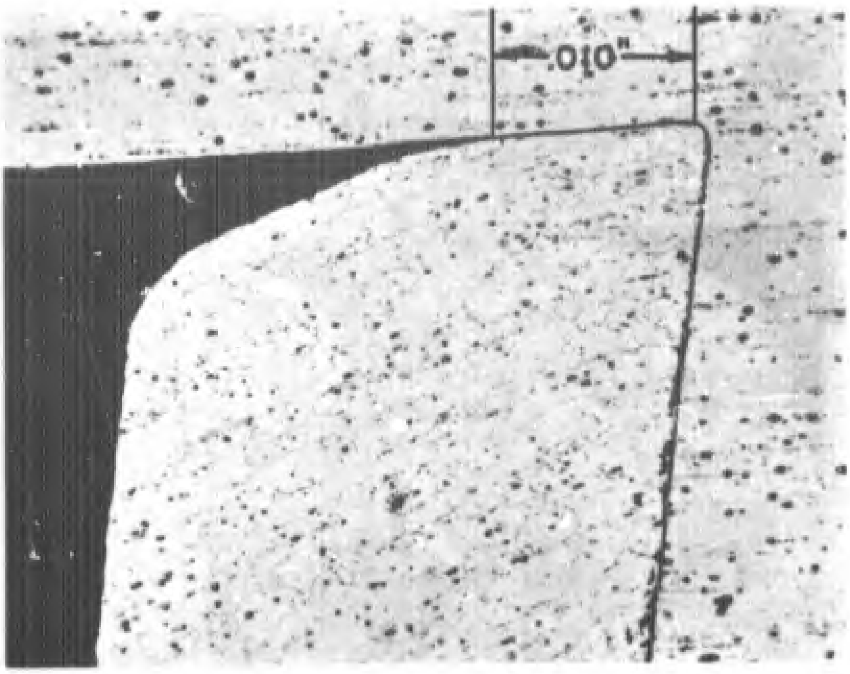
b. Specimen 6RA1

FIGURE 17. SEAL INTERFACE OF 3/8-INCH CONNECTOR



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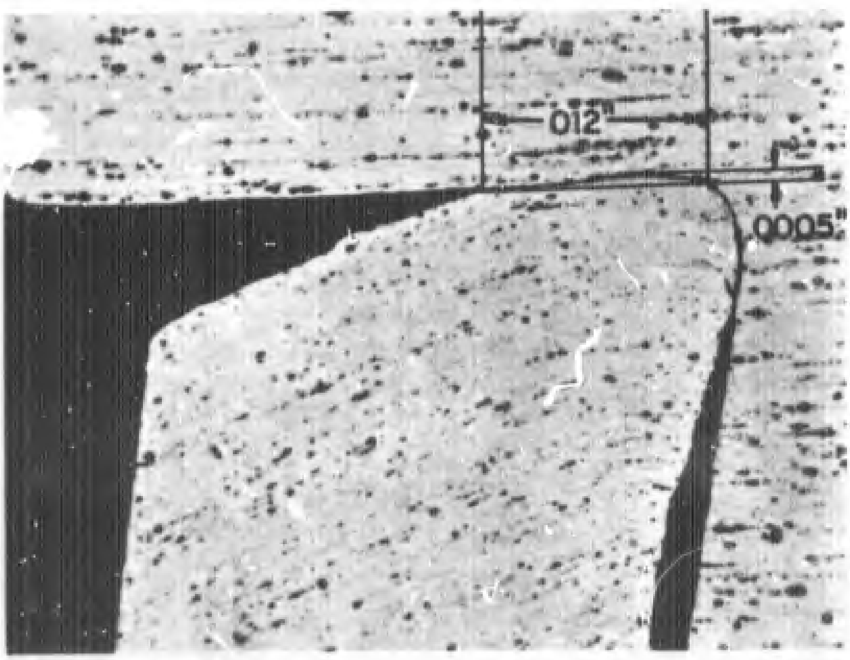
a. Specimen 12RA2



100X 1B627

b. Specimen 12RA1

FIGURE 18. SEAL INTERFACE OF 3/4-INCH CONNECTOR

regular seal caused some deformation of the flange (0.0005 inch) and the 10-degree seal did not, the length of the seal interface was greater with the regular seal.

