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BORE SEAL TECHNOLOGY TOPICAL REPORT

RESEARCH AND DEVELOPMENT PROGRAM ON MAGNETIC, ELECTRICAL CONDUCTOR, ELECTRICAL INSULATION, AND BORE SEAL MATERIALS

by

P. E. Kueger et al

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Westinghouse Electric Corporation
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NAS3-4162

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PREFACE

The work reported here was sponsored by the Space Power Systems Division of NASA Lewis Research Center under Contract NAS3-4162. Mr. R. A. Lindberg of NASA has provided the Project Management for the program. His technical contributions are also gratefully acknowledged. Mr. T. A. Moss of NASA is also acknowledged for his support and interest and for his recognition of the importance of this work. The work was accomplished at the Westinghouse Aerospace Electrical Division, which was the prime contractor, and at the Westinghouse Research and Development Center, which was a subcontractor. The Eitel-McCullough Corporation also acted as a subcontractor conducting the ceramic-metal bore seal investigation.

In a project of this type, many skilled engineers and scientists are consulted. While the reporting of electric material technology is given in three Topical Reports entitled: Magnetic Materials; Electrical Conductor and Insulation Materials; and Bore Seal Materials; no attempt will be made to single out a person's specific contribution, since, in many cases, it was in several areas. Those who actively contributed during the total program are recognized below:

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SUMMARY

This Topical Report prepared under NASA Contract NAS 3-4162 contains thermophysical, compatibility, and mechanical property data on ceramic-to-metal seal technology of interest to the design of advanced space electric power systems. It represents a thorough search of the recent world's literature on this subject and a bibliographic record on this topic.

The application of ceramic-to-metal bore seals to actual designs is described and discussed. The thermophysical and mechanical properties at elevated temperatures of selected ceramic and metallic members are reported. The materials include high-purity alumina, high-purity beryllia, columbium alloy Cb-1Zr, tantalum T-111, and columbium D-43.

Two major methods of joining ceramic to metals were investigated: the metalizing braze method and the direct-bonding, active-metal braze process. The joints produced by the metalizing method were too brittle to insure an adequate seal. The active-metal braze was used on the representative bore seals. Of the active-metal systems studied for the ceramic-to-metal seal, the 56 percent zirconium, 28 percent vanadium, 16 percent titanium system proved to be the most satisfactory.

Alkali-metal exposure tests show that for best results, the silica content of the ceramic must be held below 50 parts per million. Static capsule tests were used for the quantitative evaluation at 1000°F and 1600°F for compatibility of the bore seal materials and brazed assemblies with very high-purity potassium, lithium, and sodium-potassium eutectic alloy. Emphasis was placed upon maintaining very low oxygen level of the alkali metals throughout the loading procedure.

The most promising system for lithium exposure at 1000°F or for potassium exposure at 1600°F consists of a ceramic body of Thermalox 998 beryllia (99.8 BeO), brazing alloy of 56 percent zirconium 28 percent vanadium 16 percent titanium, and a metal member of columbium-1% zirconium. All tested specimens were vacuum tight after exposure.

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SECTION I

INTRODUCTION

This report presents the ceramic-to-metal seal technology of interest to design advanced space electric power systems where alkali metals are used as the thermodynamic working fluid or as a heat transporting medium. Ceramic-to-metal seal technology is used in a bore seal which is needed in either a motor or inductor alternator. It isolates the more vulnerable portions of the design from the corrosive effects of the alkali-metal working fluid.

This report is one of three topical reports prepared under NASA Contract NAS3-4162 for the Lewis Research Center. The others, in the area of electric materials, include; Magnetic Materials (WAED 64.52E) and Electrical Conductor and Insulation Materials (WAED 64.53E).

One portion of the program was concerned with the determination of compatibility at elevated temperatures (1000 to 1600°F), of selected ceramic materials, such as alumina and beryllia, and ceramic-metal joining systems for bore seals with very high-purity NaK, K and Li. The metal member of the test assemblies to be used in this work was selected as Columbium alloy Cb-1Zr with limited consideration given to Tantalum T-111 and Columbium D-43. The most critical area in bore seal technology is the ceramic-to-metal seal. Both refractory metal metalizing with a braze and active metal brazing were used in joining the metal and ceramic members. Static capsule tests were used for the evaluation of alkali-metal compatibility. The determination and control of the purity of the liquid metals in the capsules were stressed, particularly with regard to the oxygen level.

A literature survey was conducted to identify existing information available on bore seal materials, joining methods, and alkali-metal compatibility. Appendix A lists the more applicable documents found. In general, thermophysical and mechanical properties of ceramics and metals were found to be quite adequately documented in the literature. One exception was in the creep and welding characteristics of refractory metals. These areas however, are being investigated under other sponsored programs so no effort was undertaken on the current program.

Very little useful information was found in the unclassified literature on joining systems using ceramics and metals which are to be used in the alkali-metal environments being considered on the current program. In many cases, the purity and fabrication history of test specimens were not reported and the purity of the alkali metal was not known. The major portion of this program was therefore directed toward these areas and with emphasis on higher test temperatures.

This report is arranged in three technical areas. Section II defines the applications of bore seals to actual designs and discusses ceramics, metal members, and joining techniques. Section III describes test methods and the material chemistry. Section IV presents a summary of the thermophysical and mechanical properties of interest in the bore-seal design.

SECTION II

TECHNICAL DISCUSSION

A. APPLICATION OF BORE SEALS TO ELECTRIC POWER SYSTEMS USING ALKALI METALS AS WORKING FLUIDS

1. General Requirements

Working fluids and coolants used in turbine-driven space power applications must operate at high temperatures to reduce waste heat radiator weight. The thermal and physical properties of alkali metals such as potassium, sodium and cesium make them suitable for use as high-temperature working fluids in the vapor state. Lithium and sodium-potassium eutectic are useful as heat transport fluids.

Electrical insulation materials which are presently available are subject to corrosive attack by alkali-metal liquids and vapors. Therefore, electric power system components that operate in an alkali-metal environment must include a means of protecting the insulation system from this environment.

In the case of rotating electrical equipment, the stator insulation is protected by a stator bore seal which forms a hermetically sealed stator cavity in combination with the stator housing. All-metal bore seals have been used in some previous motor designs which operate at 60 cps. The designs covered in the present program make use of a combination of ceramic and metal bore seal components. High frequency operation results in a considerable motor or generator weight reduction compared to low frequency machines, and the use of ceramics in place of metals results in a more efficient machine at high frequencies. Generator and motor rotors must be made of materials which resist alkali-metal-induced corrosion or be enclosed in a protective can.

This study has been limited to solid rotor inductor generators

having either a radial gap or an axial gap; the gap being the length between the rotor pole face and the stator stack. The radial gap design requires a cylindrical gap liner with radial clearance between the liner ID and the rotor OD. The axial gap design requires pancake type ceramic seals in the gaps with axial clearance between the rotor pole faces and the seal members. A motor would be constructed in a manner similar to a radial gap generator.

To perform properly, a stator bore seal assembly of either design must meet several requirements.

a) The materials used must be physically capable of operation at temperatures of 1000°F and higher.

b) The materials must be impervious to attack by alkali-metal liquids and vapors.

c) Joints between seal components and between the seal assembly and housing must be vacuum leak-tight.

d) The seal material used in the air gap must have high electrical resistivity to minimize eddy current losses.

e) Air gap (seal plus clearances) must be as small as possible so that an excessive number of field coil ampere-turns are not required to overcome the reluctance of the gap.

f) The various materials used in the seal assembly must have mutually compatible physical properties.

The need for a high electrical resistivity bore seal material in the gap imposes a severe restriction on material selection. Metals are unsatisfactory as gap materials in most applications because of electrical conductivity characteristics which can cause large eddy current losses. Metals can be used as structural members away from the winding area.

Alumina and beryllia are two ceramic materials that combine a high electrical resistivity with mechanical properties and high temperature capabilities suitable for the application. However, problems such as joining metals to ceramics to make leak-tight, corrosion-resistant joints, maintaining mechanical integrity in

spite of thermal stresses and maintaining clearances over a given temperature range, must be solved before a specific design will function properly.

2. Bore Seals - Types and Design Requirements

a. AXIAL GAP GENERATOR BORE SEAL DESIGN

Figure II-1 shows one possible configuration of an axial gap inductor generator with a bore seal. The numbered components are identified in Table II-1. Many design details of the generator are omitted, and the bore seal component thicknesses are exaggerated to emphasize this assembly.

The ceramic discs (1) fit in the air gap (17) between the stator laminations (11) and the rotor pole faces (13). The discs are positioned by metal cylinders (5 and 6) through the flanged metal washers (3 and 4). Metal-to-ceramic joints (7) are brazed and metal-to-metal joints (8) are welded. Ceramic washers (2) are provided to share the shear and tensile stresses caused by differential thermal expansion at the ceramic-to-metal joints (7). The bore seal assembly is located and supported by integral ring flanges on the generator magnetic frame (9). Convolutions on the inner metal cylinder (6) are provided to reduce the axial loads resulting from differential thermal expansion between the inner and outer metal cylinders.

In a specific design for a bore seal of this type, the effects of the thermal environment must be carefully considered. Clearances in the air gaps must be maintained over the entire temperature range to which the generator is subjected. In the radial direction, thermal stresses in the brazed joints must be kept low to prevent failure of a joint, and stresses in the ceramic discs must not cause dishing and cracking of the ceramic. Caution is required during welding of the bore seal into the generator to insure that the stator winding and insulation systems are not overheated or damaged.

While interest in this type of construction was fostered in early designs, recent efforts have been shifted to the radial gap configuration because stress problems using ceramic cylinders have been less severe than those encountered using ceramic discs.

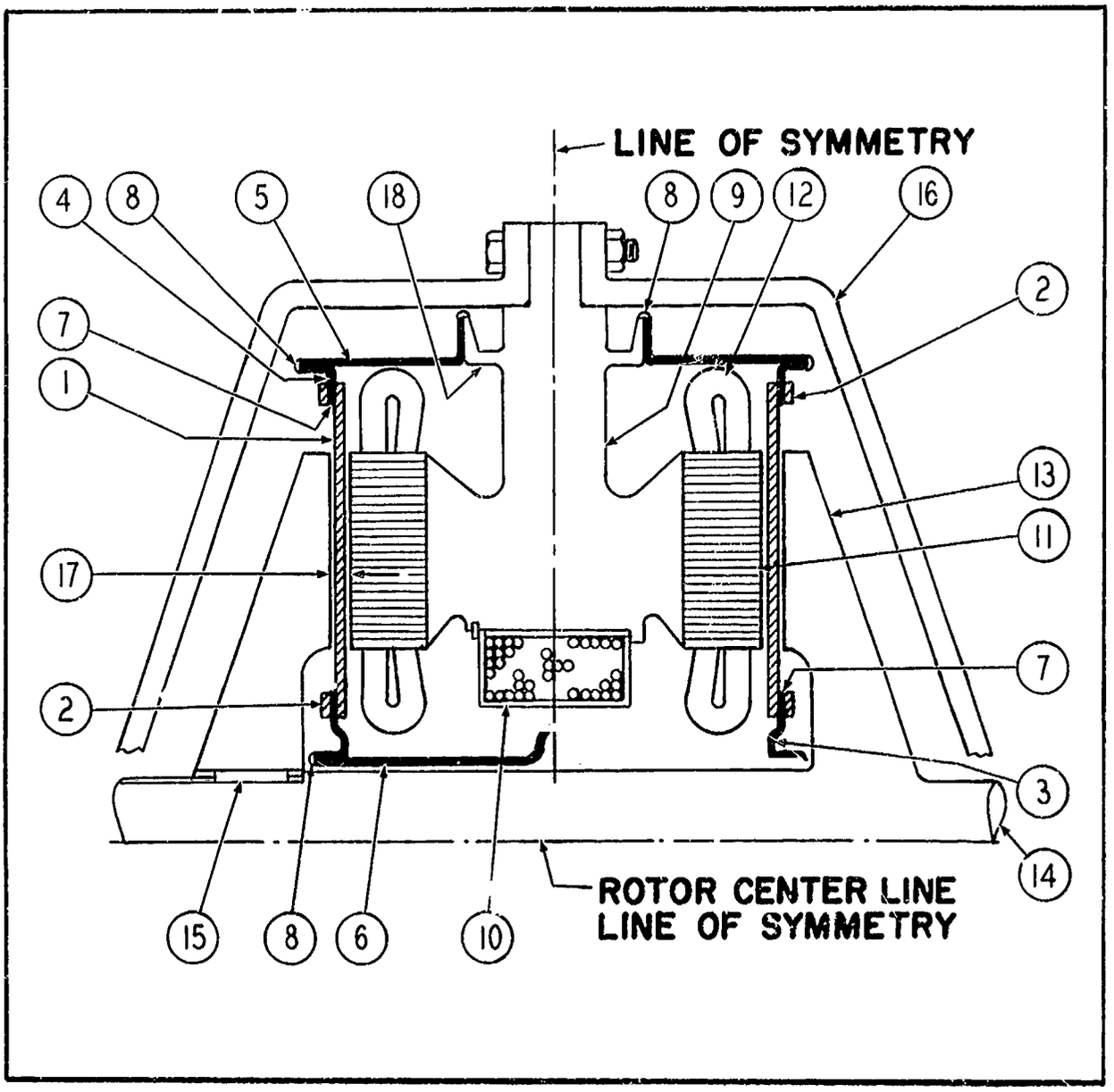


FIGURE II-1. Bore Seal Assembly, Axial Gap Inductor Generator

TABLE II-1. Axial Gap Bore Seal Assembly - List of Components

Item No.	Description
	Bore Seal Components
1	Disc - Ceramic (2)
2	Washer - Ceramic, Brazed Joint (4)
3	Washer - Inner, Metal, Flanged (2)
4	Washer - Outer, Metal, Flanged (2)
5	Cylinder - Outer, Metal, Flanged (2)
6	Cylinder - Inner, Metal, Convoluted
7	Joint - Brazed (4)
8	Joint - Welded (6)
	Generator Components
9	Frame - Magnetic
10	Coil - Field
11	Lamination - Tape, Toroidal (2)
12	Conductor - Stator
13	Pole - Rotor
14	Shaft - Rotor
15	Key - Shaft
16	Housing - Non-magnetic
17	Air Gap
18	Ring - Flange

b. RADIAL GAP GENERATOR BORE SEAL DESIGN

Figure II-2 shows a configuration for a radial gap inductor generator bore seal design. The numbered items are identified in Table II-2. Most of the design details in the generator have been omitted to emphasize the bore seal assembly, and the thickness of the bore seal components has been exaggerated. A ceramic cylinder (1) fits in the air gap (15) between the stator laminations and the rotor pole tips (11). A metal section could replace the ceramic in the area between the two laminated stacks (9), but doing so adds two additional brazed metal-to-ceramic joints which are not required with single piece construction. Metal end pieces (3 and 4) are brazed (5) to the ceramic cylinder and welded (6) to the generator frame brackets (12). Ceramic end rings (2) are added at the brazed joints to share the radial shear and tensile stresses caused by radial differential thermal expansion. The metal end piece (3) provides convolutions at one end of the bore seal to relieve axial stresses caused by axial differential thermal expansion. The metal end piece (4) at the opposite end of the seal may also function as a diaphragm to relieve axial stresses.

In a specific design for a radial gap bore seal of this type, the effects of thermal environment must be considered. As with the axial gap design, clearance in the air gap between the ceramic cylinder and the rotor pole faces must be maintained over the temperature range to which the generator is subjected. Thermal stresses in the radial direction must be kept low enough to prevent shear failures of the brazed joints during operational temperature excursions. The bore seal assembly must be welded into the stator after installation of the stator windings and insulation system. The joints must be accessible for welding and must be located to insure that welding and post-weld heat treatment do not damage the stator insulation system.

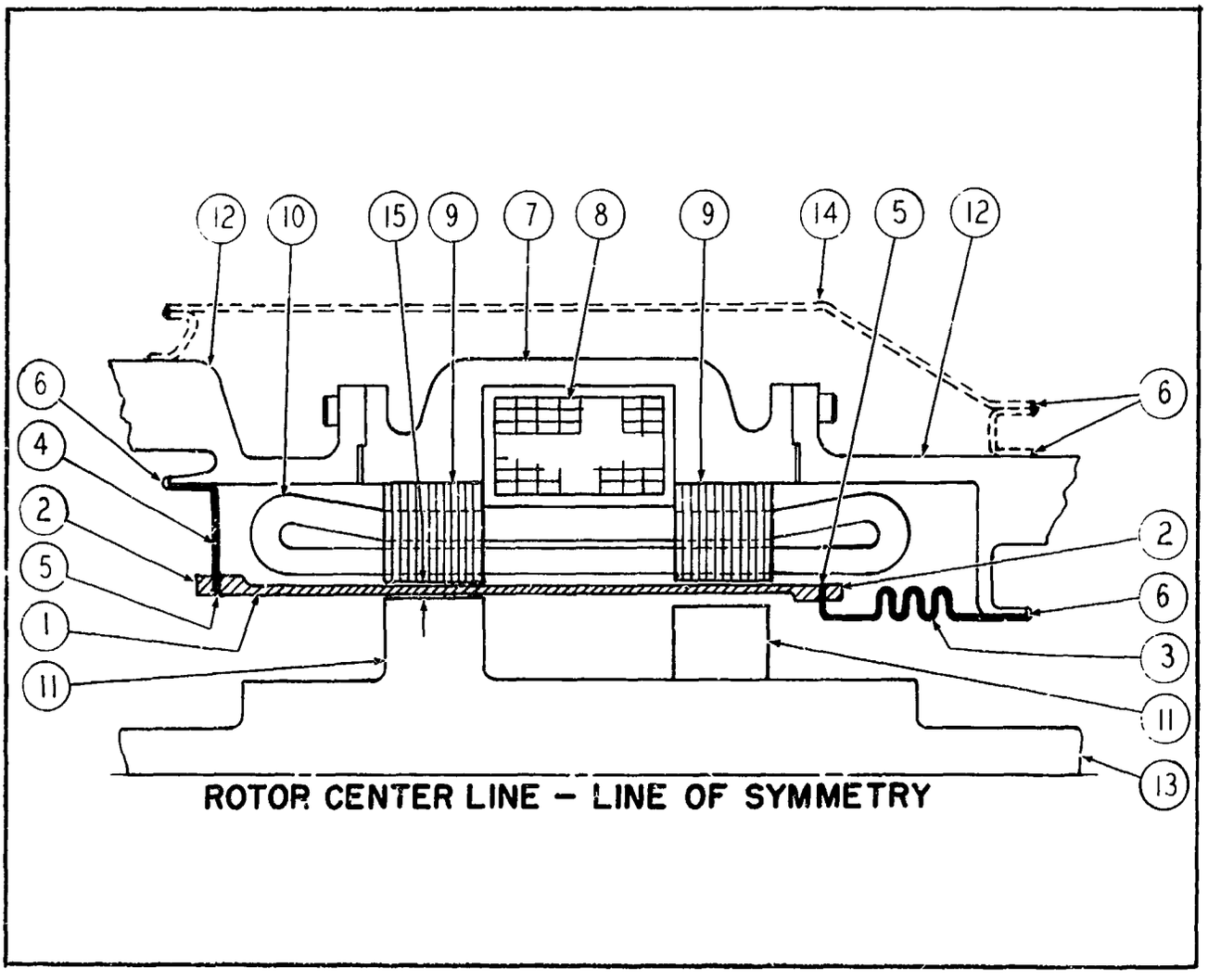


FIGURE II-2. Bore Seal Assembly, Radial Gap Inductor Generator

TABLE II-2. Radial Cap Bore Seal Assembly - List of Components

Item No.	Description
	Bore Seal Components
1	Cylinder - Ceramic
2	End Ring - Ceramic (2)
3	End Piece - Metal, Convoluted
4	End Piece - Metal, Diaphragm
5	Joint - Brazed (2)
6	Joint - Welded (6)
	Generator Components
7	Housing - Magnetic
8	Coil - Field
9	Laminations - Magnetic (2)
10	Conductor - Stator
11	Pole - Rotor
12	Bracket - Non-magnetic (2)
13	Shaft - Rotor
14	Shield - Inerting
15	Air Gap

c. DESIGN CONSIDERATIONS FOR BORE SEAL ASSEMBLIES

The temperature range over which a bore seal assembly must maintain physical integrity and chemical compatibility with alkali metals is the primary design consideration. Stresses created by differential thermal expansion increase as the effective temperature range is increased, and the corrosion resistance capabilities of the various materials decrease with increasing temperature level.

Figure II-3 is a plot of cumulative length change per unit length versus temperature for several bore seal materials based on an initial temperature of 77°F. All of these potential bore seal materials (except columbium-1% zirconium) show an expansion rate that increases with increasing temperature. The rate of change is greater for the ceramics than for the metals. Once installed in the stator, the bore seal will be exposed to temperatures ranging from room temperature or lower to 1200 - 1500°F, depending on the application. Temperatures of 2000°F and higher will be required to form the brazed and welded joints, resulting in large residual thermal stresses when the joint is cooled. Stress relieving operations should be carried out on both brazed and welded joints to reduce the initial assembled stresses as much as possible. The bore seal design must accommodate any residual stresses plus those caused by temperature changes from ambient when the generator is non-operative and cold and also when it is operating at temperature. Generator frame thermal expansion characteristics must also be considered since the frame forms part of the sealed cavity. Rotor thermal expansion characteristics are important as clearance between rotor pole tips and the bore seal air gap member must be maintained at all temperature levels.

Installation of the bore seal in the stator requires the forming of welded joints. Fusion type welded joints can be formed by electron beam welding in a vacuum or by tungsten inert gas welding in a controlled atmosphere. The electron beam process has a smaller heat-affected zone which results in less weld contamination and less severe aging problems. Post-weld heat treatment of the refractory metal member will be required to prevent embrittlement and/or reduce alkali metal attack in the weld zone. This may be done by proper posi-

tioning chill blocks and heating the weld zone with a defocused electron beam or other appropriate method.

The most common method of constructing ceramic-to-metal brazed joints for radial thermal stress resistance is to sandwich a thin gage metal washer between the main ceramic member and a ceramic ring, so that both sides of the metal member are brazed to ceramic material. This procedure results in a sharing of radial shear stresses in the brazing material on each side of the metal member and minimizes bending moments between the metal and ceramic. In most cases, axial stresses can be limited by a flexing diaphragm or by adding convolutions as required to cylindrical metal members.

Ceramic materials display good strength properties in compression but relatively poor properties in tension. This characteristic must be considered in designing the ceramic members of a bore seal assembly particularly in using flat discs such as are required in the axial gap design discussed previously. Annular convoluted washers have not been particularly successful in absorbing radial stress buildups, and dishing of ceramic discs can cause tensile failures that result in cracks through the thickness of the disc.

Figure II-3. Thermal Expansion, Bore Seal Materials

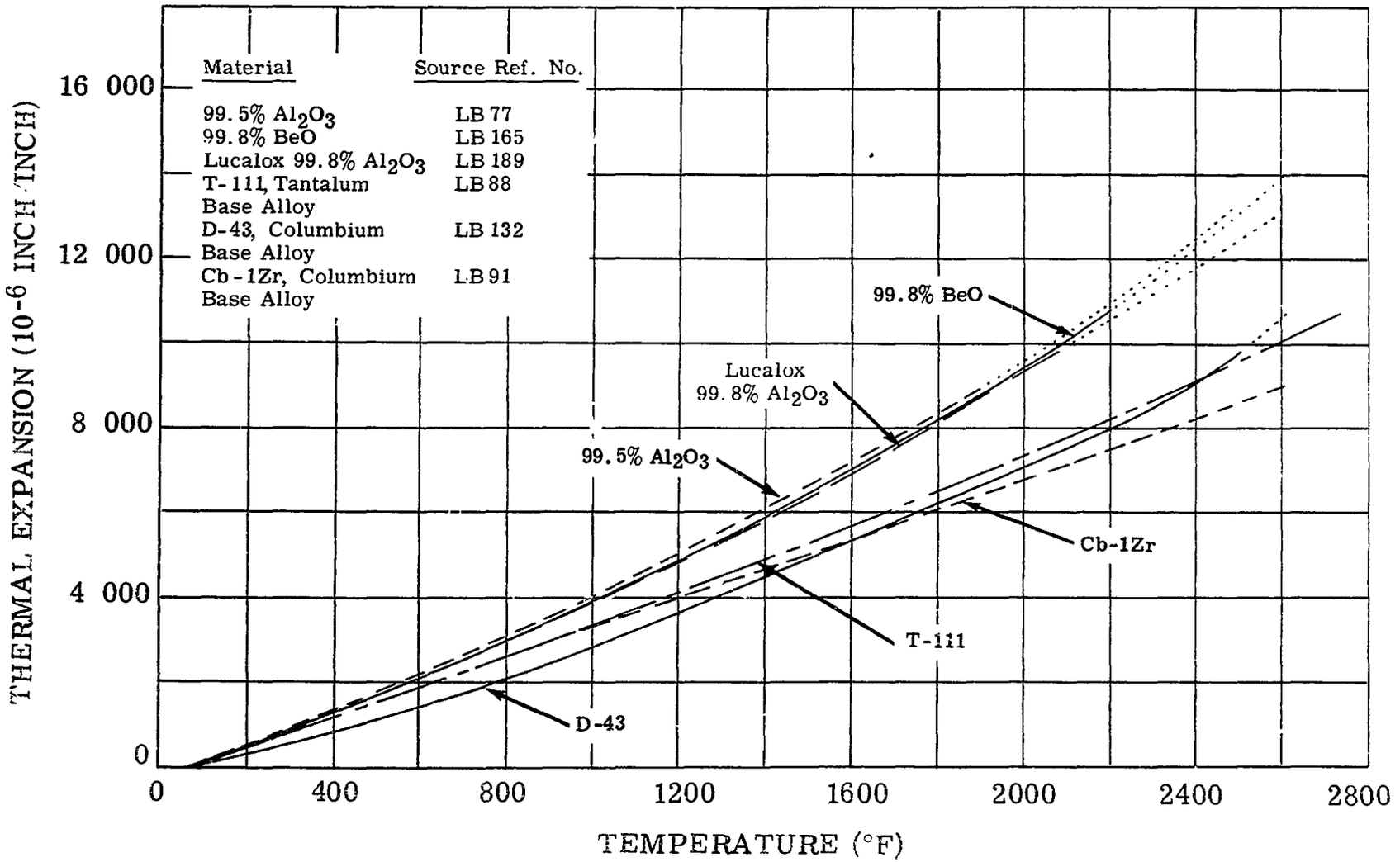


FIGURE II-3. Thermal Expansion of Typical Bore Seal Materials

d. BORE SEAL JOINT STRESS ANALYSIS

A bore seal for a radial gap inductor generator is shown in Figure II-2. By incorporating a bellows or diaphragm section, the axial stiffness of the assembly can be reduced considerably. Such arrangements can absorb most of the axial differential thermal expansion between the seal members and the generator frame with low axial loads. Thus, axial stresses in the ceramic members and the critical metal-ceramic joints are kept low.

For axial gap machines (Figure II-1), similar considerations are required. In addition, the design must insure that there is no axial interference (rubbing) between stationary and rotating components. Rubbing is a potential problem with both type machines, it is easier to maintain clearance in a radial gap type, since an axial gap seal tends to warp.

Primary efforts on this program have been associated with the bore seal for the radial-gap machine. The determination of stresses, particularly in the metal-ceramic joints, requires the construction of mathematical models of the seal components. Models have been constructed assuming no deflection in the quarter-toroid sections between the cylindrical and diaphragm sections. A similar assumption was made in the analysis of bellows sections. The deflection of the resulting diaphragm section is shown in Figure II-4.

A solution for this type of loading and deflection provide the relationship

$$W_{max} = KP\ a^2/Eh^3 \ (1)$$

where $K = f(a/b)$

E = Modulus of elasticity

P = Load

W_{max} = Total deflection of diaphragm

(1) S. Timoshenko, Strength of Materials, Part II, 3rd Ed. D. VanNostrand Co., Inc., 1956

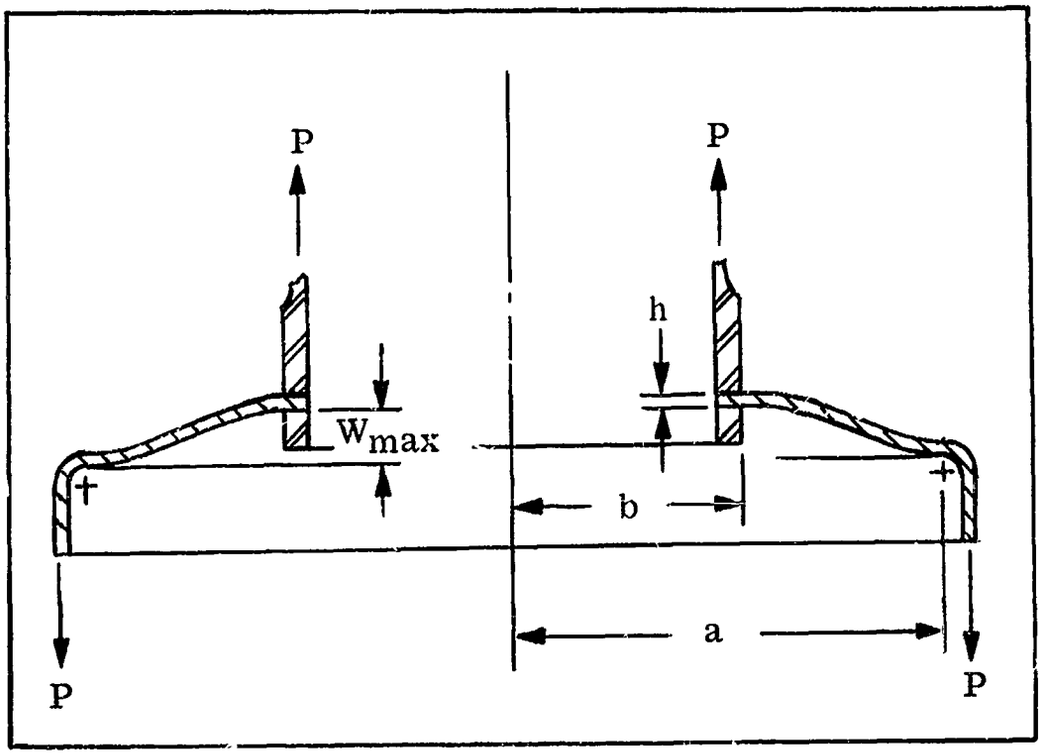


FIGURE II-4. Bore Seal Stress Model

The factor K increases as a function of the outside to inside radius ratio a/b. The inner radius b and the product Eh^3 W_{max} may be considered constant for a given design. Then the axial load P will be inversely proportional to the product Ka^2 and it can be significantly reduced by increasing the radial depth (a-b) of the diaphragm.

If the diaphragm depth is limited by the generator, a bellows, although less reliable, may be used. If this should be necessary, the bellows may be considered as a number of diaphragms (two per convolution) in series. The axial deflection per diaphragm will be $\Sigma W_{max}/2n$ (where n is the number of convolutions and ΣW_{max} is the total deflection absorbed by the bellows). The simplified analysis of diaphragms was applied to the welded bellows of Figure II-5. A similar bellows had been tested under compressive loads but assembled

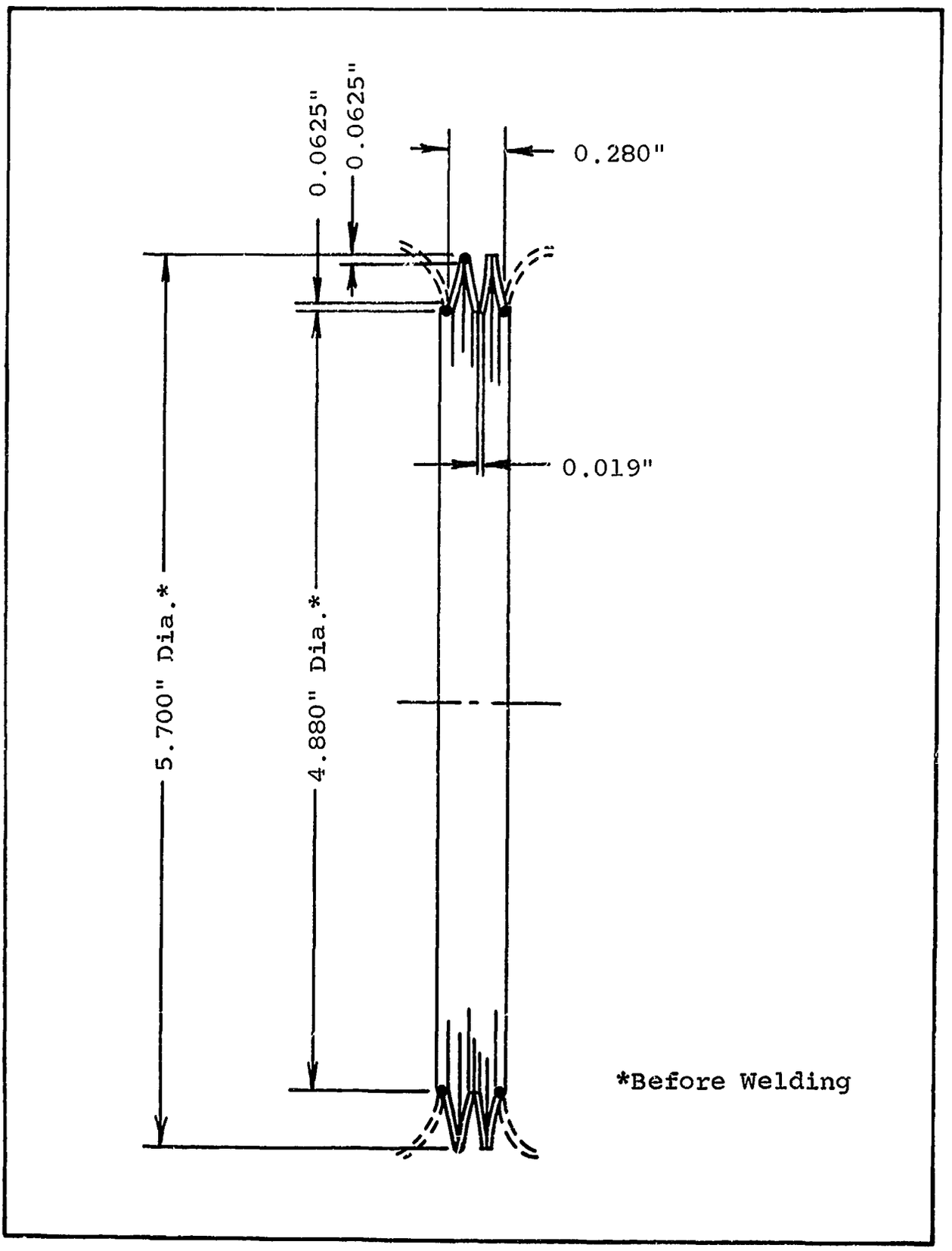


FIGURE II-5. Welded Bellows

(welded) dimensions are unavailable. Drawing dimensions of the parts, with an assumed weld depth of 1/16 inch, were used in the analysis. Results are indicated with the test values in the table below. Three of the four calculated loads are within 6 percent of the test loads. While these calculations do not establish the accuracy of this approach, they do tend to support the suitability of the analysis for preliminary design estimates.

Deflection (inch)	Test Load (pounds)	Calculated Load (pounds)	Deviation (percent)
0.0015	25	30.9	23.6
0.0016	35	33.0	- 5.7
0.0022	45	45.4	0.9
0.0025	50	51.5	3.0

The stress of the diaphragm is determined through the equation[2] $S = K_S P/h^2 = (EhK_S W_{max})/(Ka^2)$. The factor K_S also increases with the radius ratio a/b but the ratio K_S/K decreases with increasing radius ratio. Following the previous assumptions, the diaphragm stress, proportional to K_S/Ka^2, will decrease with increasing diaphragm depth. The approximate stresses of the bellows diaphragms can be similarly determined.

A detailed radial stress analysis for a similar type joint is contained in Aeronautical Systems Division Technical Documentary Report No. ASD - TDR - 63.677, dated July 1963.

(2) S. Timoshenko, Strength of Materials, Part II, 3rd Ed. D. VanNostrand Co., Inc., 1956

B. DISCUSSION OF MATERIAL PROPERTIES

1. General Discussion of Bore Seal Materials

The long term stability of ceramic-metal bore seals is a function of both physical and chemical environment produced by alkali metal and vapor at elevated temperatures. Other space power oriented programs have as their objective the development of metals and metal joining systems capable of performing under these conditions.

The work on this program has been concentrated in the area of ceramics and ceramic-metal joining systems which may be suitable for these environments. The metal member in most of these assemblies was columbium-1% zirconium alloy. This alloy has good hot strength and excellent resistance to potassium. A screening of columbium base D-43 (Cb-10W-1Zr-0.1C) and the tantalum base alloy T-111 (Ta-8W-2Hf) for back-up were included in the program. The latter two alloys exhibit higher strength (Figures IV-18 and IV-24) and excellent alkali metal corrosion resistance. The normal ceramic-metal sealing requirements of matching thermal expansion and low yield strength are not fully satisfied by these low-expansion refractory alloys with high elevated-temperature strength. The Cb-1Zr alloy which approaches alumina in thermal expansion will be assumed satisfactory unless positive contrary evidence develops in the design evaluation.

Preliminary information on another program[3] indicates that low-oxygen Cb-1Zr and D-43 alloys show excellent corrosion resistance in high-purity potassium after extended exposure at 2000° F.

High temperature creep[4] and welding and aging[5] properties under closely controlled test conditions are being determined on

(3) Evaluation of High Strength Columbium Alloys for Alkali Metal Containment. NASA Contract NAS 3-2140 by General Electric SPD.

(4) Generation of Long Time Creep Data on Refractory Alloys at Elevated Temperature, NASA Contract NAS 3-2545 by Thompson Ramo Wooldridge Corp.

(5) Determination of the Weldability and Elevated Temperature Stability of Refractory Metal Alloys Contract, NASA Contract NAS 3-2540 by Westinghouse Astronuclear Laboratory.

current programs.

Since the ductile-brittle transition temperature is increased in columbium-1% zirconium alloy as a result of welding, a post-weld anneal is required. A post-weld heat treatment of 2200°F for one hour (LB 161)[6] in a vacuum of 10^{-5} torr is adequate for Cb-1Zr with nominal oxygen content. Aging and thermal treatments for this alloy were found to be sensitive to oxygen contamination.

Welding of columbium D-43 alloy by electron beam or by TIG welding in a vacuum-purge chamber raises the ductile-brittle transition temperature considerably. Recommended post-weld anneals for columbium D-43 range from eight hours at 2200°F to two hours at 2400°F in a vacuum of 10^{-5} torr or less.

The tantalum T-111 alloy has not exhibited aging tendencies and therefore does not require a post-weld anneal.

Of the three candidate materials for the metal portion of the bore seal, columbium-1% zirconium, D-43 alloy and tantalum T-111 alloy, the T-111 has the highest creep strength and the lowest creep rate. For purposes of comparison, a summary of existing creep and rupture data obtained by various investigators on 0.012 inch thick sheet is presented on the Larson-Miller plots of Figure II-6. Sample thickness affected the creep rates of T-111 (LB 180) and D-43 alloys (LB 181). The rupture times for the D-43 alloy were insensitive to samply thickness (LB 132). The meager rupture data available for the columbium-1% zirconium alloy indicate the Cb-1Zr was probably tested in a relatively low-purity atmosphere. Those portions of the bore seal which require high-creep strength should be made from the T-111 type of alloy.

Ceramics suitable as the ceramic member of bore seal assemblies have not previously been systematically or quantitatively evaluated in high-purity-alkali-metal environments. The thermodynamic considerations of high alumina and beryllia ceramics had been reviewed by Eitel-McCullough on a related program[7].

(6) Numbers preceded by LB indicate bore seal reference tabulated in Appendix B.

(7) Westinghouse Sub-Contract on SPUR Project AF33(650) 10922.

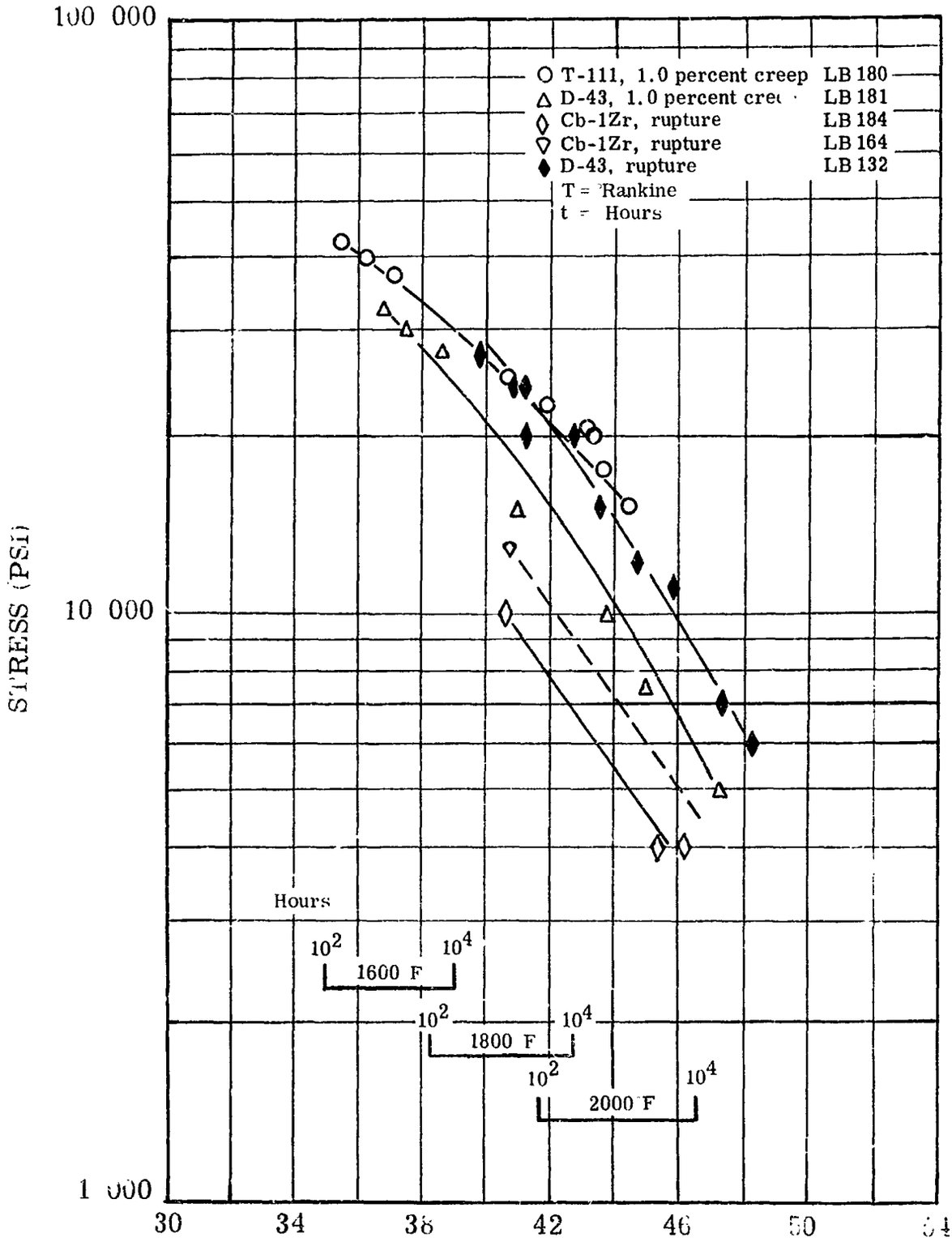


FIGURE II-6. Larson-Miller Plot of Vacuum Creep and Rupture Data for Several Columbium and Tantalum Base Alloys 0.012 Inch Thick Samples. (Reference: See Curve)

Figure II-6. Creep Curves for Refractory Alloys

The alkali metals or their vapors have two general modes of attack on ceramics.

a) The selected alkali metal is capable of reacting with any oxide that is thermodynamically less stable than the alkali-metal oxide.

b) The alkali metal may contain dissolved alkali oxide which can react with the ceramic to form compounds. This process normally proceeds much more rapidly at the grain boundaries. The decreasing order of thermodynamic stability of potentially usable refractory oxides, at room temperature, is given in Table II-3[8].

The free energies of formation of some additional rare earth oxides are presented in Table II-4. In Figure II-7 the free energies of formation of selected oxides are plotted versus temperature. While the kinetics of reaction rates are also important, the free energies represent the most readily available data for preliminary screening of materials.

From the data shown it is clear that several pure oxides will resist alkali metals. Thus, potassium will be resisted by CaO, ThO_2, BeO, MgO, La_2O_3, SrO, Y_2O_3, BaO, HfO_2, and Al_2O_3 in that order, or a modification of that order depending on the source data used. With lithium the list is reduced, but the first four oxides are still promising. The oxides of La, Sr and Y will be borderline while those of Hf and Al can be expected to be severely attacked. Most of the rare earths display favorable free energies of formation as shown in Table II-4. However, some of the promising ceramics have other deleterious characteristics (i.e. hygroscopicity, poor shock resistance) while others are available on a development basis only.

Silica in the form of quartz will resist potassium up to at least 600°F. In the form of glass and/or combined with other oxides the thermodynamic stability of silica is decreased (LB 83). A number of these materials have been tested in alkali metals by various investigators (LB 138, 145, 149). Although the experimental results generally substantiated the thermodynamic predictions,

(8) Westinghouse Sub-Contract on SPUR Project AF33(650) 10922.

TABLE II-3. Free Energy of Formation of Oxide Ceramics[a]

Compound	Melting Point		$-\Delta G°298°K$ kcal/g-atom
	°K	°F	
CaO	2873	4710	150.65
ThO_2	3573	5970	146.60
BeO	2823	4620	143.10
MgO	3073	5070	143.80
La_2O_3	2578	4180	142.85
SrO	2688	4380	141.10
Y_2O_3	2683	4370	140.00
BaO	2190	3485	133.50
HfO_2	3083	5090	133.03
Al_2O_3	2313	3700	133.20
ZrO_2	2950	4850	130.75
UO_2	3148	5207	129.60
CeO_2	2873	4710	122.50
TiO_2	2113	3345	112.75
SiO_2	2001	3140	104.95
V_2O_3	2250	3590	98.67
Ta_2O_5	2163	3430	97.76
MnO	2053	3235	92.05
NiO	2223	3540	57.30

(a) Coughlin, U. S. Bureau of Mines Memorandum, p 542, 1954

TABLE II-4. Free Energy of Formation of Some Rare Earth Oxides

Oxide	Melting Point		Free Energy (- Δ F kcal/mol)[a]	
	°K	°F	298°K	1000°K
Y_2O_3	2683[b]	4370	-298	-346.5
La_2O_3	2578[b]	4180	-406	-357.8
Ce_2O_3	1960	3070[c]	-411.5	-356.5
CeO_2	2873[b]	4710	-230	-194.5
Pr_2O_3			-420	-376
Pr_6O_{11}			-1302	-1333
PrO			-217.5	-187
Nd_2O_3	2173	3450[c]	-408	-354.7
Sm_2O_3	2573	4170[c]	-410	-366

(a) Compilation of the Properties of the Rare Earth Metals and Compounds by J. A. Gibson et al, May 1, 1959, Battelle Memorial Institute, Columbus, Ohio.

(b) Coughlin, U. S. Bureau of Mines Memorandum, p 542, 1954

(c) Reference LB 179

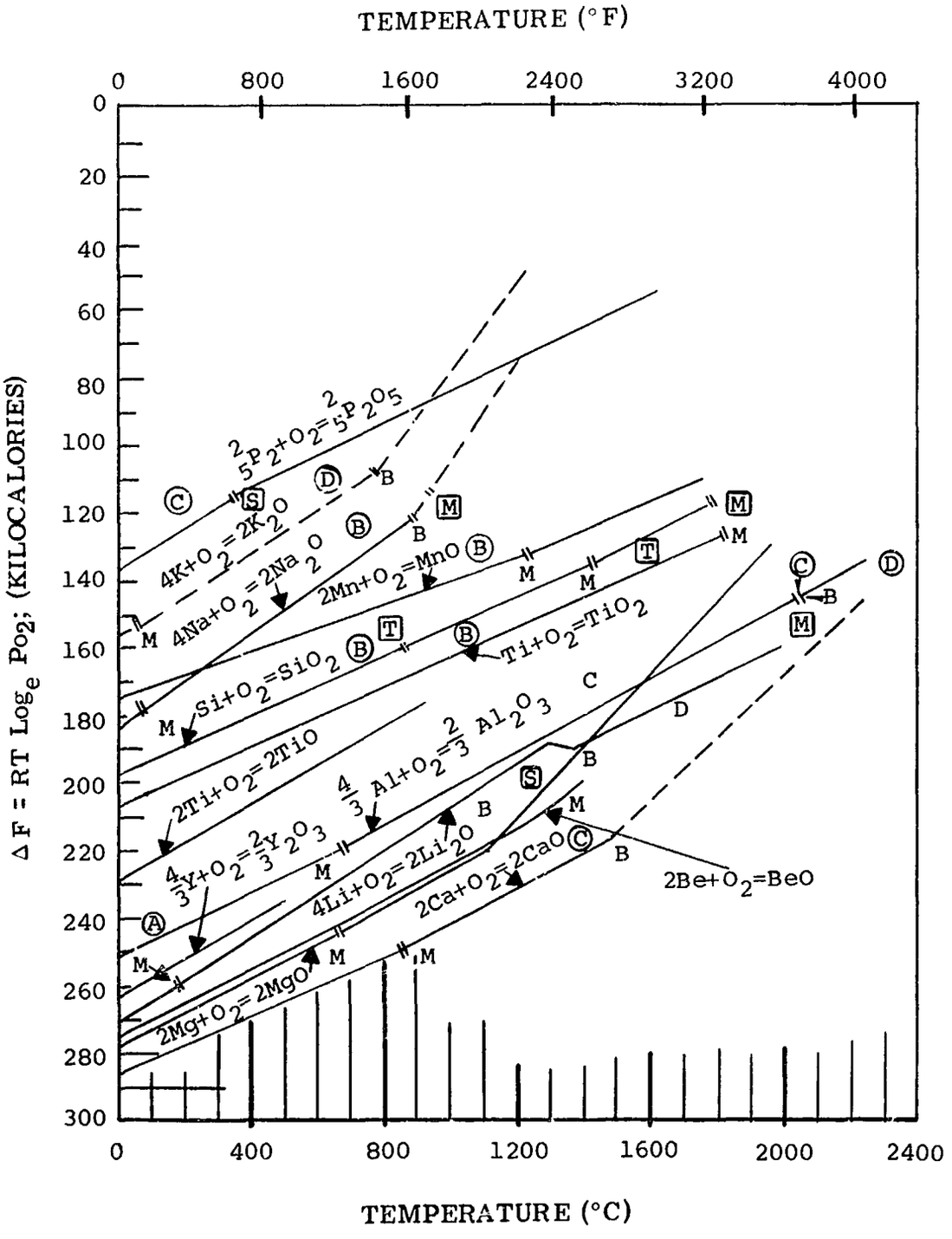


FIGURE II-7. Standard Free Energy of Formation of Oxides. Suggested accuracies (A) ± 1 Kilocalorie, (B) ± 3 Kilocalories, (C) ± 10 Kilocalories, (D) ±>10 Kilocalories (after R.F.D. Richardson, J.H.E. Jeffes, Journal of Iron and Steel Institute, London 1948 and O. Kubaschewski and E.V. Evans, Metallurgical Thermochemistry, John Wiley and Sons, 1965).