Conclusions

Except in one case, no apparent deformation of the flange sealing face resulted from 25 reassemblies of the connector. Even in that one case the deformation was negligible. Furthermore, leakage rates were well below the allowable leak rate in all but one case. Therefore, in all respects, the use of regular and 10-degree seals of overaged aluminum resulted in a high-quality seal and equivalent performance. Because the regular seal is easier to machine and easier to inspect, it was selected as the final design.

Stress-Reversal Bending

Stress-reversal bending tests were conducted with two -6 and two -12 connectors.

Experimental Procedure

The equipment used to simulate stress-reversal conditions in the unions is shown in Figure 19. The evaluation procedure consisted of the following steps:

(1) The plain flange was welded to the connecting rod, and the threaded flange was welded to the rigid support.

(2) The seal was inserted and the connector was assembled.

(3) The bellows vacuum chamber was assembled.

(4) The connecting rod was inserted in the bearing, and the entire assembly was bolted to the fixture.

(5) The eccentric was set at the proper offset as measured by the strain gage.

(6) The connector was pressurized to proof pressure and heated to maximum operating temperature. The leakage was measured.

(7) The bending moment was applied by means of the rotating eccentric for at least 300,000 cycles at a rate of 100,000 cycles per hour. The operating pressure and maximum operating temperature were maintained.

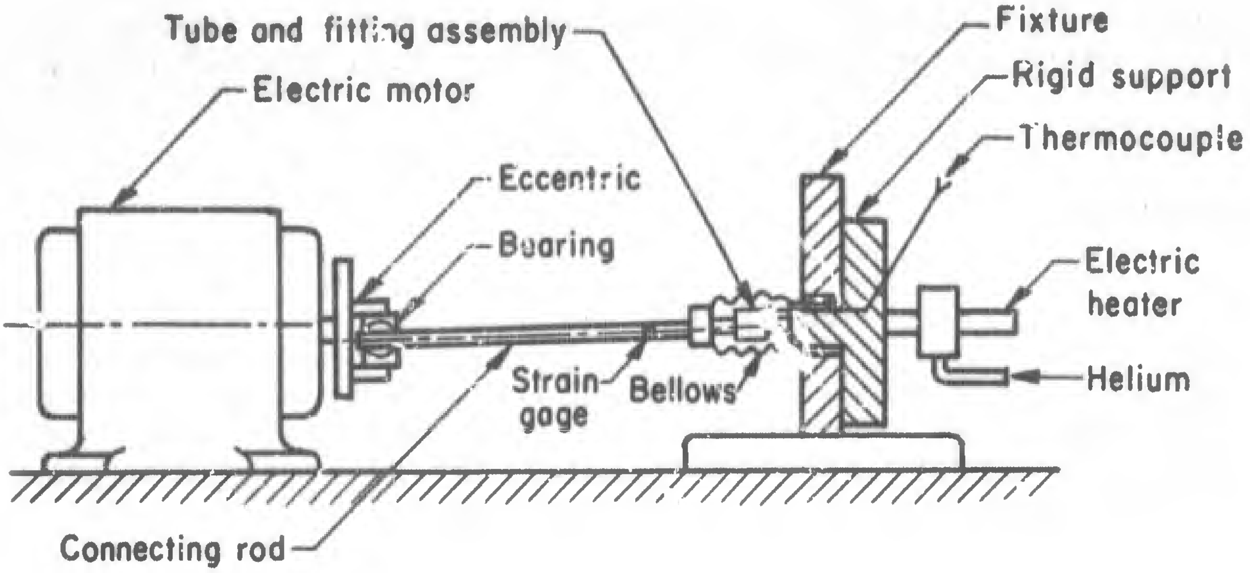


FIGURE 19. EXPERIMENTAL ARRANGEMENT FOR STRESS-REVERSAL-BENDING EVALUATION

The -6 connectors were tightened with a torque of 15 lb-ft and were pressurized with helium at 1000 psi. -12 connectors were tightened with a torque of 41 lb-ft and were pressurized with helium at 750 psi. Bending moments of 35.3 in.-lb and 192 in.-lb were applied to the -6 and -12 connectors, respectively. Table 15 presents the results of these tests after 300,000 cycles.