Changes of State	Element	Oxide
Melting Point	M	[M]
Boiling Point	B	[B]
Sublimation Point	S	[S]
Transition Point	T	[T]

data was often qualitative and varied according to test conditions and source. Important variables which are not fully documented are; the effects of material composition and purity, fabrication history, and alkali-metal purity.

With the polycrystalline high alumina (>94% Al_2O_3) and beryllia (>95% BeO) bodies, attack by potassium occurs at the grain boundaries where fluxing additions to the ceramic and impurities are concentrated. The most common fluxes contain silica, calcia and magnesia, of which silica is the most susceptible to reactions with the alkali metal oxides.

This is confirmed with the empirical evidence on this program. Alumina (Lucalox, G. E.) and beryllia (Thermalox 998, Brush Beryllium Co.) ceramics fluxed largely with magnesia and some calcia can be fabricated with silica levels of less than a hundred parts per million. This low silica level is a requisite for long term stability at elevated temperatures (1600°F to 1800°F) in alkali-metal environments. Shorter duration or lower temperature applications may tolerate several hundred parts per million silica. In each of these cases, silica is assumed to be the most significant corrosion sensitive component in the flux. These low flux bodies are recent developments and fabricability is limited.

Sapphire and Lucalox are not available in the large cylindrical bore seal geometries and will not be available for a number of years. The largest cylinder presently manufactured by the Lucalox process is four inches in diameter while sapphire is not available in sizes greater than two inches in diameter. Thus, the high-purity 99.8 percent and greater beryllia ceramic appears to be the most stable of the available commercial ceramics suitable for bore seal fabrication. The rare-earth oxides potentially offer better stability than beryllia and could probably be fabricated into bore seal geometries, if required, but much work remains in determining the fabricability and properties of bodies made from these oxides.

By consideration of the following discussion and the Material Properties Summaries contained in Section IV of this report, the designer will find that purity level and product history of ceramics are significant factors in performance of the parts. The curves of thermal conductivity versus temperature at three purity levels of beryllia are presented in Figure IV-2 showing the desirability

for high purity when high-heat transfer is required. The necessity for high purity beryllia and alumina for alkali metal containment was emphasized previously.

Nominal values of the mechanical properties of commercially available high-temperature bore seal materials are presented in Section IV. Generally, they have excellent temperature tolerance. These data reflect standard values. Strength of polycrystalline oxides are affected by grain size, porosity, surface conditions, environment and loading conditions. It is, therefore, as important to know the thermal history of a ceramic which is selected for critical applications as to know the purity.

The modulus of elasticity presented in Figure IV-12 reflects the findings of a number of investigators including Wachtman (LB65), Swartz (LB85), Coble (LB174). It was noted that there is a gradual decrease in elastic modulus in polycrystalline alumina to about 1600°F. At higher temperatures a non-linear drop was observed. This drop has been attributed to grain boundary slip. Therefore, factors which promote slip will accentuate this drop in strength. For instance, presence or formation of a glassy phase will induce slip. Beryllia follows a similar trend (Figure IV-6).

The effect of grain size on the mechanical properties of beryllia and alumina were reported in LB 165 and LB 187. The elastic modulus of polycrystalline alumina is essentially independent of grain size to 2500°F. However, as shown in Figure IV-13, the flexural strength of fine-grain alumina (1 to 15 microns) is substantially greater than for alumina of larger grain size. This holds true over the entire temperature range being considered (LB 188).

Although thermal expansion is relatively unaffected by varying porosity, the porosity (or relative density) of polycrystalline oxides must be stipulated and maintained in order that consistent mechanical properties of the insulation be realized. Coble and Kingery (LB 174) report a sharp decrease in the modulus and strength of polycrystalline alumina with increased porosity.

Electrical properties of alumina and beryllia oxides are more sensitive to impurities than are the mechanical properties. In general, the dielectric constant of aluminum oxide rises exponentially with temperature at low frequencies (e.g., 1000 cps).

At higher frequencies, it rises gradually at a shallow slope as temperature increases. Of the cationic impurities, magnesium causes the greatest rise in dielectric constant followed by silicon, titanium, calcium, chromium, and iron. Silica has by far the greatest detrimental effect on dielectric losses (LB 114).

The conventional method of joining a ceramic member to a metal is accomplished by applying a metallic coating of molybdenum to the ceramic; then brazing the resultant metalized ceramic to the metal member by standard techniques. When very high-purity (low flux) ceramics are used, a non-metallic glassy phase is incorporated into the metalizing paint. When fired, the glassy phase wets the ceramic and the molybdenum thus promoting the ceramic to metal bond.

For alkali metal systems, the constituents of the non-metallic glassy phase and the refractory metal primary phase must be compatible with the alkali metal vapor. Conventional metalizing compositions containing MnO and/or TiO_2 do not meet this requirement.

Another ceramic-metal sealing system in limited commercial use is the titanium-nickel active alloy braze process. Such active alloys react chemically and bond to the ceramic body, thus eliminating the necessity of metalizing. In joining a titanium-bearing active alloy seal to alumina, for example, the titanium may chemically reduce a finite amount of alumina by the reaction (LB 103)

$$2\,Ti + Al_2O_3 \longrightarrow 3\,Al + Ti_2O_3,$$

providing an interface containing Ti_2O_3. This compound and aluminum are less stable in potassium than the Al_2O_3 (Figure II-7) or metallic titanium.

The chemical compatibility of the active metal alloy and subsequent interface reaction products must, therefore, be considered. In this respect, active brazing alloys utilizing zirconium, hafnium, or yttrium as the more active component are thermodynamically more promising than titanium dominated alloys. In addition, ductility, thermal expansion, melting point and reactivity with the metal member must be considered.

The experimental portion of the ceramic-to-metal seal work of the current program was established with these criteria in mind.

One portion of the seal development work on this program involved the evaluation of thermodynamically stable rare-earth oxide secondary phases in a refractory metal metalizing base. Alkali-metal-compatible single-phase metalize coatings were also investigated. Although some high-strength seals were made with alumina using these metalizings, the seals were unsatisfactory because of their brittleness, low shear strength, or because the metalizings were rapidly eroded by the brazing alloys. Seals made with 99.8 percent beryllia by the above methods exhibited low strength.

Electroformed seals using nickel plating, although vacuum tight, proved to be too low in strength for load-bearing applications.

The seals which were subjected to alkali metal exposure on this program were all fabricated by the active-metal process. The active alloys which were selected for ceramic-to-metal joining evaluation (Table II-5) were being evaluated elsewhere for brazing refractory alloys and for alkali-metal compatibility (LB 20, 24, 156, 160). Assemblies made from several alloys exhibited satisfactory mechanical properties after exposure to alkali metals for 500 hours at 1000°F and 1600°F.

2. Discussion of Results

a. CERAMIC GAP LINER MATERIALS

The compatibility of the ceramic material to the selected environment was considered in a previous section on the basis of thermophysical data. The free energy of formation of the metallic oxides present in the ceramics, including any fluxing materials or impurities, was used to anticipate the relative effect of alkali metal exposure on the various ceramics.

Alumina ceramics were obtained in the form of dense modulus-of-rupture bars approximately 0.1 inch x 0.1 inch x 1.0 inch with various compositions between 94 and 100 percent Al_2O_3 to determine strength degradation from potassium exposure. Silica content, representing the least thermodynamically stable of the more common fluxing agents, was the primary variable. The nominal compositions and associated emission spectrographic analyses of the selected bodies are shown in Tables II-6 and II-7. A high-purity, low-silica, beryllia body was obtained from Brush Beryllium Company for evaluation.

TABLE II-5. Experimental Alloys for Brazing Columbium and Columbium Alloys

Number	Reference Number	Nominal Alloy Composition (weight percent)	Flow Point (°F)
1	LB 20	67Zr-29V-4Fe	2370
2	LB 20	60Zr-25V-15Cb	2335
3	LB 20	48Zr-48Ti-4Be	1920
4	LB 20	63Ti-27Fe-10Mo	2280
5	LB 20	63Ti-27Fe-10V	2340
6	LB 20	68Ti-28V-4Be	2280
7	LB 20	45Ti-40Zr-15Fe	1920
8	LB 20	75Zr-19Cb-6Be	1920
9	LB 20	46Ti-46Zr-4V-4Be	1830
10	LB 20	95Zr-5Be	1830
11	LB 20	62Ti-26Fe-8Mo-4Zr	2280
12	LB 20	80Zr-17Fe-3Be	1830
13	LB 24	56Zr-28V-16Ti (AS-537)	2280

TABLE II-6. Nominal Composition of Selected Ceramics

Ceramic Body	Major Constituent (weight percent)	Flux Phase or Impurites (weight percent)			
		MgO	CaO	SiO_2	Other
Linde Sapphire	100 Al_2O_3	--	--	--	--
Lucalox (G.E.)	99.75 Al_2O_3	0.25	--	--	--
Ei3-3W (Wesgo)	99.7 Al_2O_3	0.1	0.1	0.1	0.01
AD 99 (Coors)	99 Al_2O_3	0.25	0.25	0.5	0.1
AD 94 (Coors)	94 Al_2O_3	1.0	1.0	4.0	0.1
Thermalox 998 (Brush Beryllium)	99.8 BeO	0.130[a]	0.0085[a]	0.008[a]	Al 0.015[a] Fe 0.006[a]
Dysprosium Oxide (Eimac)	100 Dy_2O_3	--	--	--	--

(a) Lot analyses on supplied material. All other elements present at less than 0.003 percent (30 ppm).

TABLE II-7. Spectrographic Analysis[a] of Ceramics for Alkali Metal Vapor Exposure

Ceramic Body	Nominal Composition (percent)	MgO (weight percent)	CaO (weight percent)	SiO_2 (weight percent)	SiO_2[b] (weight percent)	Other (weight percent)
Sapphire (Linde)	100 $A^1{}_2O_3$	0.001	0.001	0.06	0.025	(e)
Lucalox (G.E.)	99.75 Al_2O_3	0.1	0.002	0.1	0.025	(e)
Ei3-3W (Wesgo)	99.7 Al_2O_3	0.15	0.15	0.3	0.250	0.04 Fe_2O_3
AD 99D (Coors)	99 Al_2O_3	0.25	0.2	1.25	1.5	0.1 Fe_2O_3 0.01 TiO_2
AD 94O (Coors)	94 Al_2O_3	0.8	1.0	5.0	4.5	0.15 Fe_2O_3 0.03 TiO_2
Thermalox 998 (Brush)[c]	99.8 BeO	0.13	0.008	0.008	0.008	0.015 Al_2O_3 0.006 Fe_2O_3
Dy_2O_3 (Eimac)[d]	100 Dy_2O_3	0.8	0.1	(e)	0.2	0.1 Al_2O_3

(a) Data except (b), (c), and (d) obtained by American Spectrographic Laboratories
(b) Special analyses with emphasis on relative silica. Vendor's silica analysis for Ei3-3W was 0.08 percent.
(c) All BeO data from lot analysis by Brush Beryllium Co.
(d) Values under 1 percent are ± 200 percent.
(e) Not determined

For long term compatibility in lithium, a more stable ceramic such as beryllia is required.

Dysprosium oxide was obtained from the Rare Earth-Uranium Mining and Development Corporation in their highest obtainable purity. The material was ball milled in methanol for 30 minutes in an alumina ball mill, pressed into 1/2 inch diameter discs 1/8 inch thick under pressure of 44 tons per square inch, and sintered in hydrogen with a 100°F dewpoint for 30 minutes at 3270°F. The sintered pellets showed a porosity of 50 percent.

Emission spectrograph data showed unacceptably high impurity concentration (MgO and SiO_2) at this point making potassium exposure unwarranted.

All bodies, except the dysprosia had suitable density. All bodies except dysprosia were free of surface porosity as determined by a Rhodamine B dye test. The beryllia bodies retain a faint uniform color which is within acceptability limits.

One of the prime requisites of this program was the determination of ceramic and ceramic seal compatibilities with very pure alkali metals under test conditions that minimize contamination, especially by oxygen or oxygen donors.

It had been reported that the presence of oxygen in alkali metals, or its availability in the test system, would accelerate corrosion of metals[9]. Therefore, on this program, ceramic test specimens containing various oxide modifiers were tested in individual test capsules to eliminate cross contamination. Other capsules containing a mixture of several ceramic test bars with silica content as high as four percent were loaded under identical conditions.

(9) J. R. DiStefano and A. P. Litman, Effects of Impurities in Some Refractory Metal-Alkali Metal Systems; Corrosion, v. 20, November 1964

The selected ceramics were exposed to potassium vapor in static capsule tests at 1600°F ± 30°F(10) for 500 hours in vacuum of 10^{-6} torr. Control bars from the same lots were exposed to the temperature only in vacuum. The starting oxygen impurity level of the potassium in the loaded capsules ranged from less than 10 ppm to 26 ppm as determined by analyzing the contents of similar (purity test) capsules loaded at the same time. Oxygen analyses were made by the mercury amalgamation and titration method. All ceramics were subjected to a one-half hour 1700°F clean firing in a $75N_2$-$25H_2$ atmosphere with a 86°F dewpoint before any special processing or testing.

After the capsule exposure tests, the potassium exposed samples were neutralized in methanol and water. The samples were then dried in a vacuum oven for approximately one hour at 230°F. The control pieces were dried at the same time. The exposed and control samples were then flexural-strength tested at the same time using an Instron universal testing machine with a four point loading fixture (Figure III-8). The inner and outer load points were 0.25 and 0.80 inches apart respectively. The loading rate was 0.1 inch per minute.

Flexural strengths of the cleaned fired ceramics and of the ceramics exposed to vacuum and to potassium vapor at 1600°F are shown in Table II-8. Ceramic exposure data at 1000°F in potassium, sodium-potassium eutectic and lithium are given in Table II-9. Photographs (macro and micro) of selected alkali-metal-exposed specimens are shown in Figure II-8 through II-12.

Observation of exposure tests show that the lower alumina bodies AD 94 and AD 99 were severely attacked by the 1600°F potassium vapor. The round AD94 rods were partially eroded while the AD 99 bars swelled approximately five percent. The modulus-of-rupture strength of both bodies was less than 3,000 psi after exposure. The broken modulus bars showed extensive discoloration throughout (see Figure II-8).

(10) Vapor pressure of potassium at 1600°F (870°C) in the capsules was approximately 38 psi.

TABLE II-8. Effect of 500 Hours Exposure to Potassium Vapor at 1600°F on Room Temperature Flexural Strength of Selected Ceramics.

Ceramic Body[a]	Key	Room Temperature Flexural Strength[e] Unexposed Control Specimens (psi)	Room Temperature Flexural Strength[e] Control Specimens After 1600°F, 500 hrs, Vacuum only[b] (psi)	Room Temperature Flexural Strength[e] Exposed Specimens After 1600°F, 500 hrs in Potassium[d] Vapor (psi)	
				Mixed Capsule[c]	Not Mixed
Thermalox 998 Beryllia 99.8% BeO	$\bar{x}$	20 860	19 890	17 620	18 442
	s	1 850	1 140	1 210	1 493
	n	5	5	4	5
Sapphire Alumina 100% Al_2O_3	$\bar{x}$	63 070	78 450	59 670	76 120
	s	21 100	4 310	11 250	11 200
	n	5	2	4	5
Lucalox Alumina 99.8% Al_2O_3	$\bar{x}$	34 030	33 050	29 813	29 360
	s	4 960	3 530	1 243	1 750
	n	5	2	4	5
Ei3-3W Alumina 99.7% Al_2O_3	$\bar{x}$	44 760	41 550	9 140	10 237
	s	3 680	1 907	353	227
	n	5	2	4	4
AD99D Alumina 99% Al_2O_3	$\bar{x}$	36 420	32 975	1 082	
	s	1 910	1 235	312	
	n	5	2	4	
AD 94O Alumina 94% Al_2O_3	$\bar{x}$	56 600		1 080	2 360
	s	5 990			1 006
	n	5		1	4

All test bars were fired to 1700°F for 30 minutes in $25H_2$-$75N_2$ 100°F dewpoint prior to brazing or exposure testing. Vacuum or alkali metal exposure specimens were outgassed in their respective capsules in a vacuum of 10^{-5} torr at 1380°F and cooled in vacuum. The chamber then was back-filled with high-purity helium.

Key $\bar{x}$ - arithmetic mean
s - standard deviation
n - number of specimens tested

(a) See Table II-7 for composition of ceramic bodies.
(b) The standard deviation value for the samples subjected to vacuum-only exposure at 1600°F is for reference and has little significance statistically with a sample size of 2 pieces.
(c) Two "mixed" capsules containing two modulus-of-rupture bars of each ceramic were tested in addition to the capsules containing five modulus-of-rupture bars of one ceramic type only.
(d) Oxygen levels of 21 and 26 ppm were measured in the potassium in associated purity test capsules. Analyses were made by the mercury amalgamation and titration method.
(e) Strength determinations were made by four point loading on 0.1 inch x 0.1 inch x 1 inch modulus-of-rupture specimens. Load was applied at the rate of 0.1 inch per minute.

TABLE II-9. Effect of 500 Hour, 1000°F Exposure to Potassium, NaK and Lithium Vapors on Room Temperature Flexural Strength of High Alumina and Beryllia Ceramics

Ceramic Body[a]	Key	Room Temperature Flexural Strength[e] Unexposed Controls (psi)	Alkali Metal	Room Temperature Flexural Strength[e] Exposed Samples 1000°F, 500 Hrs (psi)
Ei3-3W Alumina 99.7% Al_2O_3	$\bar{x}$	44 760	K[b]	41 980
	s	3 680		3 320
	n	5		5
	$\bar{x}$		NaK[c]	19 902
	s			2 330
	n			5
Thermalox 998 Beryllia 99.8% BeO	$\bar{x}$	20 860	K[b]	19 046
	s	1 850		1 710
	n	5		5
	$\bar{x}$		Li[d]	18 108
	s			1 405
	n			6

Key $\bar{x}$ - arithmetic mean
s - standard deviation
n - number of specimens tested

(a) See Table II-7 for composition of ceramic bodies; ceramics treated in same manner as described in note in Table II-8.
(b) Loaded capsule oxygen level of less than 10 ppm was measured on associated purity test capsule. Analysis by mercury amalgamation and titration method.
(c) Loaded capsule oxygen levels of less than 10 ppm were measured on two associated purity test capsules. Analysis by mercury amalgamation and titration method.
(d) The purity test capsule leaked after loading; no meaningful inference of oxygen or nitrogen content of lithium in the test capsule could be made.
(e) Strength determinations were made by four point loading on 0.1 inch x 0.1 inch x 1.0 inch modulus-of-rupture specimens. Load was applied at the rate of 0.1 inch per minute.

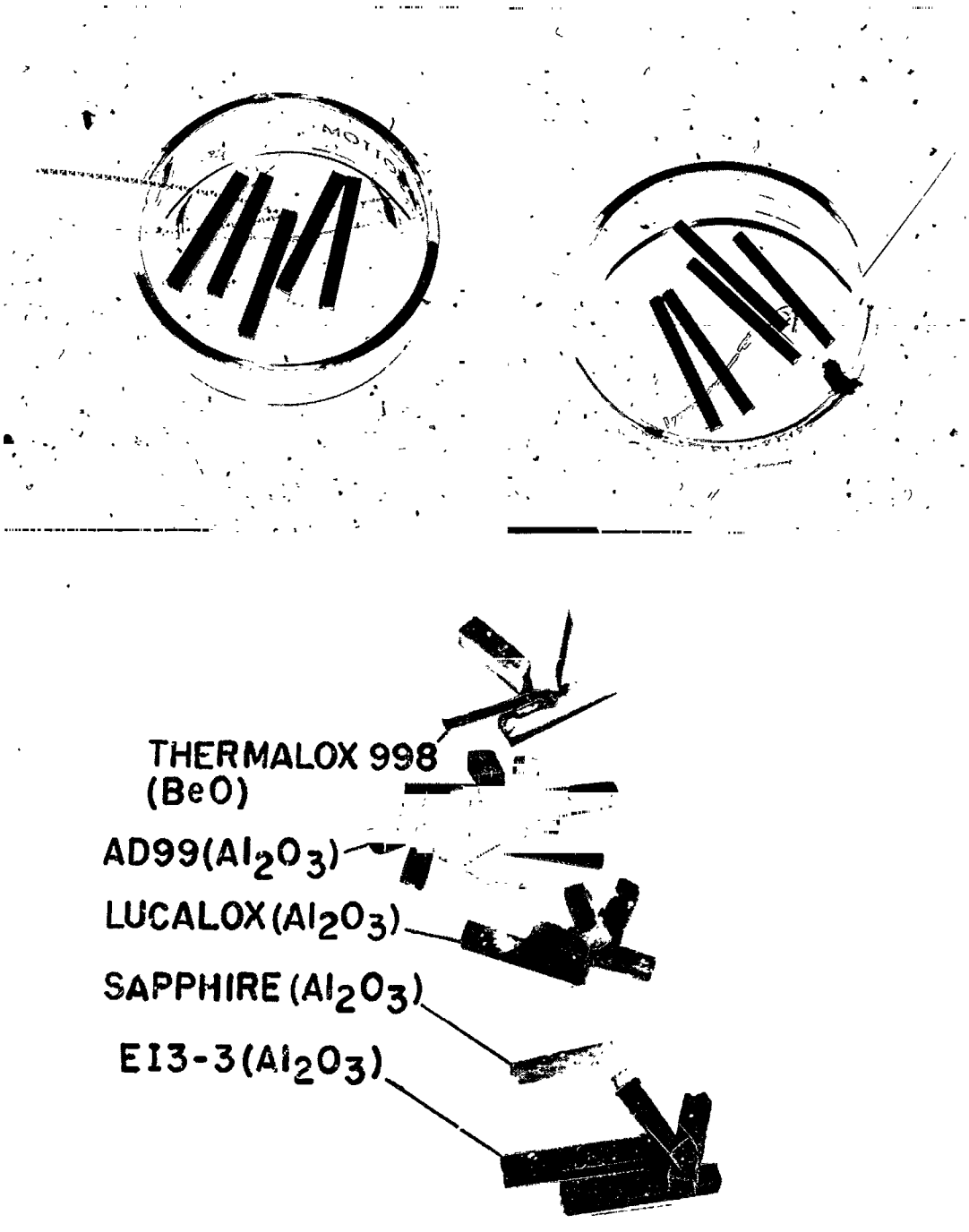


Figure II-8. Ceramic Modulus-of-Rupture Bars (one inch long) Before and After Potassium Exposure at 1600°F.

Macrophotographs of (top) typical round and square M-of-R bars before K exposure and (bottom) typical identified M-of-R bars after 500 hours in K vapor at 1600°F; broken during M-of-R testing.

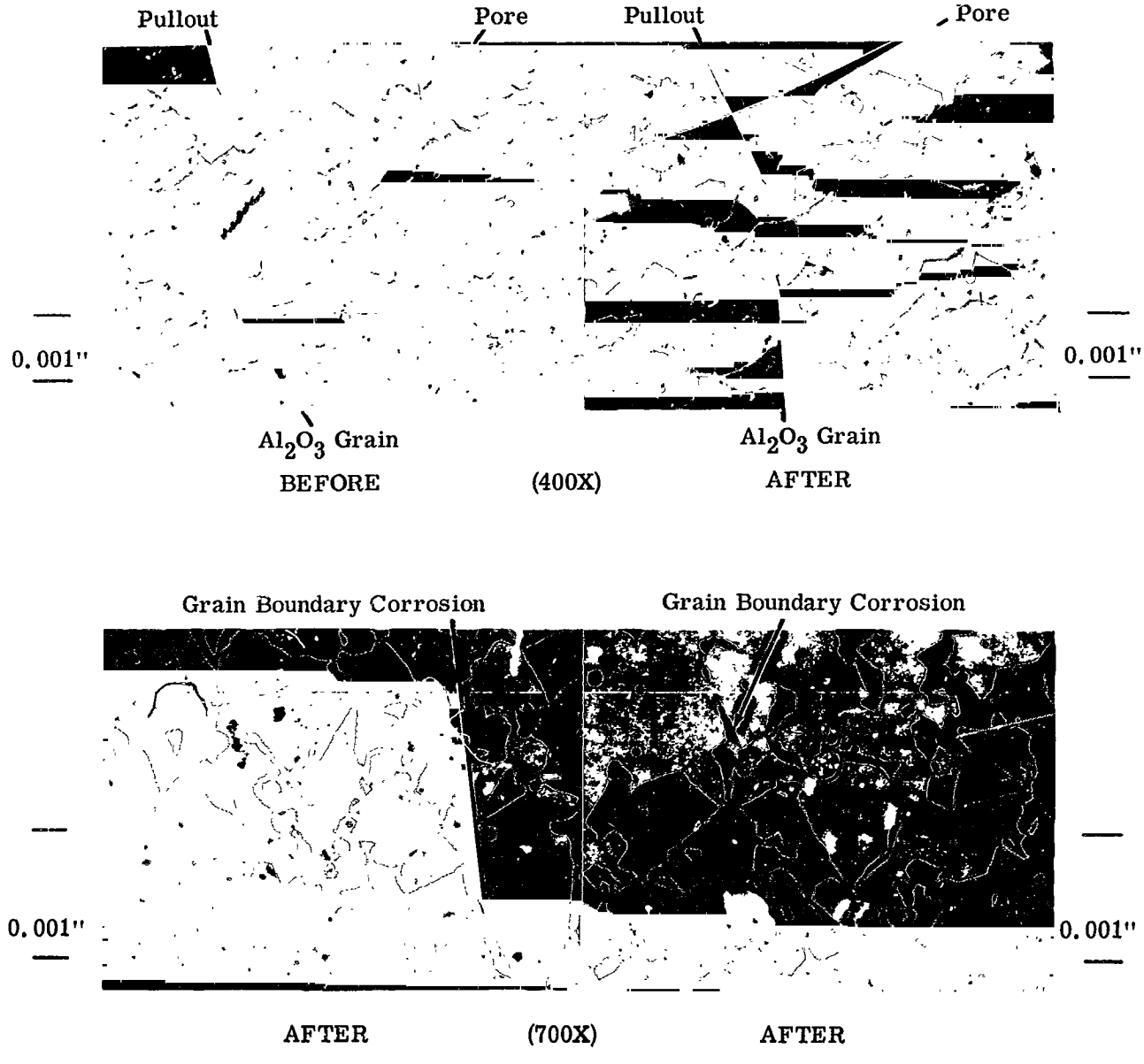


FIGURE II-9. Photomicrographs of Ei3-3W Alumina Before and After 500 Hour, 1600°F Potassium Vapor Exposure. Not Etched. Before Photo Reduction.

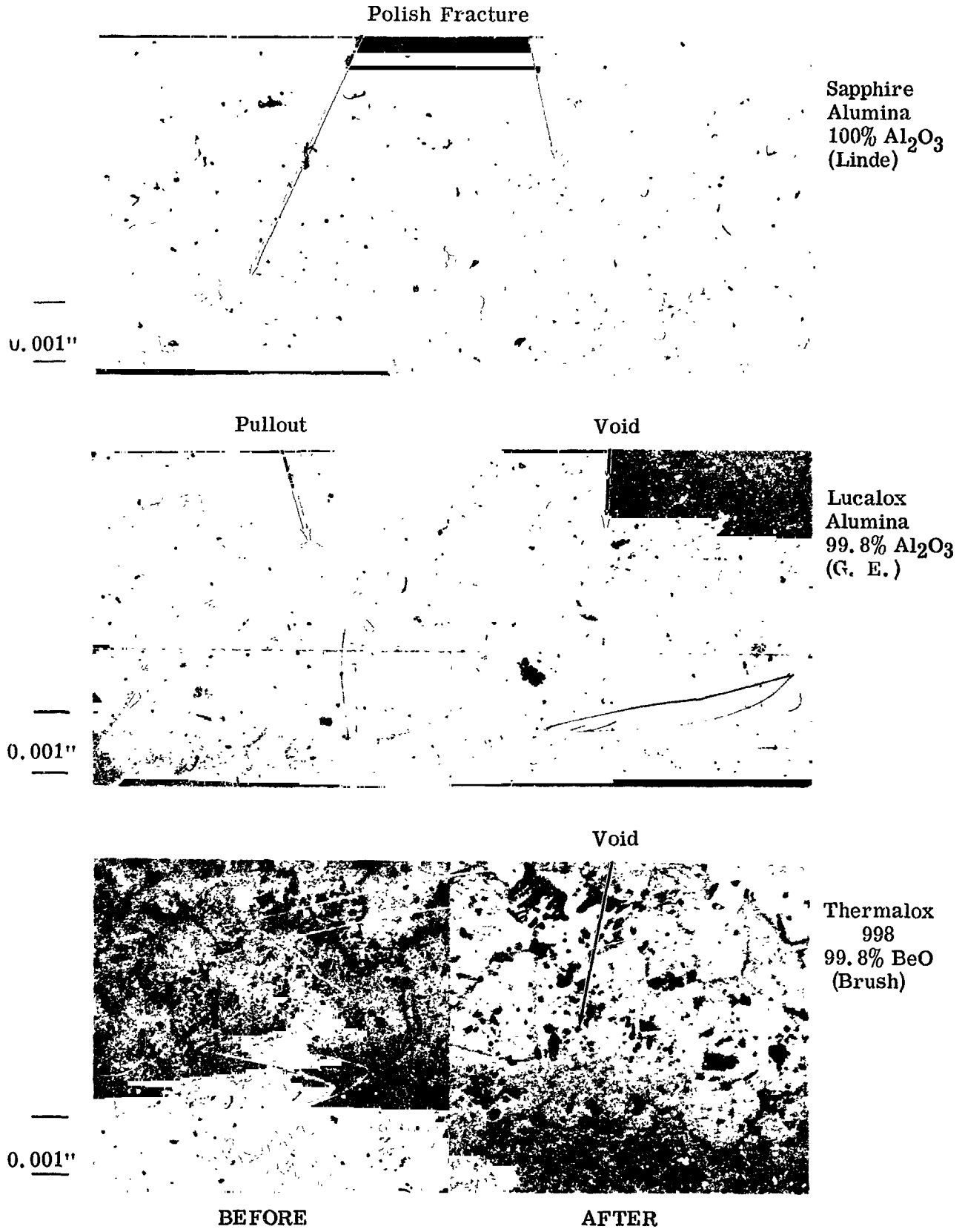


FIGURE II-10. Photomicrographs of Sapphire, Lucalox and Thermalox 998, Before and After 500 Hour, 1600°F Potassium Vapor Exposure. HF Etch (10%) 5 seconds on BeO Samples, None on Others. (400X Before Photo Reduction)

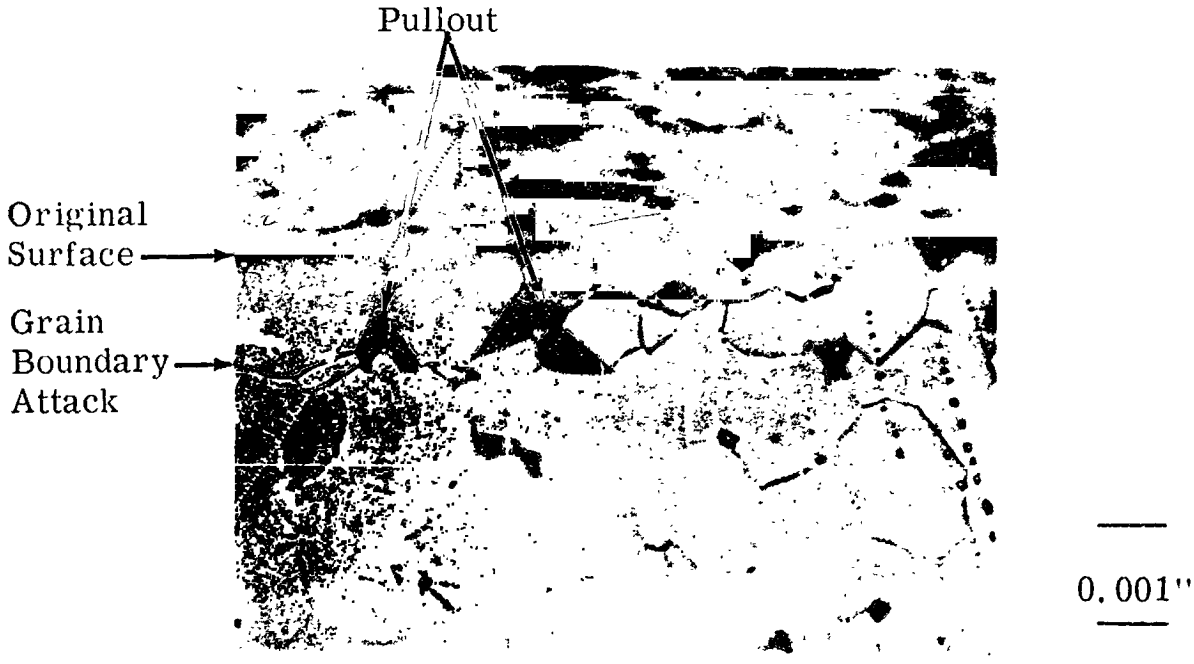


FIGURE II-11. Photomicrograph of Lucalox After 500 Hours in Potassium Vapor at 1600°F, (400X). Not Etched. Note surface grain boundary attack. Pullouts at the surface may be initiated by intergranular penetration by potassium.

FIGURE II-12. Photomicrograph of Thermalox 998 After 500 Hours in Potassium Vapor at 1600°F, (400X). Surface Grain Area. Not Etched. Note absence of intergranular attack or grain pullouts.

The Ei3-3W alumina body was attacked at 1600°F resulting in a 75 percent reduction in modulus-of-rupture. Significant grain boundary corrosion and reaction is indicated by the wide inter-crystalline gaps shown in Figure II-9. These gaps were noticeable throughout the sectioned bar. The broken bars were moderately darkened throughout (see Figure II-8). The silica content of these bars is shown by two sources in Table II-7. The second SiO_2 column shows spectrographic data obtained on all the exposed bodies at the same time under the same conditions for comparative purposes. This data shows the silica content of the Ei3-3W alumina bars used in this test to be at least 10 times that of the Lucalox bars. The corrosion data shows this lot of Ei3-3W alumina to be definitely unsatisfactory for the 1600°F potassium vapor environment.

Lucalox was slightly attacked as indicated by a faint darkening throughout the broken bars, a slight reduction in flexural strength, and occasional evidence of grain boundary erosion as illustrated in Figure II-11. This material is satisfactory for the 500 hour, 1600°F potassium vapor test environment but would probably deteriorate further with increased temperature or time.

Sapphire bars were totally unaffected as far as could be determined by flexural strength, evidence of darkening or microstructure differences (Figure II-10). This confirms the point that deterioration of alumina ceramics in high-purity potassium vapor is due exclusively to the non-alumina phases present along the grain boundaries. The large standard deviation in sapphire testing is due to random crystal orientation during tests. Sapphire displays anisotropy in physical and mechanical properties. The beryllia bars were slightly affected as evidenced by a reduction in flexural strength, no visible discoloration and no evidence of grain boundary erosion after the potassium vapor exposure (Figures II-10 and II-12).

Since neither the BeO nor the Al_2O_3 grains are themselves attacked, but only the intergranular material, a plot of degradation (or compatibility) versus SiO_2 content, regardless of primary phase, should be meaningful. Such a plot is given in Figure II-13 from data presented in Tables II-7 and II-8 and making the assumption that single crystal sapphire approaches

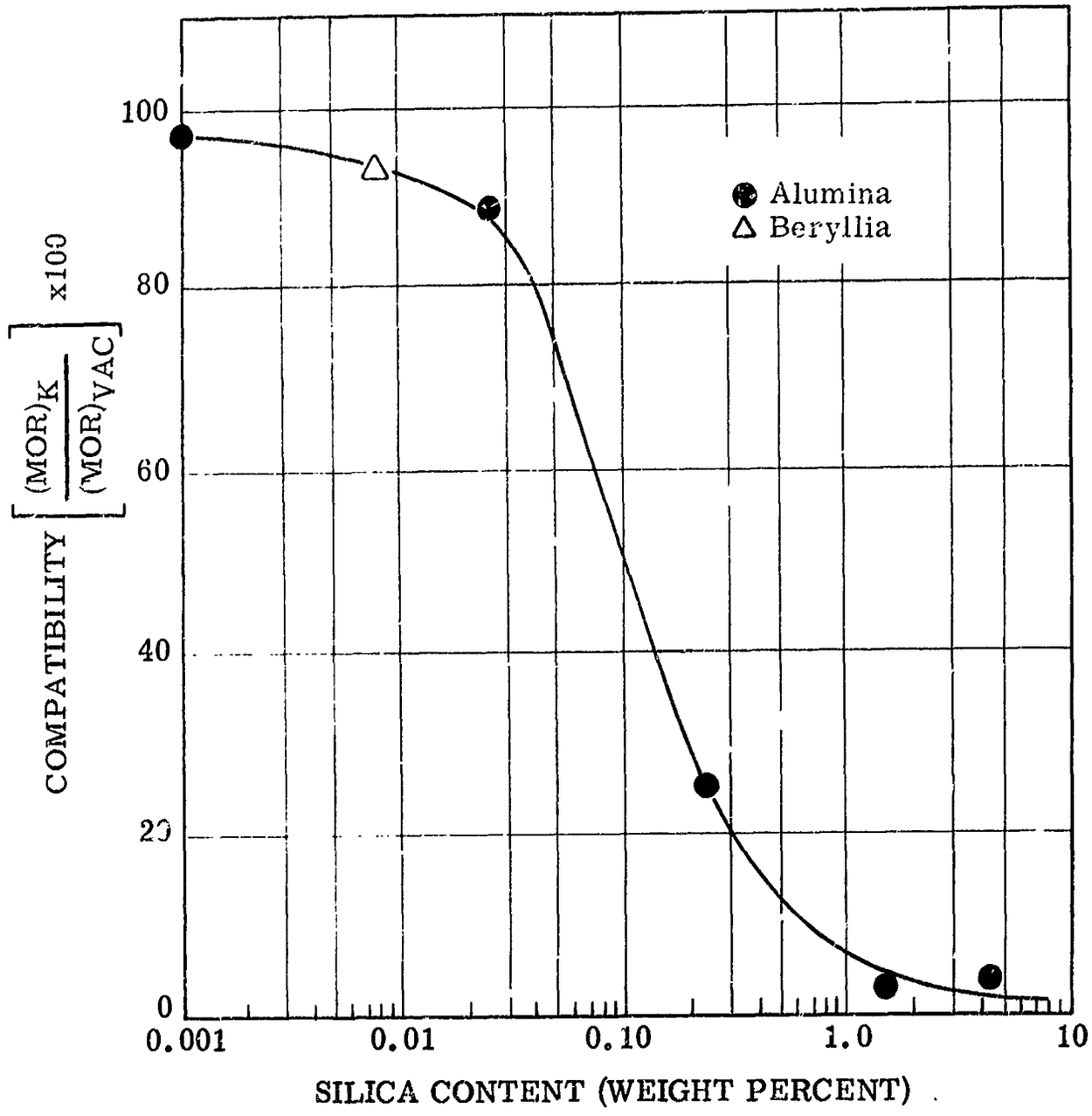


FIGURE II-13. Effect of the Silica Content of Alumina and Beryllia on Their Compatibility with Potassium at 1600°F.

Determined by ratio of room temperature flexural strength of modulus-of-rupture bars exposed to potassium vapor and to vacuum, respectively, at the exposure test temperature for 500 hours.

K = potassium test environment 1600°F
VAC = vacuum test environment 1600°F

zero percent silica. For this plot degradation was defined as the ratio, expressed in percent, of the room temperature flexural strength of the vacuum controls and the potassium vapor exposed pieces after 500 hours at 1600°F. For long-term usefulness at 1600°F it appears that silica contents of less than 0.05 percent are required. This in effect rules out any ceramic body which is fabricated and processed in equipment which is also used to produce silica containing bodies, since cross contamination rules out the possibility of achieving the less than 0.05 percent SiO_2 under such conditions.

The mechanical and physical properties of 99.8 percent beryllia are reported in Section IV-1. The strength is less than that of high purity, high density alumina, but care in design may compensate for the lower value. Recent work at Brush Beryllium Company indicates the possibility of achieving much higher flexural strengths by close regulation of the firing program and by grain size control. One of the most useful properties of high purity beryllia is the very high thermal conductivity. This property along with its low expansion coefficient results in a ceramic with superior thermal shock capabilities.

Although the low silica alumina body Lucalox is not readily fabricated into large, thin-walled bore seal geometries, it may warrant additional testing. It may withstand extended exposure at 1000°F and be useful in small bore seal applications or for insulators or feed-through terminals. Material properties of this magnesium oxide modified alumina are reported in Section IV-2.

The high-purity bodies were not significantly affected in percent loss in strength by mixing with high silica ceramics in the same test capsule (Table II-8). Loss in strength of Ei3-3W alumina was much less severe at 1000°F (6.3%) in potassium that at 1600°F (78%) as shown in Tables II-9 and II-8 respectively. However, sodium-potassium eutectic reduced the strength of Ei3-3W after exposure at 1000°F to about half of the original value. Beryllia showed no substantial difference in strength after potassium or lithium exposure at 1000°F (8.7% and 13.2%) or in potassium at 1600°F (10.8%).

b. METAL MEMBERS

Metal members for alkali metal containment systems are being specifically investigated on other programs and, therefore, extensive studies were not within the scope of the present program. The columbium-1% zirconium alloy has high hot strength and excellent alkali-metal compatibility and was utilized for most of the ceramic-metal joining investigation reported here. Columbium base D-43 alloy which provides superior hot strength and creep properties, was tested in a limited number of modulus-of-rupture ceramic to metal seal assemblies. Tantalum alloy T-111 was evaluated as a back-up metal. The columbium-1% zirconium alloy was used for the static test capsules because of the welding and corrosion information available on this alloy at the time of testing.

A sketch of the final exposure test capsule and purity test capsule designs is shown in Figure II-14. Purity test capsule and test capsule assemblies are shown in Figure II-15. In Figure II-16 photomicrographs of the bottom seam weld illustrate the grain growth near the heat-affected-zone. Table II-10 shows effects of welding and 500 hour 1600°F vacuum exposure (potassium inside capsule) on oxygen pick-up by the Cb-1Zr columbium alloy capsule. Welds were made in the controlled atmosphere glove box (Figure III-12) by TIG welding when water and oxygen levels were below 20 ppm total. Titanium sheet specimens which were welded to test the purity of the atmosphere in the welding chamber prior to welding the test capsules were clean and bright. The capsule in Item 2 of Table II-10 was a purity test capsule which had been heated in the center region with a low intensity arc to cause potassium to flow to bottom of capsule. This heating operation probably caused the relatively high oxygen in this region through gettering. The center portion of capsule C had been heated in the same manner as capsule A (Item 2). Metallographic examination of potassium exposed capsules showed no apparent corrosion of the Cb-1Zr (Figure II-17). The three refractory alloys being considered on this program exhibited equivalent wetting characteristics with the active metal braze alloys tested. Ceramic-to-metal joints made with modulus-of-rupture bars indicated similar joint strengths using columbium-1% zirconium or columbium D-43 alloy. Since the metal members appear equally suited for bore seals based on brazing test to

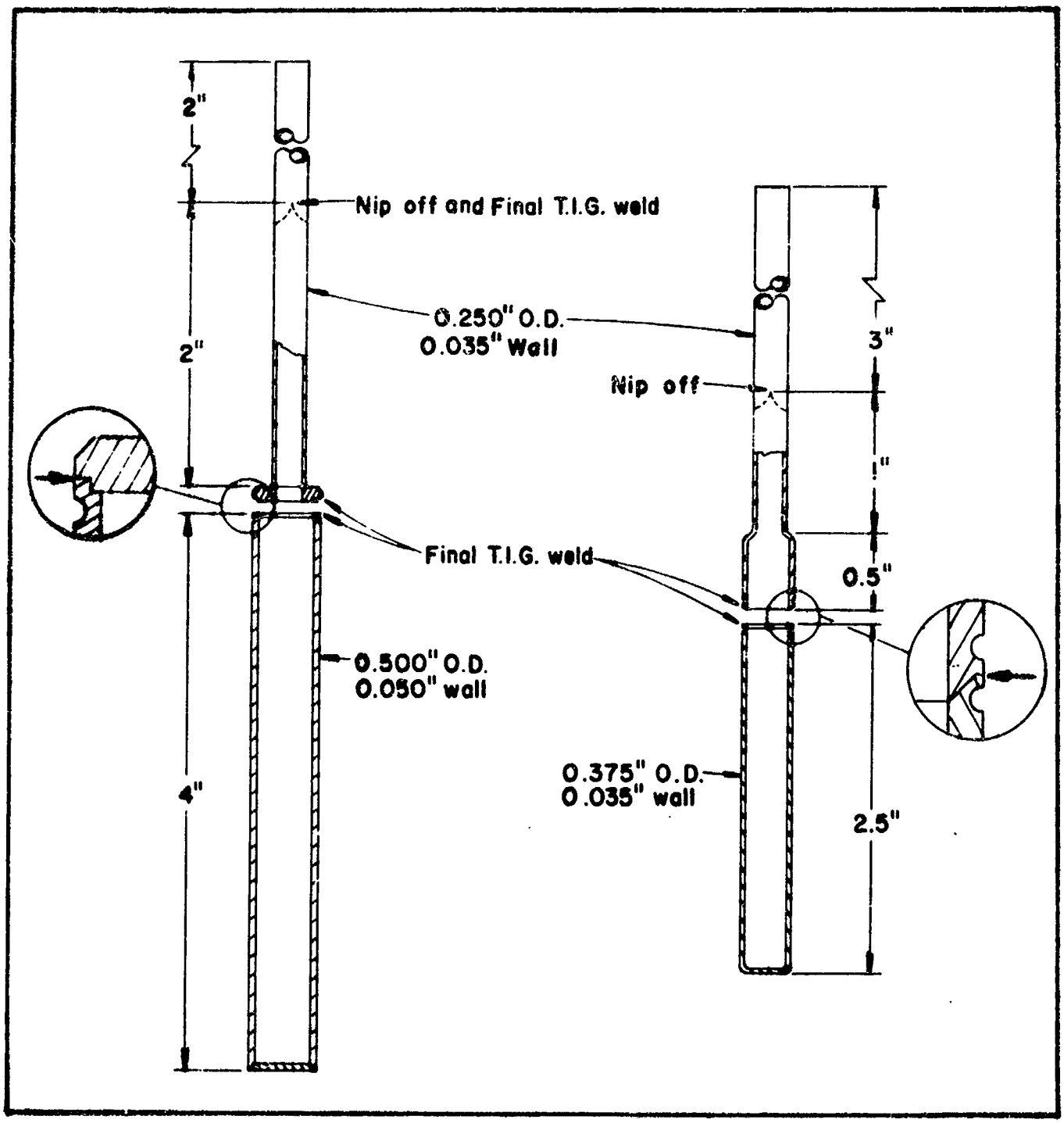


FIGURE II-14. Columbium-1% Zirconium Alloy Capsules for Ceramic Exposure and Alkali Metal Purity Control Tests

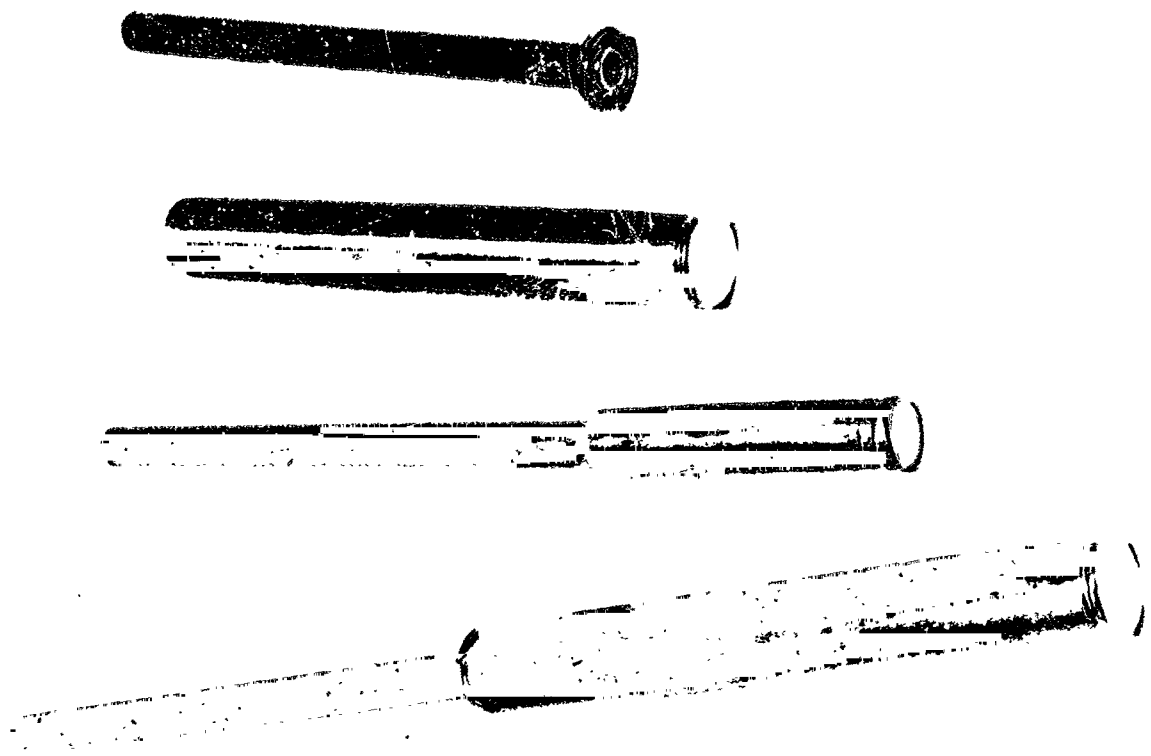


FIGURE II-15. Photograph of Welded Cb-1Zr Purity Test and Corrosion Test Capsules (Bottom) and Sub-assemblies (Top).

FIGURE II-16. Photomicrograph of Cb-1Zr Capsule Weld Area. Lower Right Insert Shows Pre-Weld Grain Size (20X). Polished with: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF. Etchant: 30 ml lactic acid, 10 ml HNO_3, 10 ml HF.

TABLE II-10. Oxygen Content of Cb-1Zr from the Wall of Capsules After Welding and After 500 Hour 1600°F Vacuum Exposure (K Inside Capsule)

Item	Sample	Oxygen Content[(a)] (ppm)
1	Manufacturer lot analysis	230
2	Center region of capsule A before potassium exposure test	294
3	Bottom weld of capsule A before potassium exposure test	266
4	Center region of potassium exposed capsule C	356

(a) Analysis of items 2, 3, and 4 by vacuum fusion by MSA Research, Inc. One test of each sample.

this time, the ultimate choice of the metal member will be based on long term aging effects and by the design and fabricability of larger (4 to 11 inch) diameter seals. An external factor which will direct final choice are metal member compatibility with the turbine coolant system.

c. CERAMIC TO METAL JOINING

(1) Metalizing - Braze Seals - Alumina

The first of the ceramic-to-metal sealing methods investigated on the currect program was the refractory metal metalizing system using a thermodynamically stable secondary phase.