TABLE 15. RESULTS OF THE 300,000-CYCLE STRESS-REVERSAL-BENDING TEST FOR -6 AND -12 CONNECTORS

Specimen	Bending Moment, in.-lb	Estimated Leakage Rate, 10^{-8} atm cc/sec
6SR1	35.3	4.4
6SR2	35.3	0.096
12SR1	192.1	1.9
12SR2	192.1	0.78

Specimens 6SR2 and 12SR2 were subsequently tested to failure. In both cases the failure occurred at the weld attachment to the tubing. Specimen 12SR2 failed after a total of 360,000 cycles. The leakage rate just prior to failure was 2.34×10^{-8} atm cc/sec.

After Specimen 6SR2 had withstood 1,940,000 cycles, the bending moment was increased by 25 percent. Failure occurred after an additional 1,950,000 cycles. The leakage rate prior to failure was 1.06×10^{-8} atm cc/sec.

Thermal Gradient

Experiments were conducted to evaluate the capability of the connector to function satisfactorily at maximum expected temperature gradients.

Procedure

Initially, four unions were tested with the equipment shown in Figure 20. Although each connector sealed helium within the required limit at room temperature, none sealed helium at -320 F. The vacuum chambers were removed and it was found that the residual nut torque had been reduced by as much as 26 percent. In all probability the leakage and reduced torque came about because of the heat, distortion, and residual stresses engendered by the process of welding the vacuum chamber to the tubing after the connectors were assembled.

The thermal-gradient tests were continued with four other connectors and with a vacuum chamber of different design, shown in Figure 21. A bath of boiling water was used to attain the upper temperature extreme and a bath of liquid nitrogen was used to attain the lower temperature extreme. The test was conducted by immersing the connector and vacuum chamber assembly first in one liquid and then in the other. Three such cycles were completed with each connector.

Because the vacuum-chamber arrangement shown in Figure 21 tended to diminish the severity of the thermal shock during the transient phase, the period of exposure at both temperature extremes was extended to permit an adequate evaluation of the steady-state thermal effects.

Experimental Results

Four connectors, 6TG1, 6TG2, 12TG1, and 12TG2, were tested. Connectors 6TG1 and 12TG1 were tightened to the maximum assembly torque of 17.8 and 51 lb-ft, respectively. Connectors 6TG2 and 12TG2 were tightened to the minimum assembly torques of 15.5 and 46 lb-ft. The -6 connectors were pressurized with 1000-psi helium and the -12 connectors were pressurized at 750 psi. Connectors 6TG1 and 12TG1 were initially submerged in boiling water, and connectors 6TG2 and 12TG2 were initially submerged in liquid nitrogen. Table 16 presents the results of these tests.

As indicated in Table 16, the maximum leakage rate of 4.00×10^{-8} atm cc/sec occurred with connector 6TG1 during the first cold cycle. Thereafter, the leakage rate decreased. Likewise, the leakage rate for connectors 12TG1 and 12TG2 decreased after a maximum leakage rate was measured during the first thermal cycle. For connector 6TG2 the leakage rate steadily increased from 1.1×10^{-8} atm cc/sec during the first cycle to 3.68×10^{-8} atm cc/sec during the final cycle. This increase may have been due to an accumulation of dirt in the

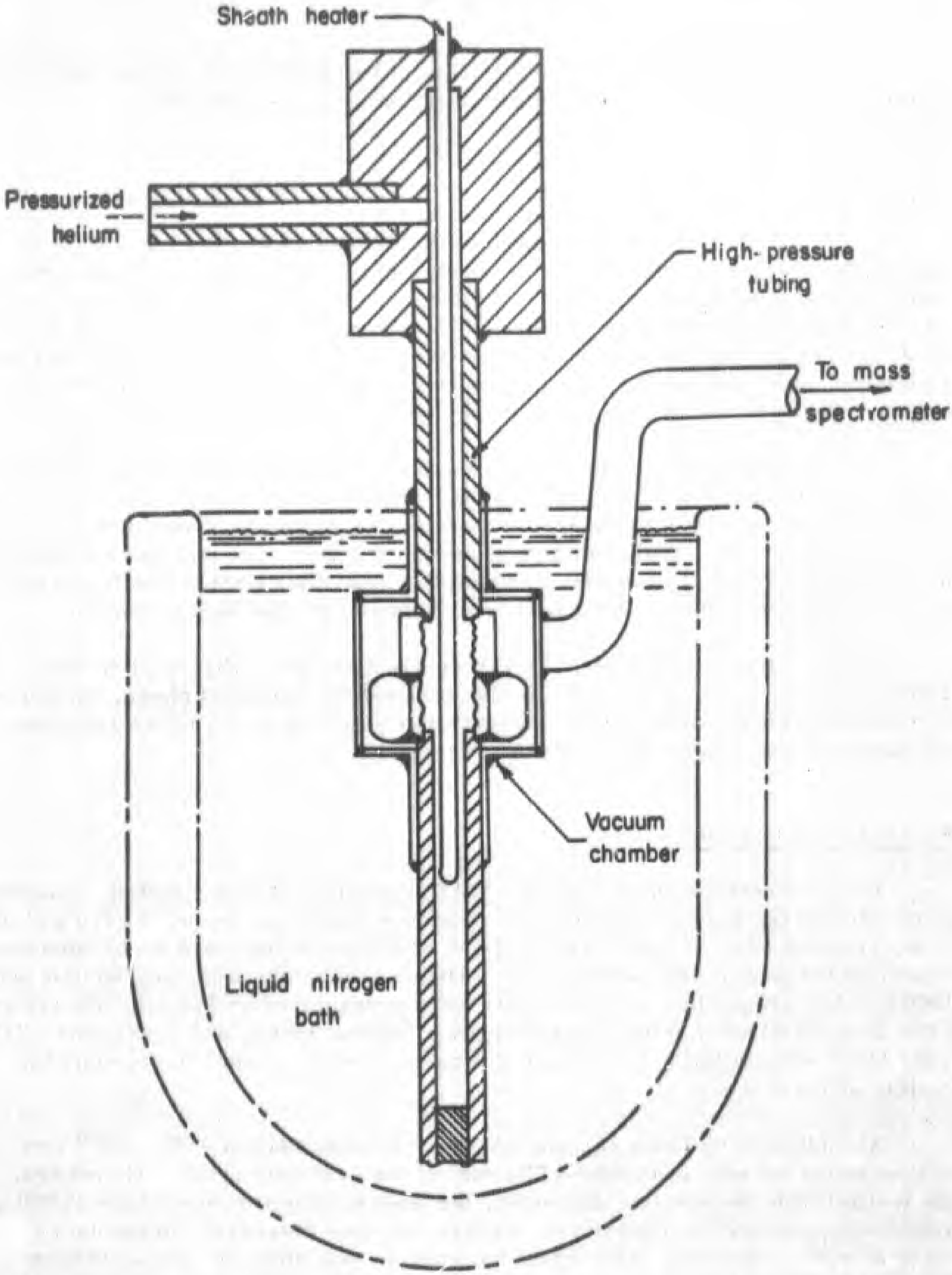


FIGURE 20. INITIAL EXPERIMENTAL ARRANGEMENT FOR MEASURING EFFECTS OF THERMAL GRADIENTS

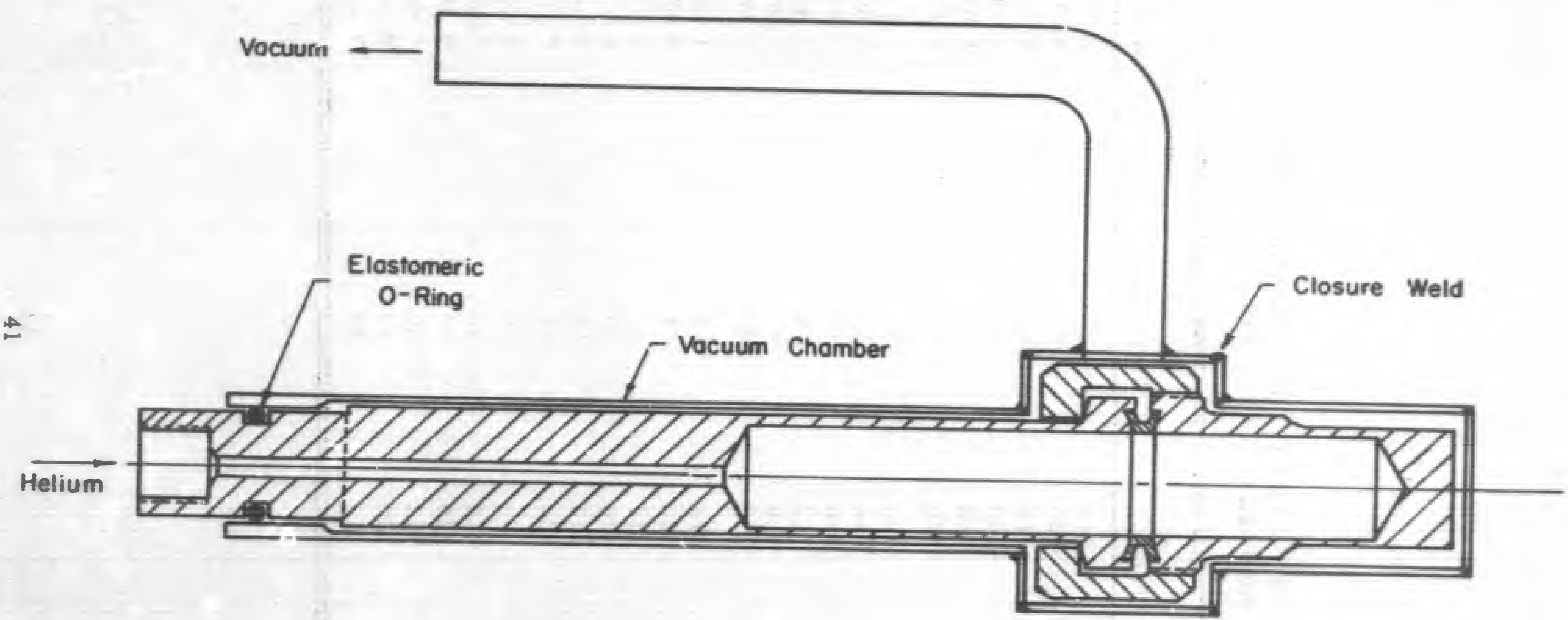


FIGURE 21. REVISED EXPERIMENTAL ARRANGEMENT FOR MEASURING EFFECTS OF THERMAL GRADIENTS

TABLE 16. RESULTS OF THE THERMAL-GRADIENT TEST FOR -6 AND -12 CONNECTORS

Specimen	Fluid Temp., F	Elapsed Time, min	Estimated Leakage Rate, 10^{-8} atm cc/sec
6TG1	212	27	2.37
	-320	50	4.00
	212	60	2.92
	-320	35	1.73
	212	30	1.62
	-320	40	1.40
6TG2	-320	35	1.10
	212	35	1.60
	-320	30	1.84
	212	40	2.46
	-320	45	2.46
	212	40	3.68
12TG1	212	50	1.08
	-320	52	0.82
	212	55	0.91
	-320	55	0.69
	212	57	0.61
	-320	53	0.57
12TG2	-320	30	0.90
	212	42	0.61
	-320	40	0.48
	212	40	0.43
	-320	40	0.35
	212	40	0.35

cold trap of the mass spectrometer, which would tend to show an increased leakage rate due to outgassing of entrapped helium.