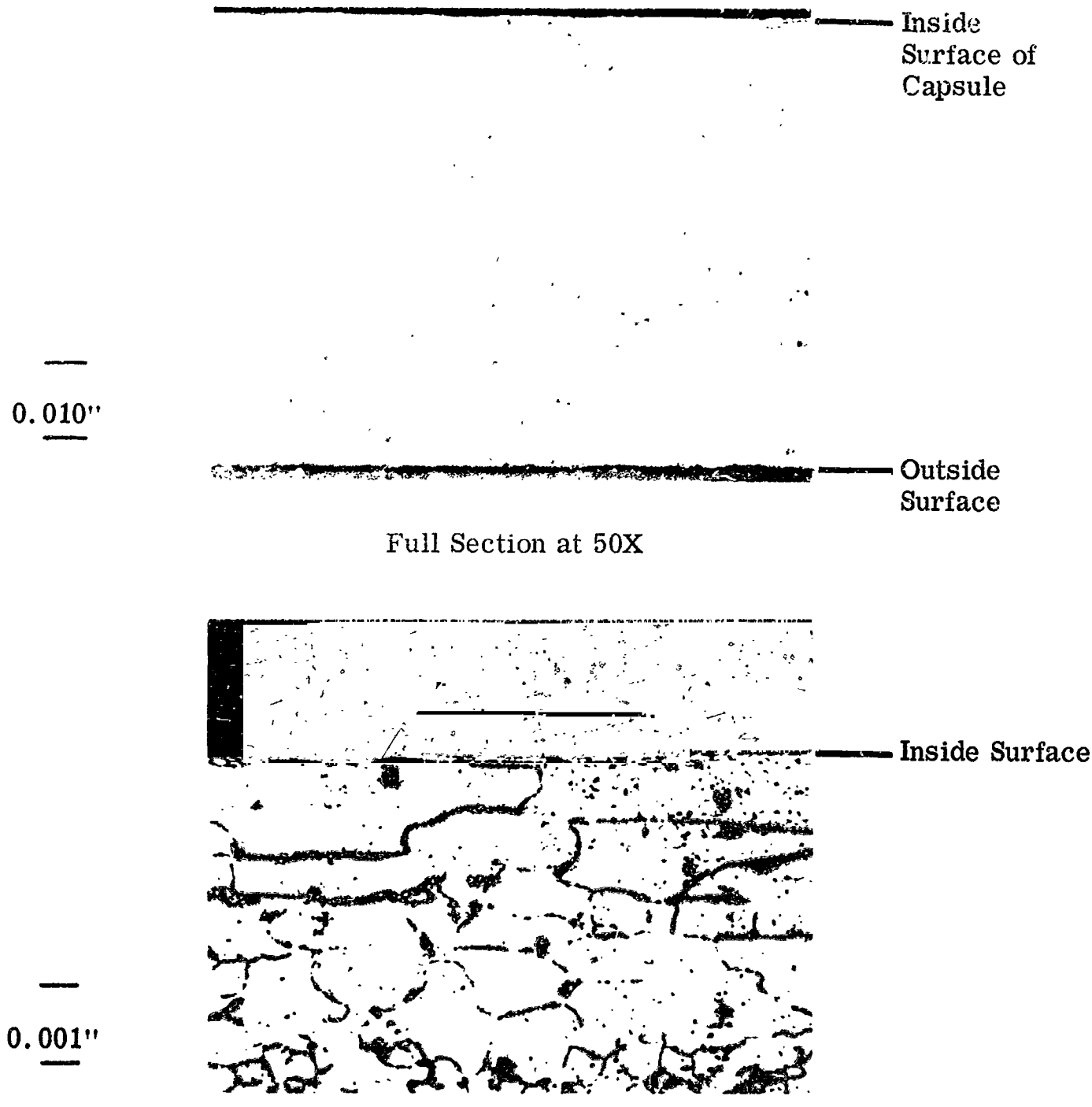


FIGURE II-17. Photomicrograph of Cb-1Zr Capsule After 500 Hours at 1600°F in Potassium Vapor. No evidence of attack by potassium was noted. Polished with: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF. Etchant: 30 ml lactic acid, 10 ml HNO_3, 10 ml HF.

The tungsten paints described in Table II-11 were developed to capitalize on the inherent resistance of calcia, alumina and the rare-earth oxides to attack by most alkali metals. Because these stable oxides have high-melting and sintering temperatures, a less refractory metal compound tungsten trioxide was utilized instead of molybdenum oxide. These paints were made in molybdenum-lined ball mills with molybdenum balls to minimize ball mill pick-up of non-metallic materials, particularly silica. Spectrographic analysis of four metalizing paints listed in Table II-12 indicate the low impurity level after ball milling. Silica content remained low after milling.

The metalizing paints were evaluated by brazing ASTM CLM 15 assemblies with copper which does not alloy with the metalizing. This made possible the evaluation of the metalizing-to-ceramic bond under the most ideal condition. Metalizings which exhibited satisfactory strength were then brazed with an alloy which is potentially useful in alkali metals (Coast Metals Braze 52 had shown stability in potassium vapor at 1100°F for 1000 hours on another program[11]. Brazing was done at 1850°F in a vacuum furnace at 1×10^{-5} torr with no holding time at temperature.

Initial tests made with the tungsten metalizing paints on 99 + percent alumina with copper braze on the current program indicated metalizing strengths of more than 10,000 psi (Table II-13). Subsequent, less promising, vacuum-leak test results with the tungsten metalizings and Coast Alloy Braze 52 (3B, 4.5Si, 0.15C, balance Ni) are shown in Table II-14. The final series of tests showed that these metalizing paints had insufficient adherence to high alumina ceramics for practical usefulness.

This was evidenced by the metalizing shearing off after the iron plating procedure (Figure II-18). The 0.0005

(11) Westinghouse Subcontract SPUR Project A F33(657)8954 and Westinghouse Subcontract A F33(657)10922. Westinghouse Report No. WAED 62.2E, January 1964.

TABLE II-11. Metalizing Paints for Alkali Metal Environments
(For Reference Only)

Paint Code (a)	Nominal Composition After Hydrogen Sintering (weight-percent)	
	Metallic Phase (b)	Non-metallic Phase
W5M	95 W	5 Y_2O_3
W8M	95 W	2 Al_2O_3, 3$CaCO_3$
W9M	95 W	2.5 Y_2O_3, 2.5Dy_2O_3
W10M	95 W	1.25 Y_2O_3, 1.25Dy_2O_3, 1.25 Nd_2O_3, 1.25 Gd_2O_3
W11M	85 W	15 Y_2O_3
W12M	85 W	6 Al_2O_3, 9 $CaCO_3$
W13M	85 W	3.75 Y_2O_3, 3.75 Dy_2O_3, 3.75 Nd_2O_3, 3.75 Gd_2O_3
W14M	95 W	2.5 Y_2O_3, 1 Al_2O_3, 1.5 $CaCO_3$
W15M	92 W	5.5 Y_2O_3, 0.5 MgO, 2 $CaCO_3$

(a) Eitel-McCullough Designation
(b) Added to the paint as WO_3; reduced to W by hydrogen during sintering at 3045°F in N_2-H_2 mixture

TABLE II-12. Spectrographic Analysis of Trace Impurities in Special Metalizing Paints (Typical Analysis)

Paint Code (a)	Impurity (weight percent)(b)							
	Mg	Al	Cu	Ca	Si	Mn	Fe	Mo
W5M	T	0.007	T	0.01	0.02	ND	0.02	0.02
W8M	0.0015	PC	T	PC	0.025	T	0.02	0.02
W9M	T	0.006	0.001	0.02	0.015	T	0.02	ND
W10M	T	0.003	T	0.01	0.01	T	0.01	ND

PC - Primary Constituent
T - Trace - Indicated less than 0.001 weight percent detected
ND - Not Determined
(a) Eitel McCullough designation. See Table II-11 for nominal compositions
(b) Semi-quantitative only ± factor of four

inch thick iron plating had been used as a barrier layer between the nickel braze alloy and the tungsten to reduce Ni-W alloying. The brittle sintered metalizing did not withstand the stresses imposed by differential thermal expansion of the electroplated iron-barrier layer. The cross-section of a W12 (Table II-11) metalizing paint on Ei3-3W alumina brazed with nickel base braze alloy (Coast Alloy 52) to a columbium metal member shows that shearing of the metalizing occurs at the tungsten to calcia-alumina glassy phase interface. In this case, the shearing probably occurred during the mounting or polishing operation. A sample of iron-plated standard number 20A (MoO_3-MnO-TiO_2) metalizing on AD 94 (94 percent alumina) ceramic was brazed to an iron plated columbium metal member with Coast Alloy

TABLE II-13. Tensile Strength and Leak Testing of Special Metalizing Paints Utilizing ASTM CLM 15 Tensile Test Assembly

Paint Symbol	Copper Braze[a]							Nickel Alloy Braze[b]		
	Ei3-3W (99.7 Percent Al_2O_3)				AD 99 (99 Percent Al_2O_3)			AD 99 (99 Percent Al_2O_3)		
	No. of Tests	Tensile Strength (psi)	No. of Tests	Leak Test	No. of Tests	Tensile Strength (psi)	No. of Tests	Leak Test	No. of Tests	Leak Test
W5M	1	>14 850	2	VT						
W8M	1	>12 900	2	VT						
W9M	1	>14 550	2	VT						
W10M	1	>13 200	2	VT						
W11M	1	>14 400	1	VT	2	>12 600	2	VT	1	LKR
W12M	1	6 030[c]	1	LKR	2	>11 250	2	VT	1	VT
W13M	1	>12 900	1	VT	2	>11 160	2	VT	1	VT
W14M	1	8 160[c]	1	VT	2	>13 110	2	VT	1	LKR
W15M	1	4 050[c]	1	VT	2	>13 290	2	VT	1	LKR

The prefix (>) indicates a metalizing strength greater than the figure shown. The specimen broke in the ceramic at the stress level noted.

VT indicates a leak rate less than 1×10^{-9} torr-liters/sec as determined in leak testing procedure Section III.C.3. LKR indicates leaker.

All paints listed were sintered for 1/2 hour at 3045°F in forming gas, $75N_2 - 25H_2$, 70°F dewpoint.

(a) Copper brazed in -100°F dewpoint hydrogen at 2040°F with 3 minute hold at temperature. 0.020 inch 70/30 cupro-nickel washer between CLM 15 pieces.

(b) Coast Metals Braze Alloy 52 (3B, 4.5Si, 0.15C, Ni balance) brazed at 1850°F in vacuum at 10^{-5} torr with no hold time at temperature. 0.015 inch Columbium washer between CLM 15 pieces. Columbium washer and metalizing plated with 0.0005 inch of Fe; vacuum sintered for 10 minutes at 1470°F.

(c) Specimens broke in the AD 94 side of the joint.

TABLE II-14. Leak Test Results - Tungsten Series Paints Ceramic-Metal Brazed ASTM C 'LM 15 Assemblies

Metalizing Paint (a)	AD 99 Alumina		Ei3-3W Alumina
	Columbium Washer	Kovar Washer	Cb-1Zr Washer
W11M	LKR	LKR	LKR(b)
W12M	VT	LKR	LKR
W13M	VT	VT	LKR
W14M	LKR	VT	LKR
W15M	LKR	VT	LKR

All paints sintered 1/2-hour at 3045°F in forming gas (75 N_2, 25 H_2) (70°F dewpoint).

All assemblies brazed with Coast Metals 52 Alloy (3B, 4.5Si, 0.15C, Ni balance): brazed at 1850°F in vacuum 10^{-5} torr; no holding time at temperature. Iron plated between washer and braze and between metalizing paint and braze.

VT - Vacuum tight (Leak rate less than 10^{-9} torr-liter/second. See Section III.C.3.).

LKR - Leaker (Leak rate greater than 10^{-9} torr-liter/second).

(a) See Table II-11 for nominal formulations

(b) Metallographic examination of similar samples showed that the nickel braze alloy penetrated the iron barrier and attacked the metalizing.

(Reference: NAS3-4162)

Scale 1 : 1

FIGURE II-18. Iron Plated Tungsten Metalize Paints on Alumina CLM 15 Specimens. Note that the tungsten metalizing has sheared off from the ceramic in some areas.

Braze 52. This sample which had been subjected to an identical brazing and polishing schedule showed no shearing. Additional effort on the non-silicate metalizing must involve the promotion of better bonding between the tungsten and the non-metallic phases.

Photomicrographs of these two systems are shown in Figure II-19a and b. In general, increasing the percentage of non-metallics in the tungsten paints from 5 percent to 15 percent (W11 through W15) resulted in sufficient 'glassy' phase being produced, see Figure II-19a; however, it may be noted that the tungsten metalizing was still somewhat porous. Apparently a high contact angle between the 'glassy' phase and the tungsten prevents complete penetration of the porous sintered tungsten. This explains why sintering temperatures of 3000°F or higher have to be used in spite of the fact that melting occurs at 2600°F in the CaO-Al_2O_3 system. The metalizing system must rely on solid state self sintering of the tungsten to occur rather than non-reactive liquid-solid sintering of a calcia-alumina-tungsten system.

At this time, it appears that non-silicate metalizing systems ($SiO_2 < 0.03$ percent) are only suitable for use in joints where stresses are at a minimum or are non-existent. The possibility of developing a stronger non-silicate bond using paints in the systems W5 through W15 exists by firing the metalized ceramics to a high temperature ($>3200°F$) and by 'glassy' phase control. However, firing of metalizing onto large thin-wall alumina cylinders at such high temperatures would result in severe distortion. Therefore, the investigation was not pursued.

The brazed Ei3-3W alumina pieces reported as vacuum leakers in Table II-14 were sectioned and studied metallographically to determine the cause of failure. Potentially useful insight to metalizing braze failures were observed. In numerous regions, the iron barrier plate over the columbium-1% zirconium washer was penetrated by the nickel braze alloy (Coast Alloy 52) and extensive solution of the columbium-1% zirconium

FIGURE II-19a. W12 Metalizing on Ei3-3W Ceramic (600X)

FIGURE II-19b. 20A Metalizing on AD 94 Alumina (600X)
Etchant: 50 ml lactic acid, 30 ml HNO_3,
2 ml HF for both micros.

metal member by the nickel braze followed. This solution of the columbium alloy by the braze alloy did not seem to be excessive along the braze-metal interface. However, opposite the regions of extensive solution of the Cb-1Zr, the metalizing layer was effectively destroyed, causing braze-ceramic interfaces of poor adherence, Figure II-20. The metallographic structures indicated that solution of the Cb-1Zr alloy by the nickel braze markedly lowers the melting point of the braze alloy and increased the activity of the braze alloy at the ambient brazing temperature.

Figure II-21 shows a crack in the Alloy 52 braze which occurred because of thermal stress in a region of high solution of the Cb-1Zr by the nickel. Attack of the metalizing and cracks in the braze layer are both undesirable effects. This might be reduced by formulating a more desirable brazing alloy in the columbium-nickel system which will allow lower brazing temperature and lower thermal stress in the joint.

Two other approaches aimed at improving the characteristics of metalizing braze systems utilizing thermodynamically stable oxides in the metalizing layer are:

a) Using the more ductile metal molybdenum in the metalizing and controlling the degree of sintering by particle size control.

b) Improving the iron barrier layer plating techniques to such an extent that no penetration by the nickel alloy occurs during brazing.

Because the active metal braze technique offered more promise, the additional work on the metalizing of alumina was discontinued.

2) Metalizing Braze Seals - Beryllia

A program to study the effect of sintering temperature on tungsten metalizing coatings which were painted on 99.8 percent beryllia (Brush Beryllium Thermalox 99.8) was completed. Scratch hardness and electrical resistivity were used as test criteria using the standard

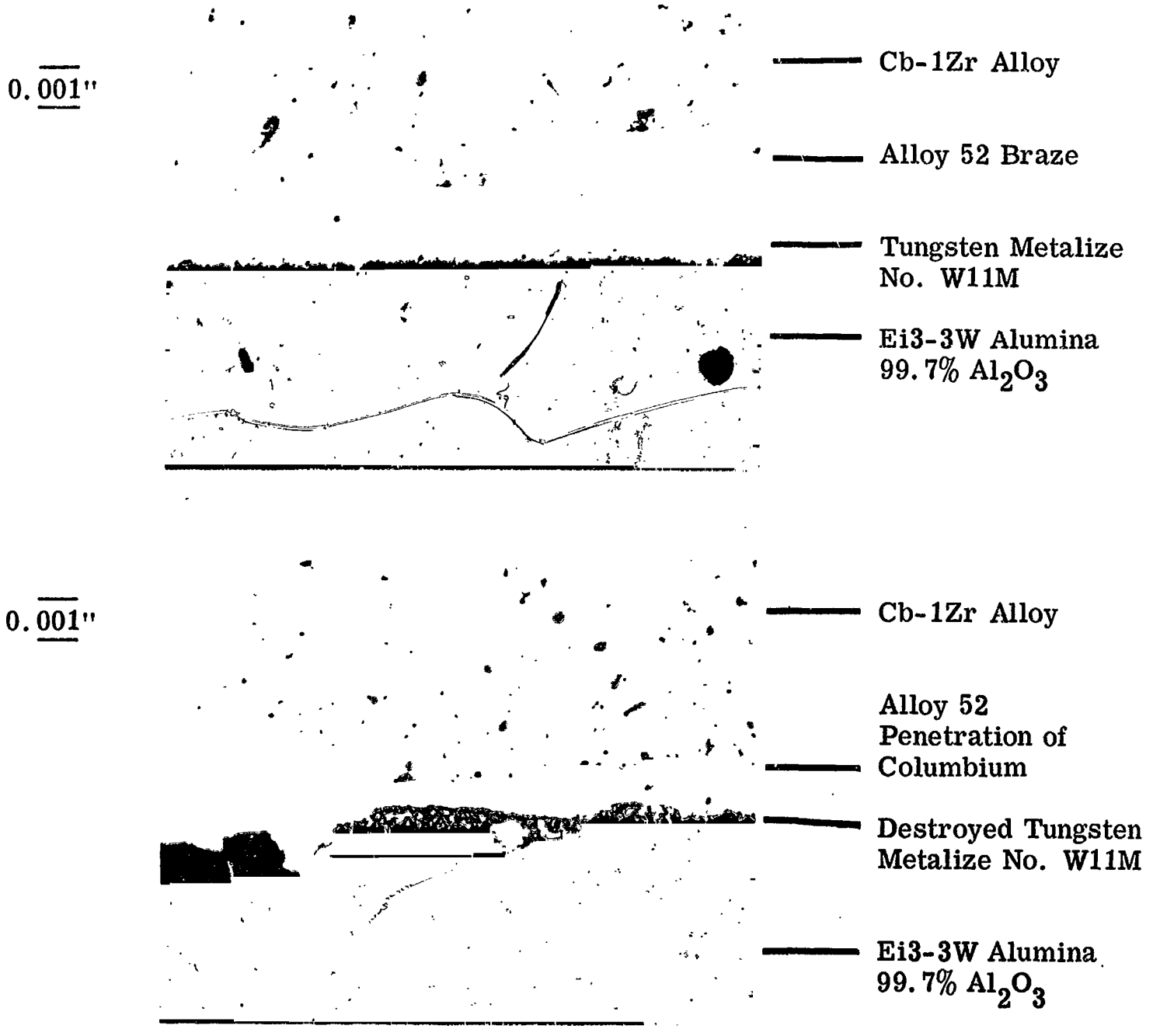


FIGURE II-20. Photomicrographs of Nickel Alloy Braze Seal Between Alumina and Columbium-1% Zirconium. (200X). Note the solution of the columbium-1% zirconium and the attack on the tungsten metalize after penetration of the iron barrier by the nickel braze alloy. Etchant: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF for both micros.

FIGURE II-21. Photomicrograph of a Nickel Alloy Braze Showing Stress Crack. (200X). Such defects appear to be associated with excessive solution of the columbium-1% zirconium metal by the nickel braze material.
Etchant: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF.

Mo-MnO-TiO_2 metalizing (20A) as a reference. The coatings did not develop the hardness and conductivity comparable with the Eitel-McCullough standard 20A metalizing until sintered for 30 minutes at 3000°F. The sintering temperature is approximately 200°F higher than the normal manufacturing firing temperature for the beryllia ceramic. The use of the high sintering temperature caused some of the beryllia pieces to deform and craze.

Copper and copper-silver brazes were used in evaluating the tungsten metalizings on beryllia bodies. Both brazes provide low thermal stress systems for determining the adherence of metalized coatings.

Vacuum leak checks on tensile test assemblies were inconclusive because the samples were deformed and cracked by the metalizing sinter treatment. In spite of the appearance of the beryllia bodies, two assemblies copper brazed to cupro-nickel alloy (70Cu-30Ni) with W13M metalizing paint and one assembly with W14M (Table II-11) paint were vacuum tight.

In general, the tungsten and rare earth oxide metalizing paints which were evaluated on this program with beryllia exhibited unusable low strength and work was discontinued in favor of the more promising area of active alloy sealing systems.

3. Active Alloy Brazing

a. DISCUSSION

Fourteen different braze alloys were studied on this program as potential 'active metal' joining materials for ceramics and metal-ceramic composites. These alloys were developed on other programs for brazing columbium and for resistance to elevated temperature alkali-metal environments (LB 20, LB 24, LB 156). Small ingots of these 14 compositions were prepared for evaluation as active metal braze alloys by the Battelle Memorial Institute using the inert-gas, non-consumable-electrode, arc melting process.

Since it was more convenient to handle the alloys as powders rather than in chunks, the cast ingots were comminuted to minus 50 mesh powder in a liquid nitrogen. A group of standard ASTM CLM 15 and modulus-of-rupture assemblies with Cb-1Zr metal members were joined using each of the 14 alloys. Results of the braze screening tests, made in a vacuum of 10^{-5} torr, are shown in Table II-15.

Two of the alloys, 75Zr-19Cb-6Be and 48Zr-48Ti-4Be, performed well enough to warrant extensive evaluation with alumina and beryllia in K and NaK. A third alloy, the beryllium free 56Zr-28V-16Ti material, showed sufficient promise in the screening tests with beryllia to warrant an evaluation in 1000°F lithium. Selection criteria for further testing was wettability, as indicated by a low contact angle between the alloy and ceramic; good strength, and hermeticity.

Three types of specimens; vacuum leak test, modulus-of-rupture (MoR) and tab peel assemblies of high purity alumina and beryllia were vacuum brazed to Cb-1Zr or D-43 alloy metal members with the above three brazing alloys. Elevated temperature 500 hour tests in the alkali metals were as follows:

Alkali Metal	Exposure Temperature (°F)
Potassium	1600
Potassium	1000
NaK Eutectic	1000
Lithium	1000

Unexposed and elevated temperature vacuum aged MoR bars were prepared for comparison with the alkali-metal exposed samples. A summary compilation of the various test data is presented in Tables II-16 and II-17. Photographs of selected capsule loadings before and after the 1600°F potassium exposure are shown in Figures II-22 and II-23. The metallography of representative exposed and unexposed 1600°F BeO brazements will be covered in the discussions on each test temperature - braze alloy - ceramic - metal member combination. However, before the individual combinations of alloy - metal member - ceramic and the actual test conditions are discussed, the basic structure and characteristics of the selected brazing alloys will be outlined.

TABLE II-15. Active Braze Alloy Preliminary Screening Using Cb-1Zr Metal Member with Designated Ceramic

Braze Composition (weight percent)	Brazing Temperature (°F)	Thermalox 998 99.8% BeO		E13-3W Al_2O_3		Remarks
		VT[b]	Strength (MoR)[a] (psi)	VT[b]	Tensile Strength[a] (psi)	
75Zr-19Cb-6Be	1940	2/4	15 260	4/4	9 750	Wets Columbium alloy D-43 well
68Ti-28V-4Be	2370	1/4	16 635	1/4 0/4	>240 >4 475*	Wets Columbium alloy D-43 & Tantalum alloy T111 well
56Zr-28V-16Ti	2270	(c)	15 740	2/4	4 850	---
48Ti-48Zr-4Be	1940	3/4	16 500	4/4 4/4	4 900 9 575*	Wets Columbium alloy D-43 well
46Ti-46Zr-4V-4Be	1830		16 650	4/4 1/4	--- 6 938*	---
50Zr-30V-20Cb	2415	3/4	15 175	0/4	140	---
65V-35Cb	3400					No test
70Ti-30V	3000					No test
60Zr-25V-15Cb	2435		14 035	0/4	50	Forms skull on Columbium alloy D-43 & Tantalum alloy T111
50Zr-30Ti-20V	2480	0/4	>4 742	0/4	>202	Forms skull on Columbium alloy D-43 & Tantalum alloy T111, Cb-1Zr alloy
40Zr-30Ti-30V	2335	4/4	13 165	0/4	245	Wets Columbium alloy D-43 & Tantalum alloy T111 well
35Ti-35V-30Zr	2595	0/4	>10 280	0/4	>500	---
50Ti-30Zr-20V	2595					No test
62Ti-30V-8Si	2480	4/4	8 370	0/4	>1 175	Wets Columbium alloy D-43 & Tantalum alloy T111 well

Brazed in vacuum furnace (10^{-5} torr) at temperature indicated; no hold time. Results shown are from the best braze run for each alloy.

* Made with AD 99 Alumina 99 + % Al_2O_3

> Indicates incomplete melting - greater strengths might be expected with increased temperature but were not attempted because of excessive pressure in the furnace at elevated temperature.

Underlined brazes and ceramic to metal assemblies were considered most favorable for further evaluation in potassium, potassium-sodium eutectic and lithium.

Notes:

(a) Modulus-of-rupture and tensile strength test procedures described in Section III.
(b) VT column gives number vacuum tight over total number tested.
(c) Vacuum tight assemblies fabricated previously on another program (SPUR, Westinghouse).

TABLE II-16. Effect of 500 Hour 1600°F Potassium Vapor Exposure on the Room Temperature Flexural Strength of Selected Ceramic-Metal Sealing Systems.

Ceramic	Braze (weight percent)	Brazing Temperature (°F)	Key	Flexural Strength (psi)		
				As Brazed	Vacuum Exposed 500 hrs, 1600°F	Potassium Vapor 500 hrs, 1600°F
Ei5-3W Alumina 99.7% Al_2O_3	75Zr-19Cb-6Be	1940	$\bar{x}$ s n	25 655 7 370 11	12 965 4 505[b] 2	0[a] - 4
Ei3-3W Alumina 99.7% Al_2O_3	48Ti-48Zr-4Be	1940	$\bar{x}$ s n	23 342 4 690 12	19 760 1 440[b] 2	0[a] - 4
Thermalox 998 Beryllia 99.8% BeO	75Zr-19Cb-6Be	1940	$\bar{x}$ s n	15 404 1 220 5	17 300 800[b] 2	< 1 000[c] - 5
Thermalox 998 Beryllia 99.8% BeO	48Ti-48Zr-4Be	1940	$\bar{x}$ s n	16 559 2 500 8	14 250 - 1	10 538[c] 3 740 5
Thermalox 998 Beryllia 99.8% BeO	56Zr-28V-16Ti	2270	$\bar{x}$ s n	13 503 2 870 6	13 985 1 715[b] 2	11 810[c] 850 5

All tests on modulus-of-rupture assemblies (MoR) using columbium-1%Zr metal member. Underlined ceramic-metal sealing systems appear to be the best of those tested

Key $\bar{x}$ - arithmetic mean
s - standard deviation
n - number of specimens tested

Notes:
(a) No MoR structures survived exposure test intact.
(b) Standard deviation has little significance for sample size of two pieces.
(c) Accompanying vacuum leak test assemblies were broken during removal from capsule.

TABLE II-17. Effect of 500 Hour 1000°F Potassium, NaK or Lithium Vapor Exposure on the Room Temperature Flexural Strength of Selected Ceramic-Metal Sealing Systems.

Ceramic	Metal Member	Braze Alloy (weight percent)	Brazing Temperature (°F)	Alkali Metal	Key	Room Temp. Flexural Strength (psi)			Alkali Metal Exposed Leak Test (e)	Remarks Room Temperature Flexural Strength and Leak Tests
						As Brazed	Vacuum Exposed 500 Hrs, 1000°F	Alkali Metal Exposed 500 hrs, 1000°F		
Thermalox 998 Beryllia 99.8% BeO	Columbium D-43	75Zr-19Cb-6Be	1940	K(a)	$\bar{x}$ s n	14 343 3 740 7	14 450 250(d) 2	14 340 490(d) 2	2/2 VT	Very good results
Thermalox 998 Beryllia 99.8% BeO	Columbium D-43	48Ti-48Zr-4Be	1940	K(a)	$\bar{x}$ s n	14 130 4 090 4	Not tested	9 710 1 940(d) 2	2/2 VT	Fair results
Thermalox 998 Beryllia 99.8% BeO	Cb-1Zr	56Zr-28V-16Ti	2270	Li(c)	$\bar{x}$ s n	13 503 2 870 6	12 895 555(d) 2	12 000(f) 1	4/4 VT	Good results
Ei3-3W Alumina 99.7% Al_2O_3	Cb-1Zr	75Zr-19Cb-6Be	1940	K(a)	$\bar{x}$ s n	25 655 7 370 11	21 100 3 300(d) 2	21 432 2 250 3	3/3 VT	Good results
Ei3-3W Alumina 99.7% Al_2O_3	Cb-1Zr	48Ti-48Zr-4Be	1940	K(a)	$\bar{x}$ s n	23 342 4 690 12	16 335 2 140 4	6 120 3 500 3	4/4 VT	Fair results
Ei3-3W Alumina 99.7% Al_2O_3	Cb-1Zr	75Zr-19Cb-6Be	1940	NaK(b)	$\bar{x}$ s n	25 655 7 370 11	22 130 3 300(d) 2	9 587 1 320 4	4/4 VT	Fair results
Ei3-3W Alumina 99.7% Al_2O_3	Cb-1Zr	48Ti-48Zr-4Be	1940	NaK(b)	$\bar{x}$ s n	23 342 4 690 12	16 335 2 140 4	10 390 1 820 4	4/4 VT	Fair results

All tests on modulus-of-rupture assemblies (MoR) using columbium-1% zirconium metal member.

Key: $\bar{x}$ - arithmetic mean
s - standard deviation
n - number of specimens tested

Notes.

(a) Oxygen level in associated purity test capsule was less than 10 ppm.
(b) Oxygen levels in two associated purity test capsules were less than 10 ppm.
(c) Associated purity test capsule leaked: no meaningful determination.
(d) Standard deviation has little significance for a sample size of two pieces.
(e) VT indicates helium leak rate of less than 1×10^{-9} torr-liter/second.
(f) One sample only

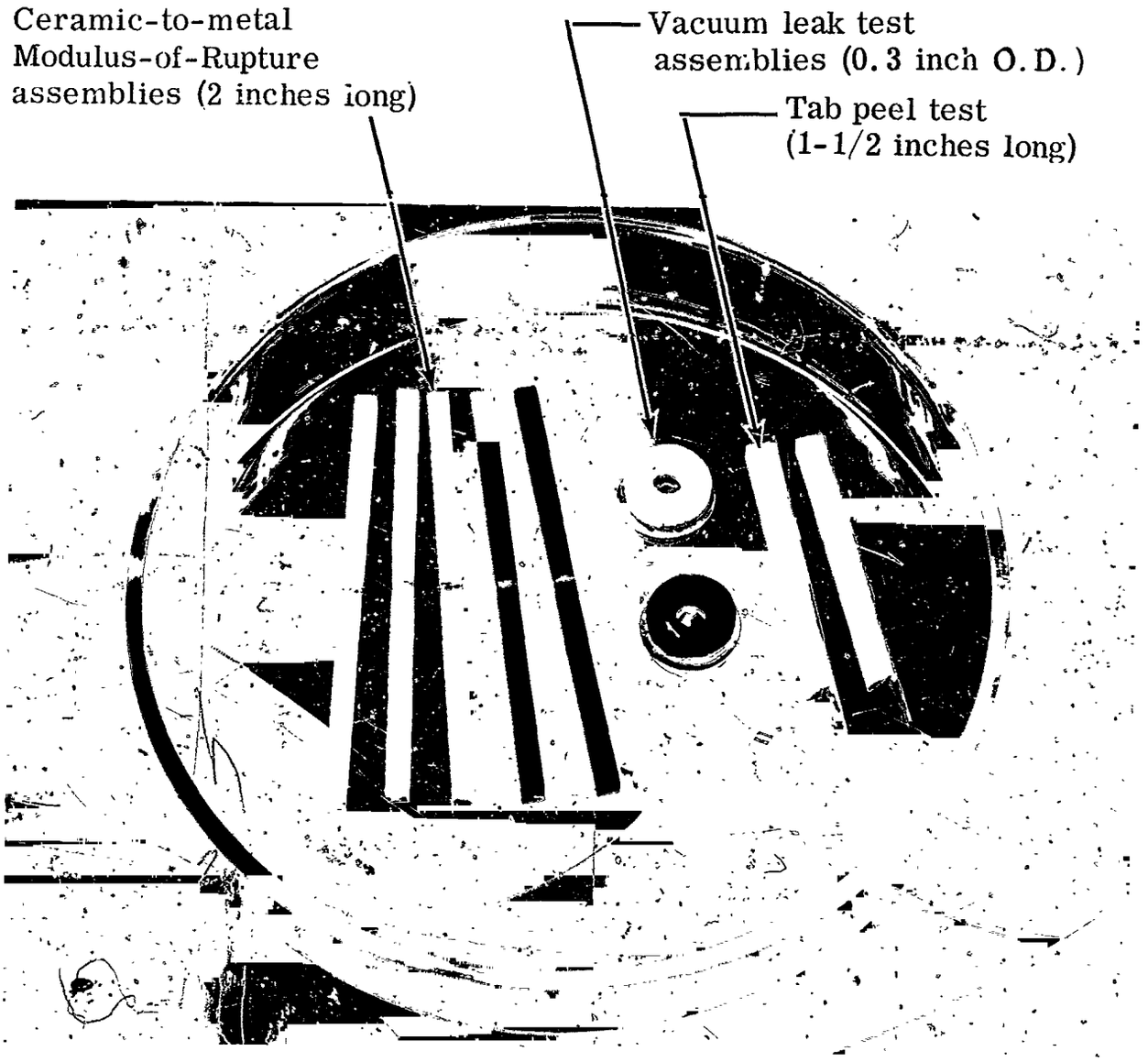


FIGURE II-22. Representative Set of Specimens for Alkali Metal Exposure

Example of poor ceramic-to-metal seals

Example of best ceramic-to-metal seal system tested

FIGURE II-23. Representative Sets of Ceramic-to-Metal Specimens After 500 Hour, 1600°F Potassium Exposure.
Top - Alumina Ei3-3W, Cb-1Zr, Alloy #4 (48Ti-48Zr-4Be), Bottom - Thermalox 99.8% BeO, Cb-1Zr, Alloy #3 (16Ti-28V-56Zr)

All three of the above brazing alloys selected for evaluation in alkali metal environmental testing are essentially eutectic in nature. The phase diagrams call for some primary or terminal solid solution. Those alloys which are wholly solid solution, have the disadvantage of melting at temperatures above 2900°F. Through the use of eutectic formers, such as beryllium, one is able to lower the melting and flow temperature of the braze alloys to the 2000°F - 2200°F level.

During the brazing cycle the V, Zr and Ti components of the braze can diffuse into the Cb-1Zr metal member, thus depleting the braze volume of V, Zr and Ti. In the case of the Be containing alloys this means that intermetallics such as $CbBe_{12}$, $ZrBe_2$ and $TiBe_2$ will isothermally deposit as the primary crystal phase. On cooling further precipitation will occur until the eutectic composition is reached.

The columbium from the metal member can dissolve in the altered braze alloy thus providing the possibility of a columbium rich phase. The solution can be large in the case of the columbium free braze alloys and limited in the case of the Zr-Cb-Be alloy. If columbium solution occurs in the Be containing brazes then intermetallics such as $CbBe_8$, $CbBe_5$ and $CbBe_{12}$ can successively precipitate[12]. In the case of the Zr-V-Ti braze the strong possibility of Cb-(Zr, Ti) eutectoid formation exists.

The reaction of the braze material with the ceramic is as follows:

$$(Ti, Cb, Zr, V) + BeO \longrightarrow TiO_x + CbO + ZrO_2 + V_2O_3 + Be.$$

b. RESULTS

1) Potassium Exposure Tests; 500 hour, 1600°F

The beryllia - columbium - 1% zirconium assemblies joined with 48Zr-48Ti-4Be and 56Zr-28V-16Ti alloys maintained flexural strengths above 10,000 psi after

(12) Arzhanyl, P.M., Volkava, R.M., Prokoshkin, D.A., Investigation of the System Niobium-Beryllium, Doklady Akademii Nauk SSSR, v 150, No. 1, pp. 96-98, May 1963.

the 500 hour test in 1600°F potassium. The performance of these parts in such an extreme environment is superior to that of any other known ceramic-metal-braze alloy combination (Table II-16).

A metallographic evaluation of each of the beryllia base (Cb-1Zr) brazed systems was performed. Samples were taken (1) in the as-brazed condition, (2) after 500 hours exposure to vacuum (10^{-6} torr), (3) after the 500 hour potassium exposure at 1600°F.

The micrographs of three samples brazed with the 48Zr-48Ti-4Be alloy are shown in Figure II-24. The eutectic phase consisting of a mass of beryllium intermetallics perhaps imbedded in a solid solution phase of (Zr, Ti, Cb) can be seen. The primary phase in all cases are most probably beryllides.

Micrographs of the system beryllia 75Zr-19Cb-6Be columbium-1% zirconium are shown in Figure II-25 before and after potassium and vacuum exposure. The photographs reveal structures which are quite different from each other. These differences are again probably the result of variable joint spacing. Any analysis of these micro structures must take note of differences in joint spacing into account.

As brazed, the structure appears as a fairly uniform Cb-Zr-Be eutectic containing some primary intermetallics nucleated on the Cb-1Zr member (top photo). Those grains of primary solid solution which nucleated in the liquid braze alloy formed dendritically as the composite cooled.

The BeO and Cb-1Zr combination, brazed with 56Zr-28V-16Ti alloy was the second of two ceramic-metal braze alloy combinations to withstand 1600°F potassium exposure. Micrographs of the as-brazed, vacuum control, and potassium exposed samples are shown in Figure II-26. As-brazed, the joint area is essentially four phase and may be divided into two areas. One area is probably a primary solid solution of Cb-Zr-Ti-V and the second area is probably the above combined with ZrV_2 in eutectic proportions. The structure of this alloy

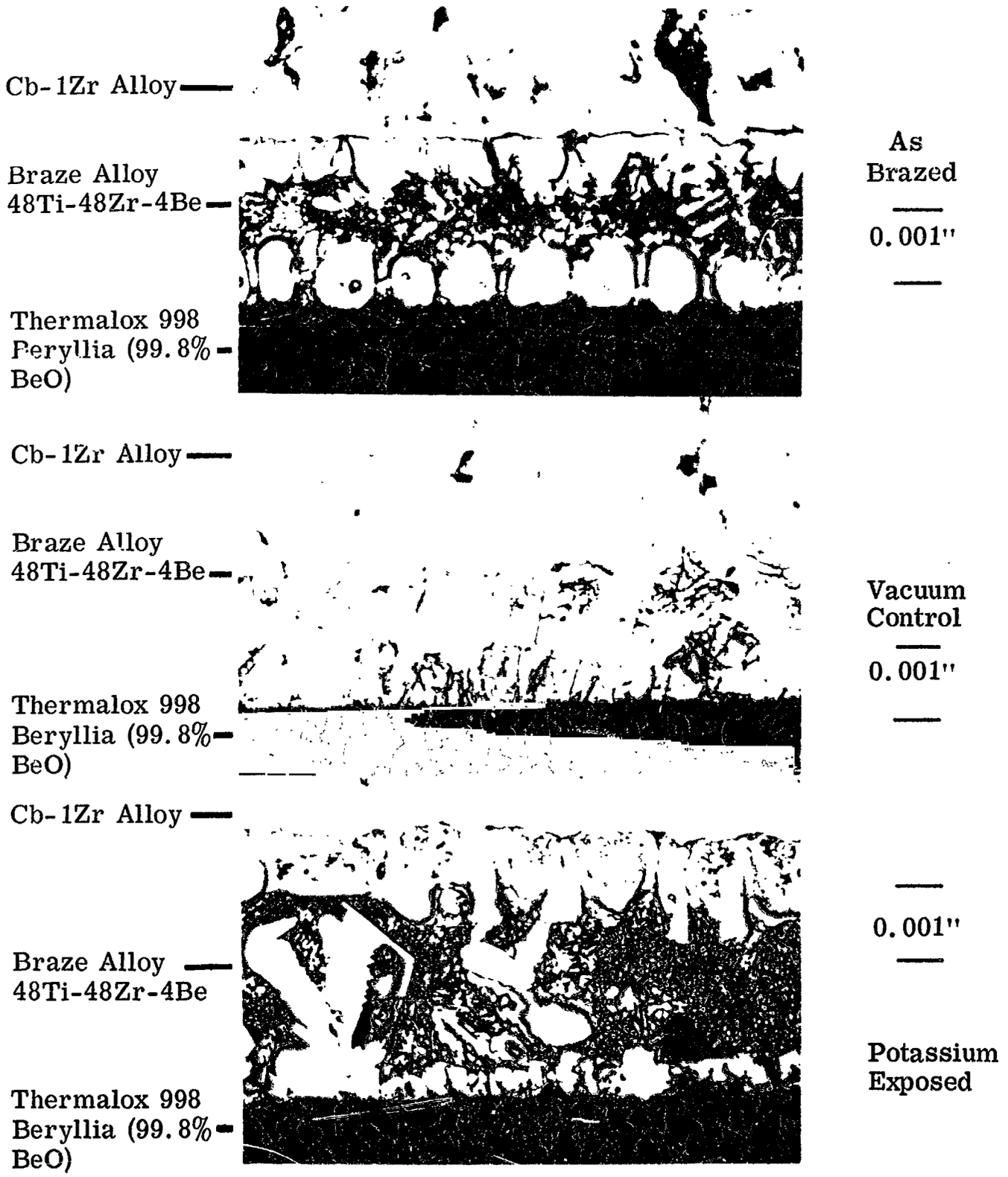


FIGURE II-24. Photomicrograph of BeO-(48Ti-48Zr-4Be)-(Cb-1Zr) System; as Brazed, After 500 Hours at 1600°F in Vacuum, After 500 Hours at 1600°F in Potassium Vapor. (400X) (Excellent System)
Etchant: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF.

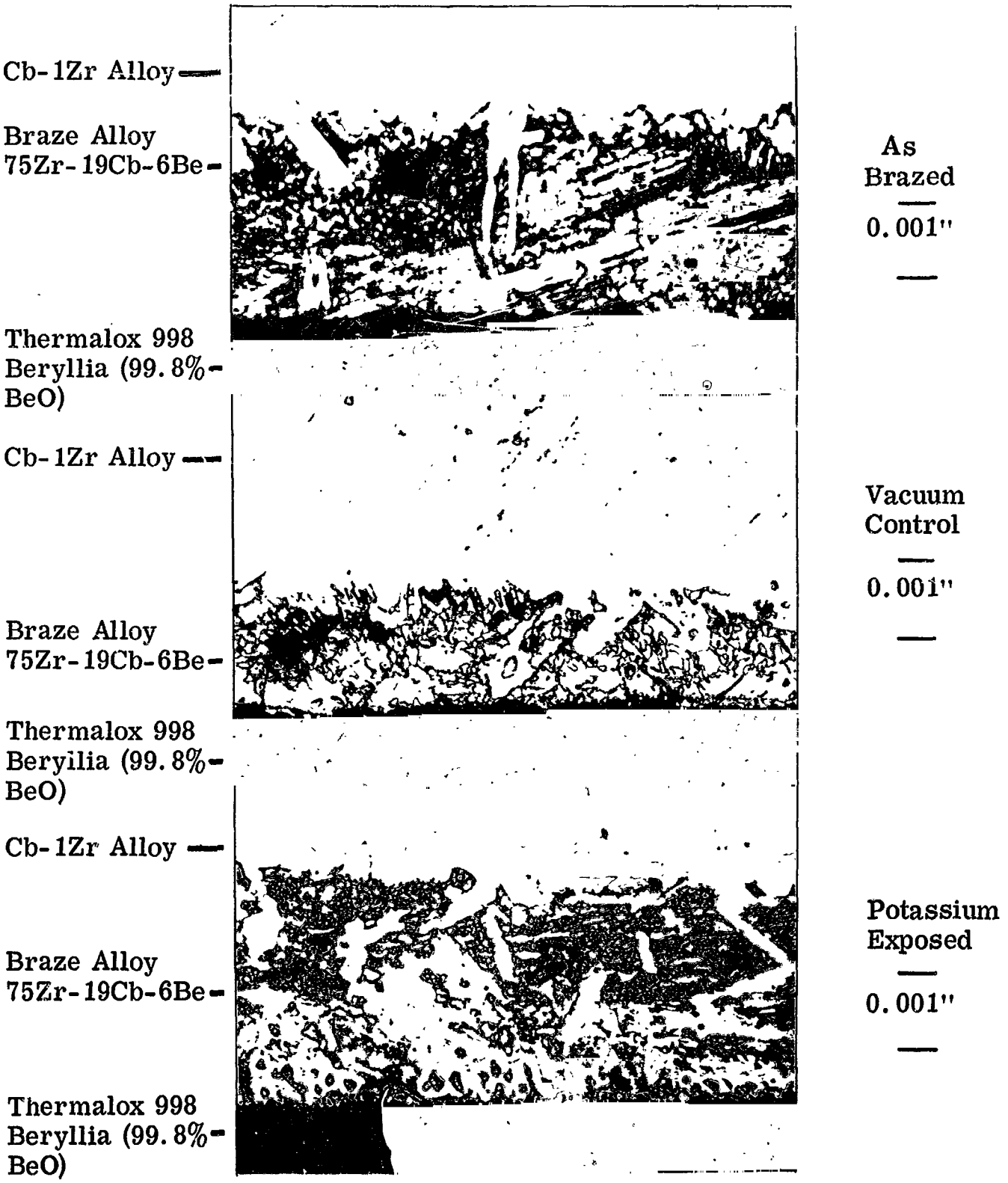


FIGURE II-25. Photomicrograph of BeO-(75Zr-19Cb-6Be)-(Cb-1Zr) System; as Brazed, After 500 Hours at 1600°F in Vacuum, After 500 Hours at 1600°F in Potassium Vapor. (400X) (Unsatisfactory System)
Etchant: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF.

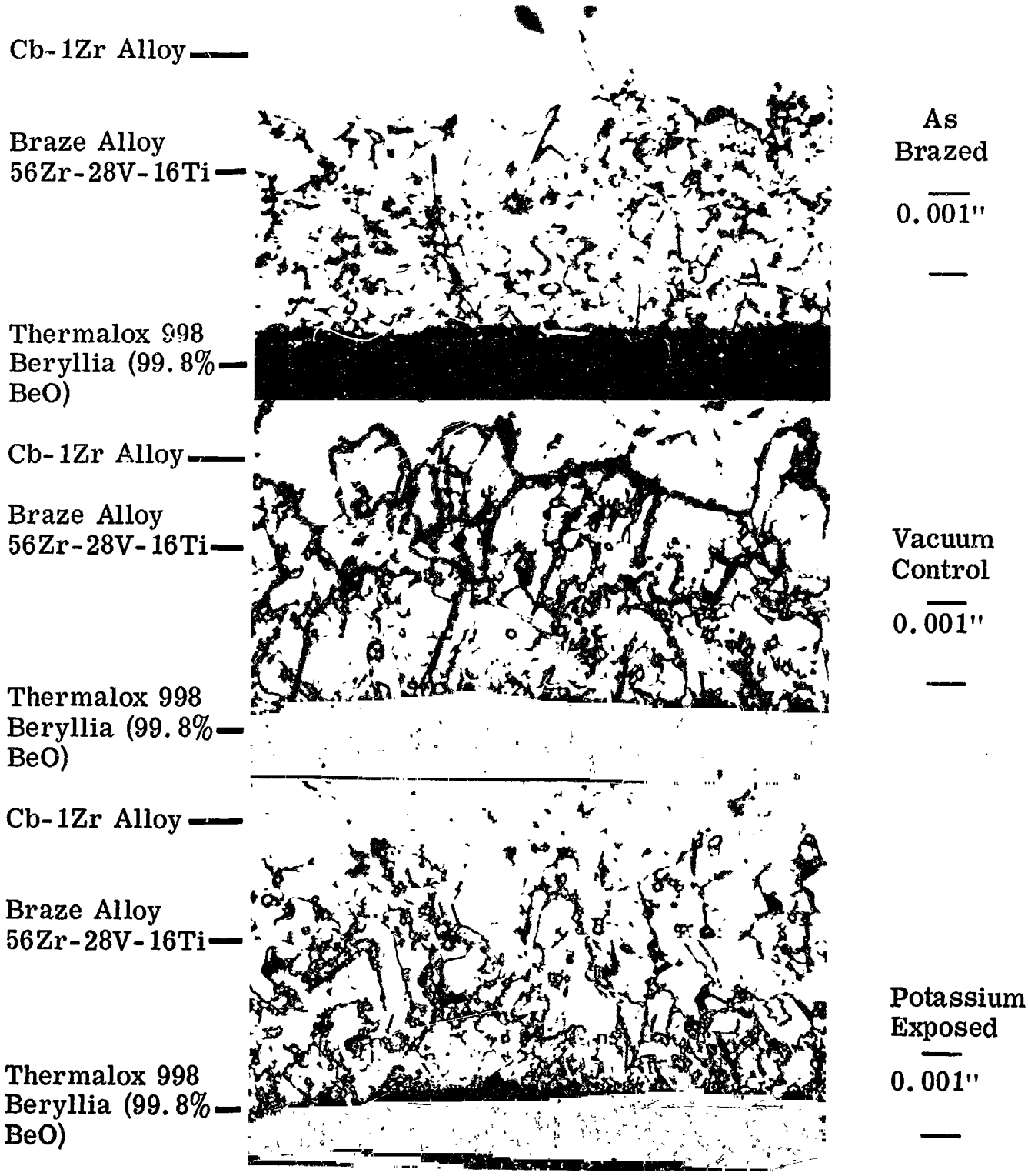


FIGURE II-26. Photomicrograph of BeO-(56Zr-28V-16Ti)-(Cb-1Zr) System, as Brazed, After 500 Hours at 1600°F in Vacuum and After Exposure in Potassium Vapor for 500 Hours at 1600°F. (400X)
Etchant: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF.

is complicated by the potential precipitation of eutectoids notably Cb-Zr at the 600°C level on cooling.

The differences in microstructure in all three cases are attributed to the amount of braze material, brazing time and temperature rather than to exposure for 500 hours at 1600°F.