REVISIONS OF MS SPECIFICATIONS

As a result of the Phase I effort it was necessary to revise the following tentative specifications and standards: MIL-F-27417, MS27850, and MS27860.

MIL-F-27417, Fittings, Rocket Engine, Fluid Connection

To revise MIL-F-27417 it was necessary to determine the mechanical properties of overaged 6061 aluminum and to change the wording of certain paragraphs.

Mechanical Properties of Overaged 6061 Aluminum

Test specimens were prepared from Alcoa-, Reynolds-, and Kaiser-produced 6061-T6 aluminum. Three specimens were prepared from 6061-T6, and six were prepared from overaged 6061 aluminum (6061-T6 aluminum overaged for 10 hours at 425 F). The mechanical properties of 6061-T6 aluminum are given in Table 17 and the properties of overaged 6061 aluminum are given in Table 18.

Tensile and yield strengths of the 6061-T6 material ranged from high values of 50,600 psi and 43,400 psi, respectively, for specimen Reynolds-1 to low values of 45,500 psi and 40,500 psi, respectively, for specimen Kaiser-1. The values for specimen Alcoa-1 are approximately equal to the average values for the three specimens. Further comparison of the elongation and reduction in area shows that specimen Kaiser-1 is slightly more ductile than the remaining two specimens.

Tensile and yield strengths of the overaged 6061 material ranged from high values of 42,600 psi and 35,700 psi, respectively, for specimen Reynolds-2 to low values of 39,300 psi and 34,600 psi, respectively, for specimen Kaiser-3. The average yield strength of the overaged 6061 was 83.8 percent of the 6061-T6 material and the average hardness was 82.5 percent. The elongation and the modulus of elasticity are approximately equal for both materials, but the reduction in area for the overaged material was 13 percent greater.

TABLE 17. MECHANICAL PROPERTIES OF 6061-T6 ALUMINUM

Specimen	Ultimate Tensile Strength, psi	Yield Strength (0.2 Percent Offset), psi	Elongation, percent	Reduction in Area, percent	Brinell Hardness (500 Kg)	Modulus of Elasticity, 10^6 psi
Alcoa-1	47,300	42,100	17	49.0	91	11.2
Kaiser-1	45,500	40,500	18	51.4	86	10.4
Reynolds-1	50,600	43,400	15	47.6	92	10.8
Average	47,800	42,000	16.7	49.3	89.7	10.8

TABLE 18. MECHANICAL PROPERTIES OF OVERAGED 6061 ALUMINUM

Specimen	Ultimate Tensile Strength, psi	Yield Strength (0.2 Percent Offset), psi	Elongation, percent	Reduction in Area, percent	Brinell Hardness (500 Kg)	Modulus of Elasticity, 10^6 psi
Alcoa-2	40,400	35,300	16	59.2	76	10.4
Alcoa-3	40,800	35,600	17	49.0	71	9.6
Kaiser-2	39,400	34,700	17	59.2	74	10.0
Kaiser-3	39,300	34,600	15	57.0	74	10.4
Reynolds-2	42,600	35,700	16	56.3	74	10.8
Reynolds-3	41,800	35,200	15	53.7	67[a]	10.4
Average	40,716	35,183	16	55.7	74	10.3

(a) Low reading probably due to poor surface. Not included in average.

Proposed Revisions to MIL-F-27417

The proposed revisions to MIL-F-27417 are presented below.

SPECIFICATIONS

Military

Delete AMS 4156

3.2.1 Heat Treatment.

3.2.1.1 Aluminum alloy. Mechanical properties and final temper of aluminum fittings shall be as specified in paragraphs 3.2.1.1.1 and 3.2.1.1.2. All heat treatment shall be done prior to finish machining.

3.2.1.1.1 Nuts, flanges and forgings. Unless otherwise specified, aluminum material used for nuts, flanges and forgings shall conform to AMS 4127. Final temper shall be T6.

3.2.1.1.2 Seals. Unless otherwise specified, aluminum alloy material used for seals shall conform to AMS 4127, and shall be solution heat treated, overaged, and stabilized prior to finish machining. Overaging shall consist of heating AMS 4127 temper T6 at 425 $^{+10}_{-0}$ degrees Fahrenheit in a vacuum or electric furnace for 10 hours and air cool. The overaged aluminum material shall have the following properties:

(a)	Ultimate tensile strength, psi	39,000 to 43,000
(b)	0.2% yield strength, psi	34,000 to 36,000
(c)	Modulus of elasticity, x 10^6 psi	9.5 to 11
(d)	Hardness, 500 kg Brinell	70 to 76
(e)	Elongation in 2-in. length, percent	15 to 17

Military Standard MS27850

Because of the helium leakage and the lower residual torque recorded during the first series of thermal-gradient tests, the wrench torque values listed in MS27850 were reexamined. The latest studies with larger diameter seals, in Phase II, indicate that the minimum seal load should be equal to 40 percent of the seal-seating load. Therefore, the preload diagrams were revised, and it is recommended that the table in MS27850 be changed in accordance with Table 19, below.

TABLE 19. REVISED INSTALLATION DATA

TUBING TO TUBING FITTING INSTALLATION

DASH NO. REF.	TUBING OD INCHES	WRENCH TORQUE FOR TIGHTENING CORROSION-RESISTANT STEEL FITTINGS (LB-IN) MS27851		WRENCH TORQUE FOR TIGHTENING ALUMINUM ALLOY FITTINGS (LB-IN) MS27856	
		MINIMUM	MAXIMUM	MINIMUM	MAXIMUM
-2	1/8	75	90	--	--
-3	3/16	--	--	--	--
-4	1/4	200	225	100	125
-5	5/16	--	--	--	--
-6	3/8	380	435	175	200
-8	1/2	490	555	280	320
-10	5/8	--	--	--	--
-12	3/4	1240	1400	530	600
-16	1	2300	2580	1070	1200

Military Standard MS27860

Although the use of overaged 6061 aluminum has no effect on the overall dimensions of the Bobbin seal, some design revisions resulted from the Phase I effort. For the smaller diameter seals, this involved the specification of a radius and an undercut at the junction of the seal leg and tang. These revisions are shown in Figure 22.

SUMMARY OF RECOMMENDATIONS

As a result of the Phase I effort, the following recommendations have been formulated:

(1) For 6061-T6 aluminum connectors, the Bobbin seal should be made from 6061 aluminum overaged from the T6 condition at 425 F for 10 hours

(2) MIL-F-27417, MS27850, and MS27860 should be revised as indicated in this report

(3) Aluminum AFRPL connectors of the latest design should be included in an industry-evaluation or an Air Force evaluation similar to those programs now being carried out with stainless steel connectors.

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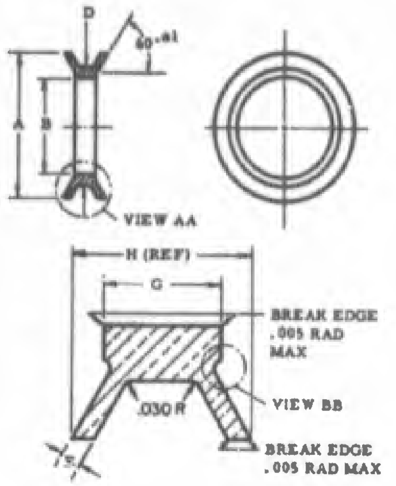