Metallographic studies performed on the alumina-(Cb-1Zr) active alloy seals revealed as-brazed structures which were nearly identical to those achieved on the BeO-(Cb-1Zr) seals. Because of this similarity, and since none of the alumina seals survived 1600°F K metal exposure, no micrographs of the alumina seals will be presented in this report. Failure of these seals also occurred at the ceramic-braze alloy interface.

2) 500 Hour Exposure in 1000°F Potassium, Sodium-Potassium Eutectic and Lithium

All test data from the 1000°F exposures are presented in Table II-17. Three of the systems tested survived both potassium environment and vacuum endurance tests with little or no reduction in strength. None of the 1000°F leak test specimens failed the post exposure hermeticity tests. Systems BeO-(D-43)-(75Zr-19Cb-6Be), BeO-(Cb-1Zr)-(56Zr-28V-16Ti) and alumina-(Cb-1Zr)-(75Zr-19Cb-6Be) all offer the promise of long life capability at 1000°F. The strength of the remaining systems listed in Table II-17 were reduced appreciably by the exposure tests. Such behavior indicates the occurrence of chemical or metallurgical changes at the braze-ceramic interfaces which cause a reduction in strength for the system.

Beryllia assemblies utilizing the 75Zr-19Cb-6Be braze alloy and D-43 metal members indicated no deterioration in flexural strength after either vacuum or potassium exposure. The beryllia-(D-43) (48Ti-48Zr-4Be) modulus-of-rupture assemblies suffered about 30 percent degradation in the potassium. Beryllia to Cb-1Zr modulus-of-rupture assemblies brazed with 56Zr-28V-16Ti were subjected to 500 Hour, 1000°F lithium exposure. The

modulus-of-rupture assemblies were very difficult to remove from the capsule and were damaged during removal. None of the five assemblies broke in the braze joint indicating seal strengths on the order of that of the ceramics themselves, or about 19,000 psi. One of the seals had sufficient ceramic attached to permit unconventional flexural test which indicated a strength in excess of 12,000 psi. All cylindrical vacuum test assemblies of the above systems were vacuum tight. A leak occurred in the lithium purity test capsule in the pinch-off area. Inference of the lithium purity in the exposed capsule was therefore impossible.

The microstructures of the 1000°F alkali metal vapor exposed seal systems showed no new details when compared to the 1600°F exposed specimens.

c. ACTIVE ALLOY BRAZE EVALUATION

At least one potentially useful ceramic metal member and braze alloy has been evaluated to 500 hours in each test environment (Table II-18). In some cases, such as 1000°F NaK, the tested systems were being attacked and longer exposure would probably result in complete degradation.

Most conclusions to this point have been based on modulus-of-rupture data, with some vacuum leak test data. Tab peel test pieces were also made for each system, although in most cases there was insufficient space in the test capsule to include tab peel samples for alkali metal vapor exposure. A summary of tab peel test data is given in Table II-19.

The tab peel strengths were all under 30 lb/in. except for that of 75Zr-19Cb-6Be on Ei3-3W which was over 200 lb/in. No lower practical limit has been determined from these tests. The trend of lower strength with higher brazing temperature observed with the 48Zr-48Ti-4Be alloy, if significant, indicates that the brazing temperatures can be optimized for stronger seals. While considerable effort was made to achieve reproducible brazing cycles for the assemblies made on the present program, the microstructure variations noted between supposedly identical samples indicate that additional controls are required to eliminate brazing time-temperature differences as a variable.

TABLE II-18. Best Ceramic-to-Metal Seal System Tested in Each Alkali Metal-Temperature Environment

Environment	Best Ceramic to Metal Seal System Evaluated		
	Ceramic	Braze Alloy	Average Room Temperature Flexural Strength[b] (psi)
Potassium 1600°F	Thermalox 998 Beryllia 99.8% BeO	56Zr-28V-16Ti	11 810
Potassium 1000°F	Ei3-3W Alumina 99.7% Al_2O_3	75Zr-19Cb-6Be	21 432
NaK 1000°F[a]	Ei3-3W Alumina 99.7% Al_2O_3	48Zr-48Ti-4Be	10 390
Lithium 1000°F	Thermalox 998 Beryllia 99.8% BeO	56Zr-28V-16Ti	> 12 000

All seal systems were made with 0.015 inch thick columbium-1% zirconium alloy metal member.

(a) Marginal usefulness after 500 hours.
(b) Post exposure flexural strength.

TABLE II-19. Tab Peel Strength of Active Alloy Braze Systems at Room Temperature

Ceramic	Braze Alloy	Braze Temperature (°F)	Average Room Temperature Tab Peel Strength (lb/in.)	Number of Specimens
Ei3-3W Alumina 99.7% Al_2O_3	75Zr-19Cb-6Be	1985	214	5
Ei3-3W Alumina 99.7% Al_2O_3	48Zr-48Ti-4Be	1940 2010	16.4 14.0	4 5
Thermalox 998 Beryllia 99.8% BeO	75Zr-19Cb-6Be	1985	19.5	8
Thermalox 998 Beryllia 99.8% BeO	48Zr-48Ti-4Be	1940 2010	15.0 9.3	7 3
Thermalox 998 Beryllia 99.8% BeO	56Zr-28V-16Ti	2265	23.0	4

All systems were made with 0.015 inch thick columbium-1% zirconium metal member.

Another significant variable from the standpoint of alkali metal corrosion is the oxide content of the braze material. This is important since the oxides of vanadium and titanium, when present, represent relatively unstable compounds in the seal. The oxide content of the braze is affected by:

1) absorbed surface gas in the braze powder.
2) oxidized surfaces in the braze powder.
3) brazing atmosphere.
4) reaction with ceramic being brazed.

These parameters are all controlled to some extent. The amount of oxide contributed to the first two items should be ascertained. The later two parameters represent recorded items in the brazing log book (vacuum, time, temperature).

4. Other Sealing Systems

a. ELECTROFORMED SEALS

Two samples of metalized AD 94 ceramic were joined to a columbium washer by electroforming in a low-stress nickel sulphamate solution. One electroformed seal was designed as shown schematically in Figure II-27. Difficulty was encountered in building up a nickel deposit of desirable thickness in the root region because of the limited throwing power of the plating bath. Although the nickel plate in the critical root region was less than 0.003 inches thick, the seal was leak tight. The sample was tensile tested but proved to be very low in strength.

Another sample was designed to eliminate the necessity of plating nickel in a deep groove and is shown in Figure II-28. The ends of the AD 94 alumina pieces were metalized and butted against the columbium washer. A small fillet of graphite powder was applied between the columbium washer and the metalized ceramic to assure a continuous electrical field at the joint. This sample was also leak tight.

Seals formed in this manner are too weak for use in load bearing structures.

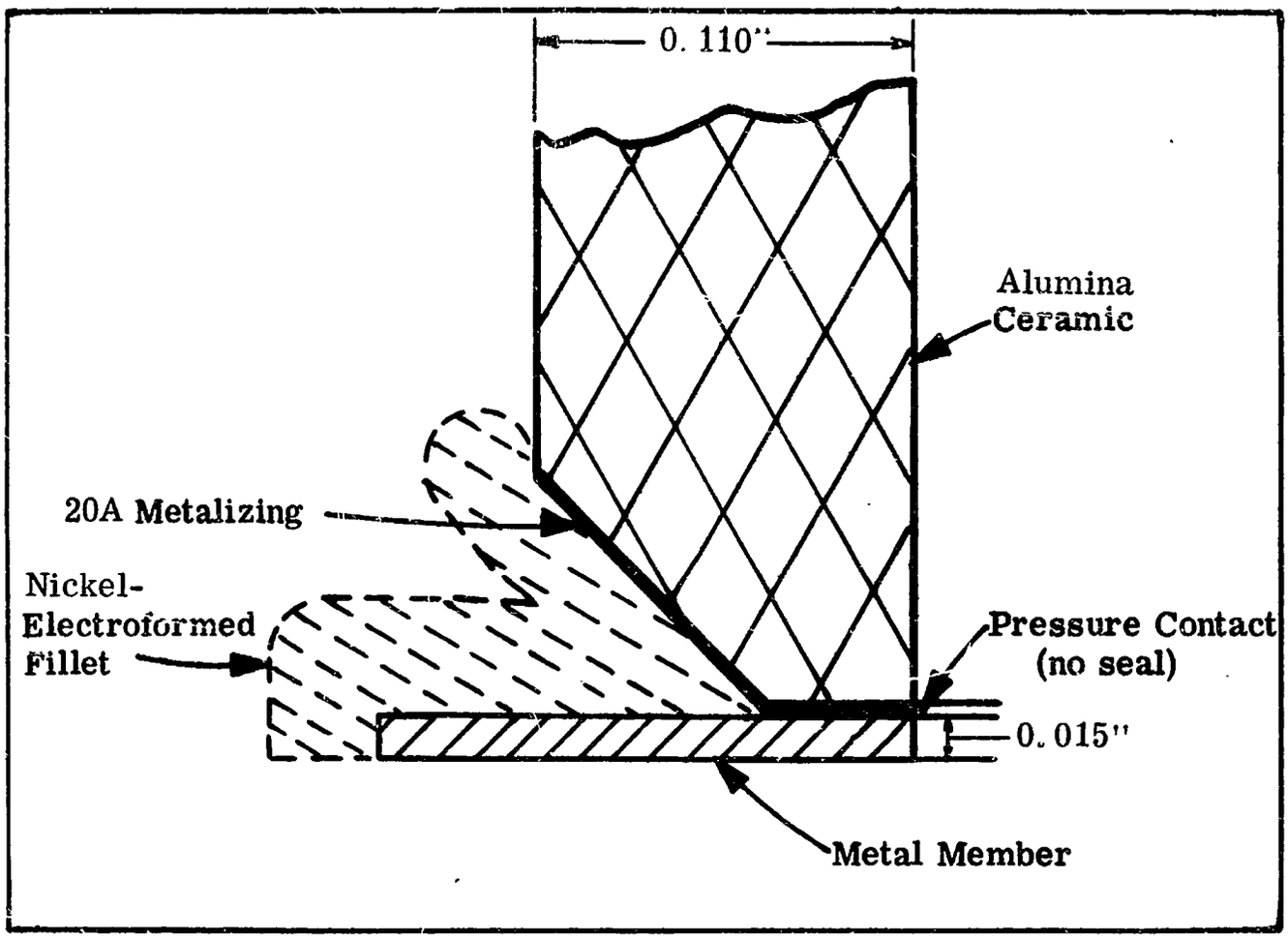


FIGURE II-27. Idealized Geometry of Electroformed Seal

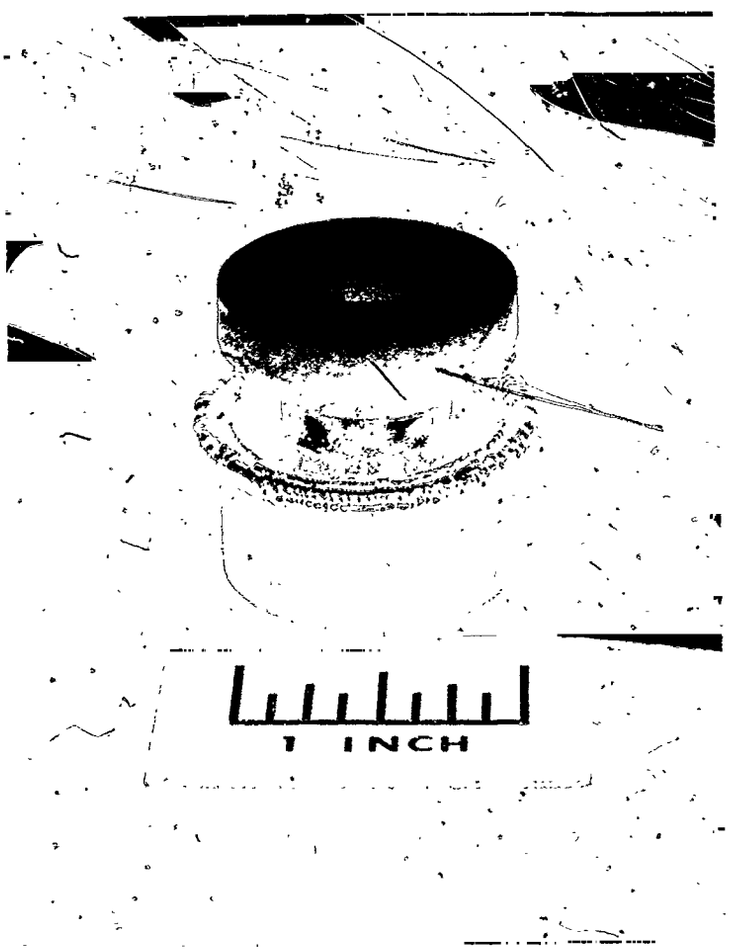


FIGURE II-28. Alumina to Columbium-1% Zirconium Seal Joined by Electroforming.

b. THIN FILM METALIZING-BRAZE SEAL

Evaporated thin film refractory metal metalizing was previously developed at Eitel-McCullough[13] and appeared promising for bore seal applications. Tensile strengths of over 15,000 psi had been obtained in the absence of a non-metallic phase in the sealing system. Vacuum-tight seals to 96 percent BeO had been fabricated by this method. Preliminary work was done with evaporated molybdenum and copper brazing. Nickel alloy brazing was then evaluated with iron and chromium barrier layers over the thin metalizing to prevent erosion during the brazing operation.

A summary of both copper and nickel alloy brazing results of thin film-metalized ceramics on this program is presented in Table II-20. A seal, which had been fabricated with a Coors AD 99 (99% Al_2O_3) ASTM CLM 15 tensile specimen using thin film metalizing techniques, was sectioned for metallurgical examination. The seal had been made by evaporating a film of titanium less than one micron thick on the ceramic body followed by a layer of molybdenum slightly greater than one micron thick. After a 0.0005 inch thick plating of iron was applied, the part was sintered at 1475°F for ten minutes in vacuum. The prepared ceramic was then brazed in a vacuum of 6×10^{-5} torr at 1850°F to iron plated 0.015 inch thick columbium with Coast Metals Braze alloy 52 (3B, 4.5Si, 0.15C, balance Ni). The other half of the CLM 15 assembly consisted of an AD 94 alumina ceramic, metalized with 20A metalizing and iron plated as described above. The seal tested at room temperature indicated a vacuum leak and had a tensile strength of 6,400 psi. In Figure II-29 a photomicrograph of the thin film metalized side of the seal is shown. The iron plating is intact on both the columbium and metalized ceramic. The possibility of ruptures in the iron plate and erosion of the thin molybdenum layer by the brazing alloy in other portions of the seal exists, with associated degradation of the metalizing-ceramic bond. The iron plate may also be reacting with the thin molybdenum layer with similar degradation. The separation of molybdenum

(13) Patent No. 3, 115,957 - others pending.

TABLE II-20. Tensile Strength and Leak Testing of Thin Film Metalizing on CLM 15 Assemblies

Ceramic	Metalizing	Plate	Vacuum Braze	Metal Member	Tensile Strength (psi)	Leak[f] Test
AD 995[a] polished	(b)	Cu flash	Cu[d]	Cupro Nickel (70Cu-30Ni)	>15 500	1 VT
AD 995[a] polished	(b)	Cu flash	Cu[d]	Cupro Nickel (70Cu-30Ni)	>13 375	1 VT
AD 995	(b)	Cu flash	Cu[d]	Cupro Nickel (70Cu-30Ni)	>16 250	1 VT
AD 99[g]	(b)	0.0005 Fe	Ni[c]	Fe plated Cb[e]	6 400	1 LKR
AD 995	(b)	0.0005 Fe	Ni[c]	Fe plated Cb[e]	4 200	2 LKR
AD 995[a] polished	(b)	0.0005 Fe	Ni[c]	Fe plated Cb[e]	1 200	2 LKR
AD 995	(b)	0.0005 Cr	Ni[c]	Fe plated Cb[e]	600	2 LKR

(a) Ceramic surface polished prior to metalizing.
(b) Evaporated thin films; Ti $< 1\,\mu$, then Mo $> 1\,\mu$.
(c) Coast Metals Alloy 52 (3B, 4.5Si, 0.15C, bal Ni) brazed at 1850°F in vacuum 5×10^{-5} torr, no hold time.
(d) Copper brazed at 2000°F in vacuum 5×10^{-5} torr, no hold time.
(e) 0.015 inch thick columbium washer between CLM 15 pieces plated with 0.0005 inch thick iron.
(f) VT indicates leak rate less than 10^{-9} torr liters/second. LKR indicates leaker. See Section III.C.3.
(g) See Figure II-29.
(>) Indicates that specimen broke in the ceramic.

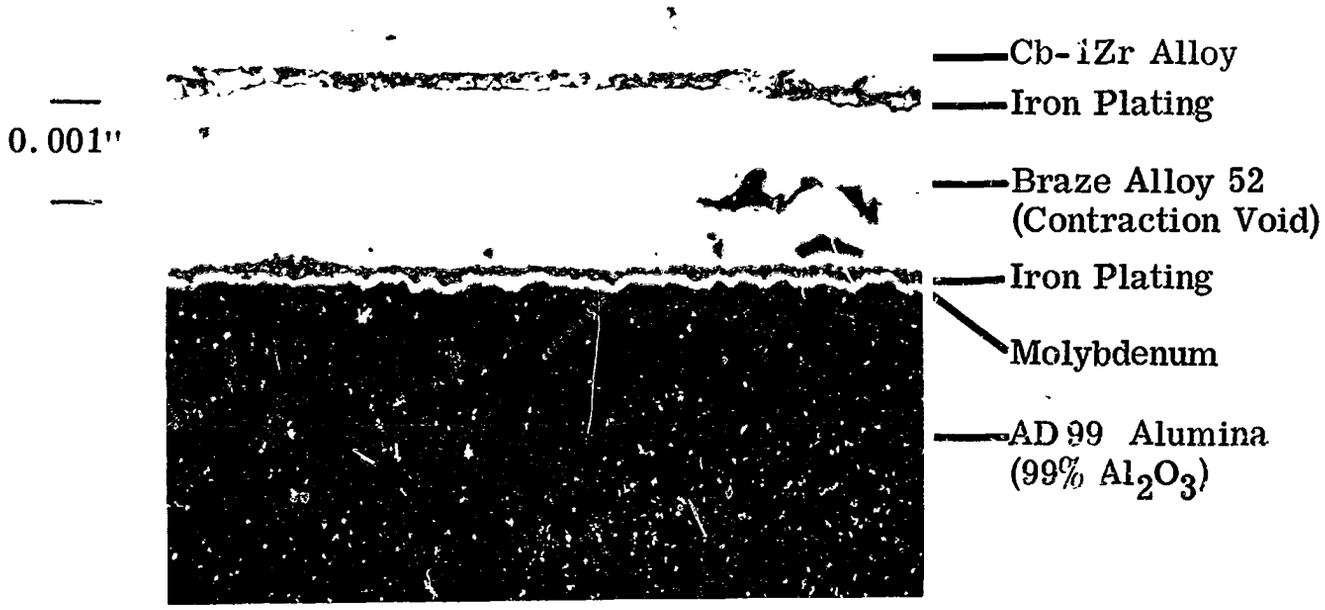


FIGURE II-29. Photomicrograph of Thin Film Metalizing With Alloy 52 Braze (400X)
Etchant: 50 ml lactic acid, 30 ml HNO_3, 2 ml HF.

and ceramic is significant and indicates that the evaporated molybdenum metalizing is not suitable for use in a highly stressed seal. None of the nickel alloy brazed assemblies were vacuum tight. Copper brazes which are more ductile than Alloy 52, have consistently produced vacuum-tight seals. Copper brazes, however, are not resistant to alkali metal vapors at elevated temperatures.

c. THIN FILM METALIZING TO PROMOTE WETTING

The use of evaporated coatings on ceramics with nickel-braze alloy resulted in seals which leaked due to attack of the braze material on the thin molybdenum through the barrier layer, but the application of evaporated coatings to promote ceramic wetting by active alloy is very encouraging.

Relatively thick, evaporated molybdenum coatings were prepared to aid the wetting of alumina by active metal braze materials. A sectioned CLM 15 tensile test which illustrates the additional braze flow obtainable is shown in Figure II-30. The bottom AD 99 ceramic piece was evaporation metalized with less than one micron thickness of titanium and a layer of molybdenum greater than one micron thick; the other ceramic piece was not metalized. The 19Cb-75Zr-6Be active braze alloy was placed on the outside of the columbium-1% zirconium metal member. The assembly was brazed at 1910°F (no holding time) in a vacuum of better than 4×10^{-5} torr. The braze alloy moved through the joint forming a satisfactory fillet on the inside of the metalized ceramic. A similar sample had a tensile strength in excess of 10,000 psi.

Other alkali metal resistant metalizing compositions may show similar usefulness as a wetting aid. This technique can promote hermeticity in the long braze joints encountered in large bore seals.

FIGURE II-30. Section of a CLM 15 Test Sample With 75Zr-19Cb-6Be Active Metal Braze Between Metalized AD 99 Ceramic and Cb-1Zr (2X).
The lower section was evaporation metalized with 1 μ Ti and $>$ 1 μ Mo. Superior wetting of this sample is indicated by the smooth inside fillets of braze metal not present on the upper unmetalized ceramic joint.

SECTION III

MATERIALS, PREPARATION AND TEST PROCEDURES

A. MATERIALS

Materials specifications and analysis are presented for those metals and ceramics which may be fabricated into bore seal configurations and which indicated compatibility with alkali metal environments. Nominal properties of these materials are reported in Section IV.

Braze materials and alkali metal specifications are listed separately. The following summary lists material description, form and source.

Material and Form	Source	Product Specification
	Metals & Ceramics	
Beryllia, 99.8% bar, rod, plate, tubes; other shapes made to order	Brush Beryllium Co.	Beryllia ceramic marketed under trade name Thermalox 998. Table III-1
Alumina, 99.75% bar, rod, plate, tubes	General Electric Co.	Alumina ceramic marketed under trade name Lucalox. Table III-2
Columbium D-43 sheet, plate, bar, tubing	DuPont	DPC (P) 1101 6/28/63 Table III-3
Tantalum T-111 sheet, plate, bar, tubing	Westinghouse Electric Corp., Astronuclear Laboratories	No commercial specification. Table III-4

Columbium-1% Zirconium sheet plate, bar, tubing	Wah Chang Co.	No commercial specification. Table III-5
	Braze Alloys	
Alloy 56Zr-28V-16Ti Cast alloy or powder	Battelle Memorial Institute	Available on developmental basis
Alloy 75Zr-19Cb-6Be Cast alloy or powder	Battelle Memorial Institute	Available on developmental basis
Alloy 48Zr-48Ti-4Be Cast alloy or powder	Battelle Memorial Institute	Available on developmental basis
	Alkali Metals	
Potassium	Mine Safety Appliance Research Corp.	High-purity potassium, hot-trapped and supplied in special hot-trap container. Table III-6
NaK	Mine Safety Appliance Research Corp.	High-purity sodium potassium eutectic with 78% K, plus zero minus one weight percent; 22% Na. Hot-trapped and supplied in special hot-trap container. Table III-6
Lithium	Foote Mineral Co.	High-purity reactor grade lithium packed under argon in sealed cans. Table III-7

TABLE III-1. Nominal and Supplied Material Analyses - Beryllia Body, 99.8% BeO

Oxide Constituent	Composition (weight percent)	
	Nominal	Supplied [a]
BeO	99.8 (by difference)	PC
CaO	0.01	0.008
MgO	0.15	0.13
SiO_2	0.01	0.008
Al_2O_3	0.015	0.015
Fe_2O_3	0.0100	0.006

(a) Spectrographic analysis by Brush Beryllium Co.

PC - Primary Constituent

1. Forming by isostatic press.
2. Firing sequence - proprietary
3. Grain size approximately 40 μ average.
4. Density 2.907.

TABLE III-2. Nominal and Supplied Material Analyses - Alumina Body, 99.8% Al_2O_3

Oxide Constituent	Composition (weight percent)	
	Nominal	Supplied[a]
Al_2O_3	99.75	PC
MgO	0.25	0.10
CaO	< 0.05	0.002
SiO_2	< 0.05	0.025

(a) Spectrographic analysis by Eitel-McCullough, Inc.

PC - Primary Constituent

1. Forming and firing sequence - proprietary
2. Grain size approximately 25 μ average
3. Density 3.98

TABLE III-3. Nominal and Supplied Material Analyses - Columbium Alloy D-43

Element	Composition (weight percent)			
	Nominal		Supplied[c]	
	Minimum	Maximum	(a)	(b)
W	9.0	11.0	9.4	9.9
Zr	0.75	1.25	0.92	1.1
O	-	0.010	0.0124	0.0148
H	-	0.010	0.0002	0.0001
N	-	0.0075	0.0040	0.0034
C	0.08	0.12	0.075	0.11
Cb	remainder			

(a) Sheet 0.010 thick used in ceramic-to-metal test assemblies.
(b) Sheet 0.040 thick used in thermophysical tests.
(c) Analyses supplied by DuPont Metal Products.

TABLE III-4. Nominal and Supplied Material Analyses - Tantalum T-111

Element	Composition (weight percent)		
	Nominal		
	Minimum	Maximum	Supplied[a]
W	7.0	9.0	7.9
Hf	1.8	2.4	2.32
O	-	0.010	0.0015
N	-	0.005	< 0.0010
C	-	0.005	0.0026
Ta	remainder		

(a) Heat DX 570 from which bars for thermophysical tests and sheet specimen were made. Material and analysis from Westinghouse Astronuclear Laboratories.