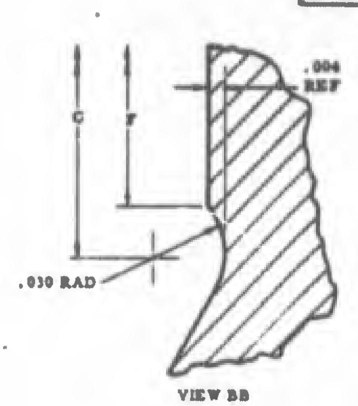

MS PART NO.	TUBE OD	A DIA +.000 -.002	B DIA +.002 -.000	C DIA REF	D DIA +.000 -.002	E ±.001	F DIA REF	G ±.002	H REF	WEIGHT
MS27860-02	1/8									
MS27860-03	3/16									
MS27860-04	1/4	.468	.220	.268	.298	.027	.238	.150	.248	
MS27860-05	5/16									
MS27860-06	3/8	.606	.340	.406	.436	.027	.376	.150	.248	
MS27860-08	1/2	.722	.440	.522	.552	.027	.492	.150	.248	
MS27860-10	5/8									
MS27860-12	3/4	.984	.680	.794	.824	.027	.[illegible]64	.150	.248	
MS27860-16	1	1.280	.920	1.080	1.110	.027	1.050	.150	.248	

1. ALL DIAMETERS SHALL BE CONCENTRIC WITHIN .002 FIR.
2. DIMENSION ARE TO SHARP CORNERS UNLESS OTHERWISE SPECIFIED.
3. MATERIAL: ALUMINUM ALLOY, AMS 4127, OVERAGED PER MIL-F-27417 PARAGRAPH 3.2.1.1.2
4. CLEANING: FINISHED PARTS SHALL BE CLEANED PER MIL-F-XXXX.
5. SURFACE ROUGHNESS: AS 291. UNLESS OTHERWISE SPECIFIED, SURFACES TO BE 32 MICROINCHES (AA).
6. BREAK SHARP EDGES .003-.015 UNLESS OTHERWISE SPECIFIED.
7. DIMENSIONS IN INCHES. UNLESS OTHERWISE SPECIFIED: TOLERANCES, LINEAR DIMENSIONS ±.010, ANGULAR DIMENSIONS ±50.
8. RUBBER STAMP P/N AND MFR IDENT ON CONTAINER OR PACKAGING PER AS [illegible]78-30.
9. DO NOT USE UNASSIGNED PART NUMBERS.

REVISED

APPROVED

TENTATIVE

P.A. Other Cust	TITLE SEAL, AMS4127	MILITARY STANDARD MS 27860
PROCUREMENT SPECIFICATION MIL-F-27417	SUPERSEDES:	SHEET 1 OF 1

FIGURE 22. REVISED STANDARD MS27860

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12. SPONSORING MILITARY ACTIVITY

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13. ABSTRACT

During earlier research under Contract No. AF 04(611)-9578, it was shown that damage to the flanges of the 6061-T6 aluminum AFRPL connector could be prevented if the seal material was softer than the flange material by 20 points Brinell. Under Phase I of follow-on Contract AF 04(611)-11204, five method of accomplishing this were studied: (1) soft coatings, (2) skin annealing, (3) softer aluminum alloys, (4) anodizing the flange, and (5) special aging of 6061-T6. The overaged 6061 aluminum for the seal was selected because of economic and technical reasons. Repeated-assembly, thermal-gradient, and stress-reversal-bending tests were conducted with helium-leakage rates below the limit of 7×10^{-7} atm cc/sec. Tentative specifications and standards were revised.

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14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
Thermal Gradients						
Materials						
Manufacturing						
Metallic Seals						
Seal Design						
Nickel Plating						
Threaded Connectors						
Qualification Tests						
Thermal Shock						
Stress Reversal Bending						
Helium Leakage						
Repeated Assembly						
Military Specifications						
Military Standards						

END-8-67

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