TABLE III-5. Nominal and Supplied Material Analyses - Columbium-1% Zirconium

Element	Composition (weight percent)						
	Nominal		Supplied[a] (Ingot Analysis)				
	Finished Sheet	Starting Ingot	Heat 912-1211	Heat 98-7056	Heat 8-3856	Heat 355-70303	Heat 88-1687
Cb	98.5 min.	98.5 min.	98.85	98.9	98.65	98.85	98.9
Zr	0.8-1.2	0.8-1.2	1.04	0.87	1.10	0.98	0.89
O	0.030 max.	0.030 max.	0.0145	0.0200	0.0200	0.0260	0.0210
H	0.0015 max.	0.0015 max.	0.0005	0.0003	0.0004	0.0004	0.0004
N	0.030 max.	0.030 max.	0.0065	0.0055	0.0061	0.0055	0.0050
C	0.010 max.	0.010 max.	<0.0030	0.0065	0.0052	0.0043	0.0055

Analyses of material supplied by Wah Chang Corp.

(a) Average results from top and bottom of ingot.

TABLE III-6. Typical and Supplied Analyses of Potassium and Sodium-Potassium Eutectic[a]

Material	Ti	Fe	B	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Na	Ni	Mo	V	Ca	Ba	Sr	Be	Zr	Ag	N	C	O
Potassium Typical ppm	< 5	50	<10	< 5	10	10	5	< 5	30	<5	<5	<25	<50	< 25	<3	2	10	<1	<1	1		<1	ND	<50	<50
Potassium Supplied ppm	< 5	5	<5	< 5	<1	3	<1	< 1	<1	<1	<1	10	3	< 1	<5	<1	4	<3	<1	<1	<10	1	<10	49	<10
NaK Typical ppm	<10	10	10	<10	2	20	5	<10	5	<5	<10	<25	PC	<10	<5	<10	<50	<3	<1	<5	<20	<5	ND	<50	<50
NaK Supplied ppm	< 5	< 5	<5	< 5	<1	<5	<1	< 5	<2	<5	<5	10	PC	< 5	<5	<5	<10	<2	<1	<1	<10	<1	ND	50	<10

All analyses by emission spectrograph except carbon, nitrogen, and oxygen which were done by mercury amalgamation, followed by appropriate method - acid titration, Kjeldahl, etc. Most of the typical values given are the sensitivity limits for the element.

ND - Not determined
PC - Primary Constituent

(a) Data by Mine Safety Appliance Research Corp.

TABLE III-7. Typical Analysis of Lithium

Element	Composition (ppm) Typical	Supplied
Li	99.8% (by difference)	
Na	30	
K	45	
Cl	41	
O	200	(a)
N	31	(a)
Ca	<10	
Fe	<10	
Ni	<10	
Cr	<10	
Al	<10	
Si	<10	
Co	<10	
Cu	<10	

(a) Analyses of lithium purity test capsules resulted in very high nitrogen content by Kjeldahl method and oxygen by neutron activation. Although capsules were mass spectrometer leak-tight before loading, post-analysis examination revealed a leak in the crimped area of the purity test capsule tubulation.

Typical Composition supplied by the Foote Mineral Co.

B. SPECIMEN CONFIGURATIONS.

A series of drawings and sketches shown in Figures III-1 through III-5 depict typical examples of thermophysical, mechanical, and ceramic-to-metal braze specimens. A number of those in the latter category are non-standard and are discussed later in this section.

C. TEST PROCEDURES.

A tabulation of materials, type of test, and test method or specification is found in Table III-8. A number of tests required in the bore seal materials evaluation were non-standard in nature. These tests and procedures are discussed here.

1. Thermophysical Properties

a. SPECIFIC HEAT

Precise measurement of specific heat was made in a drop-water calorimeter according to a method described by J. Valentich of the Westinghouse Research Laboratories(1). The only specimen requirement for measurement of specific heat is that a compact mass of approximately 30 grams be available for test.

Oxidation was prevented by sealing the specific-heat specimens in evacuated (5×10^{-5} torr) quartz capsules. During normal testing, the specimen temperature was measured with a platinum-rhodium thermocouple mounted in a quartz well inserted halfway down the center of the specimen. Since quartz is not as good a heat conductor as the metal, it was necessary to determine the difference in temperature between the quartz well and the specimens. To do this, a 1/16 inch diameter hole was drilled in a standard copper specimen to within 3/16 inch of the quartz well, and thermocouples were positioned in both the specimen and the quartz

(1) Valentich, J. - "Equipment and Methods for the Continuous Measurement of Heat Content of Metals to 1100°F". Westinghouse Materials Engineering Report No. 5973-3031, Westinghouse Electric Corporation, East Pittsburgh, Pa., 19 November 1959.

TABLE III-8. Test Procedures

Material	Type of Test	Figure[(a)] Number	Test Method
Ceramic (All)	Flexural Strength (Modulus of Rupture)	III-7a, b	ASTM D 116-63
Ceramic-to-Metal Braze Specimens	Flexural Strength (Modulus of Rupture)	III-7c	ASTM D 116-63
	Tensile Strength	III-6	ASTM F19-61T
	Peel Strength	III-10	Drum Peel Test
	Peel Strength	III-7d	Tab Peel Test
	Hermeticity	III-7e	Helium Mass Spectrometer
Metal Member			
Columbium D-43	Electrical Resistivity	III-2	ASTM B-70 Kelvin Bridge
	Poisson's Ratio	III-5	ASTM E 132
Tantalum T-111	Thermal Conductivity	III-4	Comparison Bar
	Specific Heat	III-1	Drop-Water Calorimeter
Braze Alloy			
48Ti-48Zr-4Be	Thermal Expansion	III-3	ASTM B-95

(a) All dimensions given in inches.

well. Temperatures were recorded at both locations as the specimen was taken through a complete test. Test results show that the temperatures in the quartz well and in the specimen were within one percent except at 1500°F where they differ by about 1.5 percent. This means that the thermocouple in the quartz well measures the specimen temperature with good accuracy over the entire testing temperature range.

b. ELECTRICAL RESISTIVITY

The standard Kelvin Bridge method of ASTM B70 was used for all measurements of electrical resistivity. Strip materials were wound on a 5/8 inch diameter quartz mandrel.

A Leeds and Northrup Kelvin Bridge was used to measure the resistance. Short pieces of alumel wire were used in the furnace hot zone and silver wire in the room temperature zone as lead wires. Resistance welding was used to fix the alumel leads to the specimens. The tests were conducted in a vacuum of 5×10^{-5} torr and the average temperature variation over the two inch coil length was less than ± 1 percent. All the samples were heated at a rate of 10°F/min and the resistance of each specimen was measured at 100°F increments with increasing and decreasing temperatures. Preliminary tests on a sample of TD nickel wire showed that the resistance measured at this heating rate duplicated the results obtained by soaking at each temperature increment for twenty minutes. For this reason, all specimens were tested at a constant heating rate of 10°F per minute. In all tests, the integrity of the elevated temperature leads was checked at room temperature by comparing the resistance measured with the special high-temperature leads and the resistance measured using the standard room temperature clamps.

The Kelvin Bridge used to measure the resistivity of the specimens has a resolution of 10^{-8} ohms. Resistivity was computed and reported in ohm-cm.

c. THERMAL EXPANSION

The thermal expansion measurements were made in a quartz-tube dilatometer in which the specimen is heated with a resistance wound furnace. The furnace and tube are orientated in a horizontal position. The furnace is stationary while the

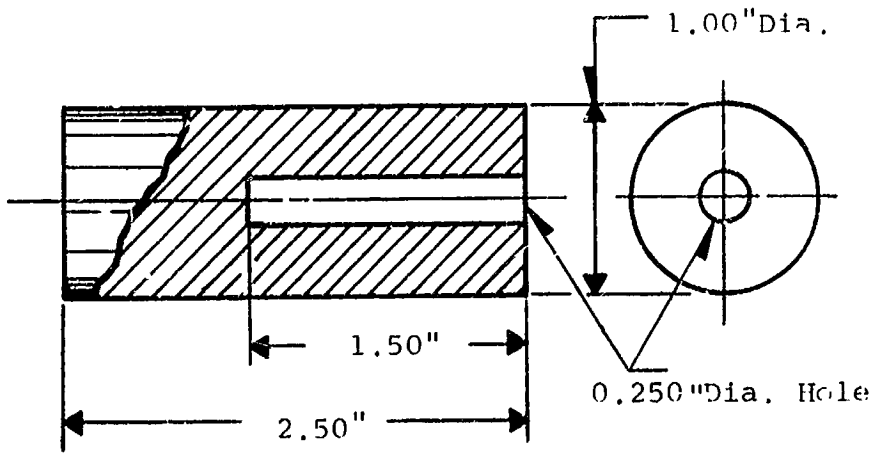
tube and associated measuring apparatus can be moved in and out of the furnace on a rail. The quartz tube is slotted at the closed end so that a two inch long specimen can be placed in it with one end contacting the bottom. A quartz rod, attached to a Statham linear-displacement transducer, is in contact with the other end of the specimen. As the specimen expands, the quartz rod moves, and the transducer measures the amount of the movement. The transducer is an unbonded, Wheatstone bridge circuit whose sensitivity can be varied by regulating the voltage input. Length changes as small as one micro-inch can be measured. The output of the transducer is recorded on one axis of a Moseley recorder; the output of a chromel-alumel thermocouple wired to the specimen is recorded on the other axis of the recorder. The resultant curve is then corrected for the expansion of quartz. The temperature rise of the specimen is pre-programmed at 3°C per minute using a Leeds and Northrup program controller. Argon gas is continuously flooded over the specimen to limit oxidation at the higher temperatures.

d. THERMAL CONDUCTIVITY

This property was only measured on tantalum alloy T-111 using the comparison bar technique. In this method the specimen, 1/2 inch in diameter by 4-1/2 inches long, is fixed to a heater block through a snug tapered fit. The other end of the specimen is fixed through a threaded connection to a comparison bar of nickel, 1/2 inch in diameter by four inches long, whose thermal conductivity is known. A heat sink, cooled by circulating water, is fixed to the free end of the nickel rod. The nickel and specimen rod assembly is held in a vertical position with the heater at the bottom. The rod system is surrounded with a high purity alumina insulation which is enclosed with a 2-1/2 inch diameter shield. The shield is made from 302 stainless steel and nickel. The stainless steel portion is as long as the specimen, and the nickel portion is as long as the comparison bar. The nickel and stainless steel sections are buttwelded and the joints located as to be in line with the specimen-nickel joint. A heater is fixed around the shield circumference at this joint. Three chromel-alumel thermocouples are fixed to the specimen, the first is 1/2 inch down from the nickel joint and the remaining two at one inch intervals below the first. Four thermocouples are fixed to the

comparison bar, the first is 1/2 inch above the specimen joint and the other three at one inch intervals above the first. Seven thermocouples are similarly placed on the shield at the same heights as those on the bars. The entire assembly is set on high purity alumina insulation which is on a steel base plate and surrounded with a five inch I.D. Transite tube. The area between the shield and the Transite tube is filled with the alumina insulation. A bell jar is placed around the Transite tube and the system is baked out and evacuated to 5×10^{-5} torr.

As the heater temperature rises, the specimen temperature rises, and heat flows up the specimen through the joint and to the water sink at the end of the nickel bar. Unidirectional heat flow up the specimen is obtained by adjusting the heaters on the shield and the heater block, and by adjusting the water flow. The thermocouples on the bar and shield at the same height are maintained at equal temperatures to prevent radial heat flow. After these conditions have been established for about four hours at a test temperature, all thermocouples on the comparison bar and the specimen are read and recorded. The thermal conductivity of the specimen is then computed.



Westinghouse Dwg. Ref. No. 627A075

FIGURE III-1. Specific Heat Specimen

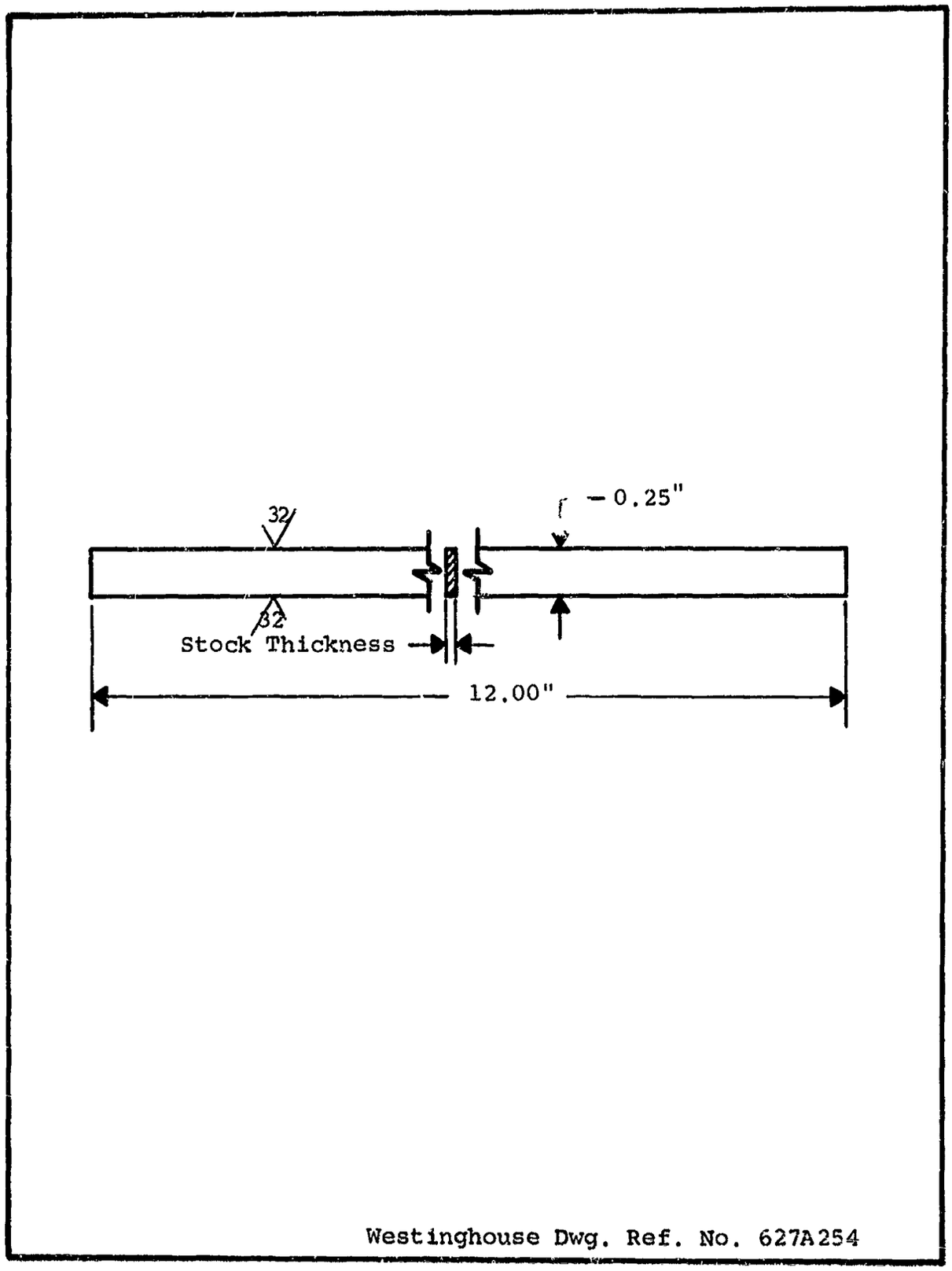
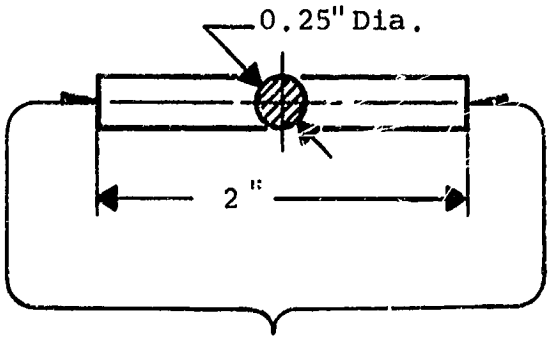


FIGURE III-2. Electrical Resistivity Specimen



Westinghouse Dwg. Ref. No. 627A072

FIGURE III-3. Thermal Expansion Specimen

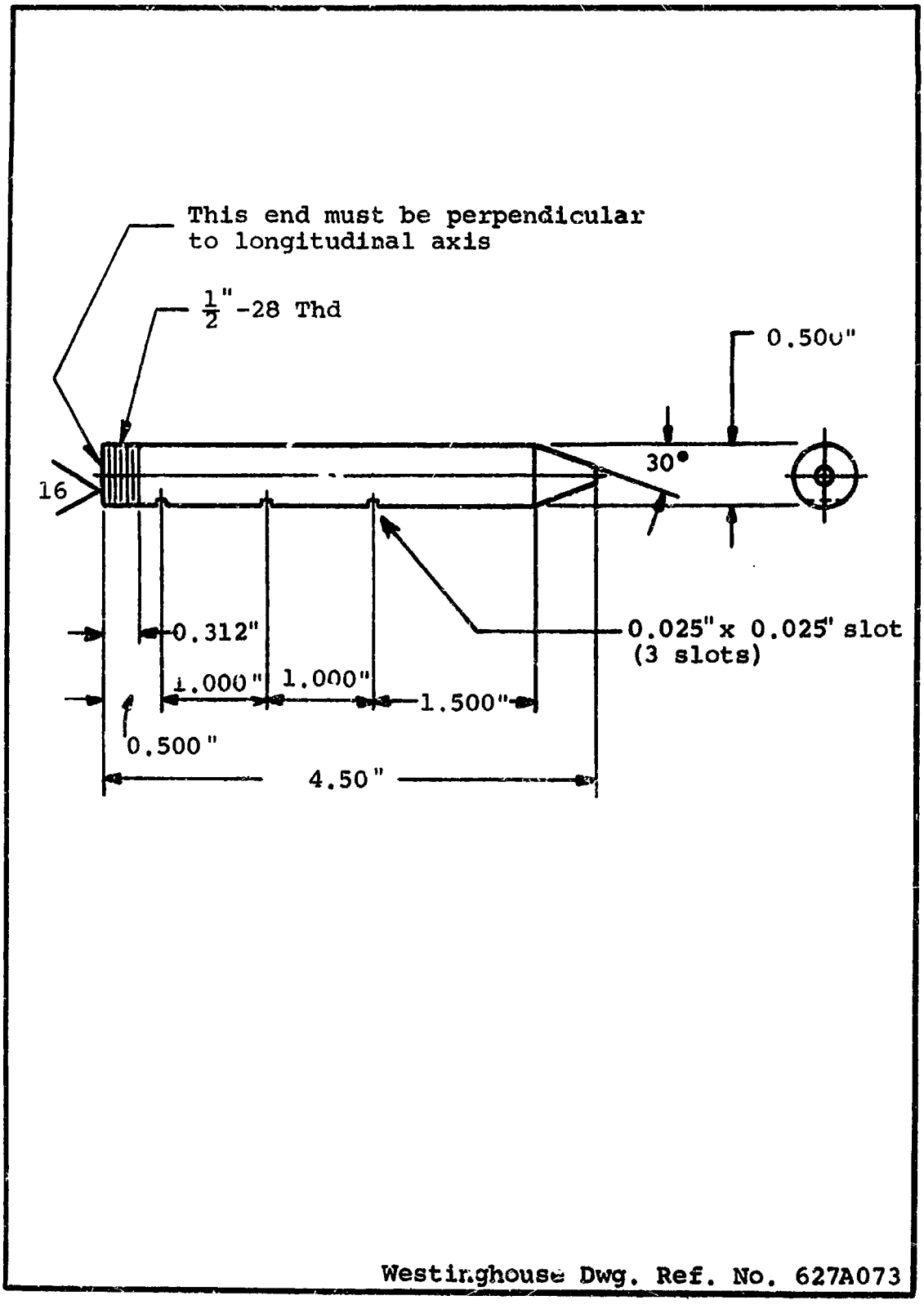


FIGURE III-4. Thermal Conductivity Specimen

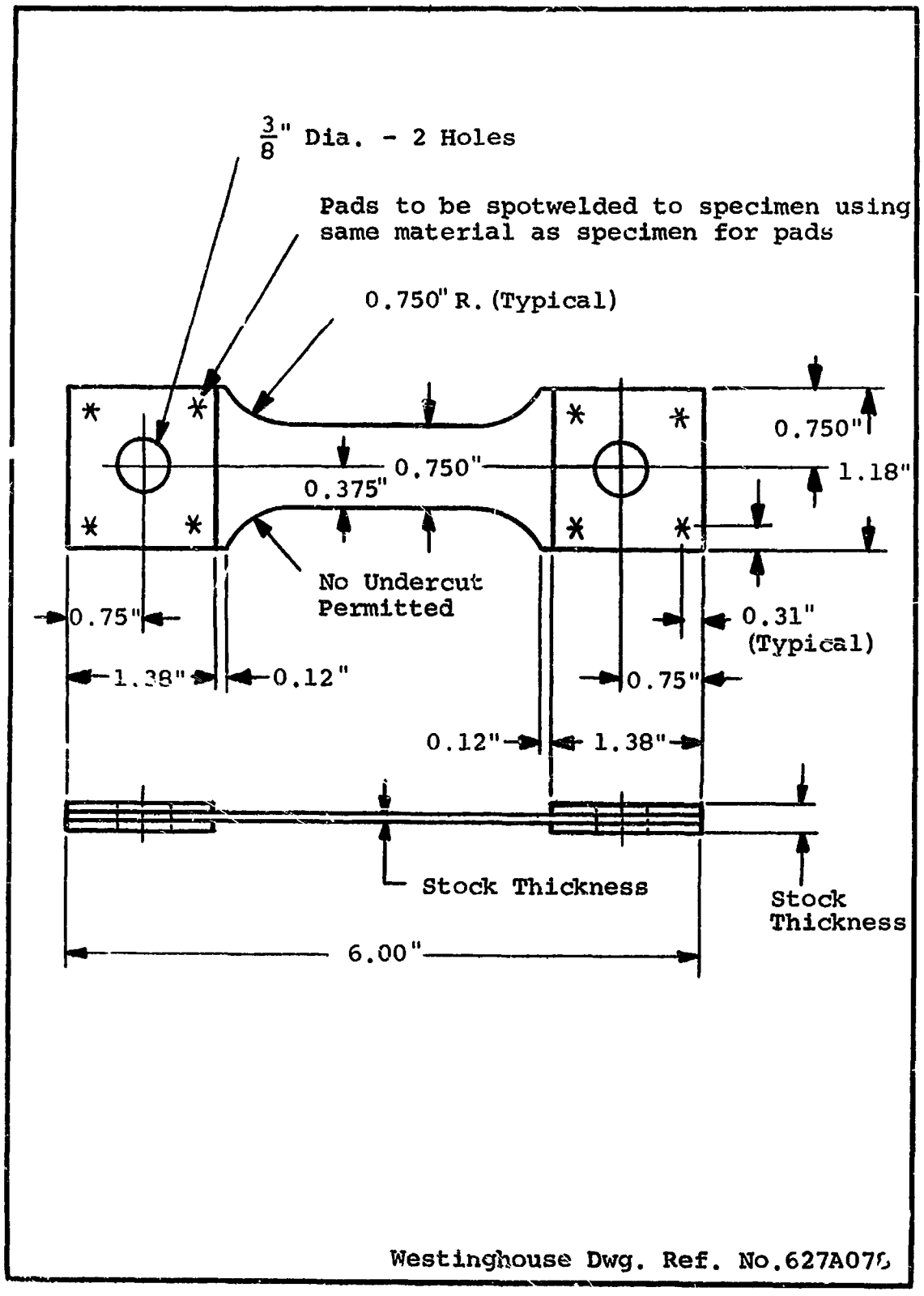


FIGURE III-5. Poisson's Ratio Specimen

2. Mechanical Test Procedures for Ceramic-Metal Assemblies

The ceramics and ceramic-metal seals were evaluated mechanically and metallurgically by means of six basic geometries.

a) ASTM CLM 15 Tensile test piece (Figure III-6).
b) Modulus-of-rupture bar (0.1 inch x 0.1 inch x 1.0 inch)(Figure III-7a).
c) Modulus-of-rupture rod (0.1 inch diameter x 1.0 inch long)(Figure III-7b).
d) Modulus-of-rupture assembly (Figure III-7c).
e) Tab peel assembly (Figure III-7d).
f) Vacuum test assembly (Figure III-7e).

The modulus-of-rupture bars and assemblies were used in alkali metal compatibility tests (Figures III-7a, b, c). This test configuration permitted quantitative evaluation of the ceramic as well as the ceramic-to-metal seal before and after alkali metal exposure. Such quantitative data had not been obtained on the ceramic portion of a seal in previous alkali metal corrosion studies because of the limitations of the standard ASTM CLM 15 (Figure III-6) geometry normally used.

However, the latter specimen does provide a larger braze area for evaluating brazing techniques. Preliminary metalizing and active braze alloy screening were tested by the method described in ASTM F19-61T "Tension and Vacuum Testing Metalized Ceramic Seals".

An Instron Universal test machine was used for mechanical testing. Test results were recorded on a strip chart. Modulus bars or assemblies were tested using the four point fixture shown in Figure III-8. The inner and outer load points are 0.25 and 0.80 inches apart respectively. Formulas for computing modulus-of-rupture (flexural strength) are shown in Table III-9. A head speed of 0.1 inch per minute was used in performing the modulus-of-rupture tests. The pull rate for the drum peel test and tab peel test was 1.0 inch per minute. Other brazing and test fixtures for the test pieces are shown in Figures III-9 and III-10.

Results of a test series to determine correlation between the non-standard specimens and the CLM 15 tensile test and drum peel geometries are summarized below. The effect of processing variables on mechanical properties are also reported.

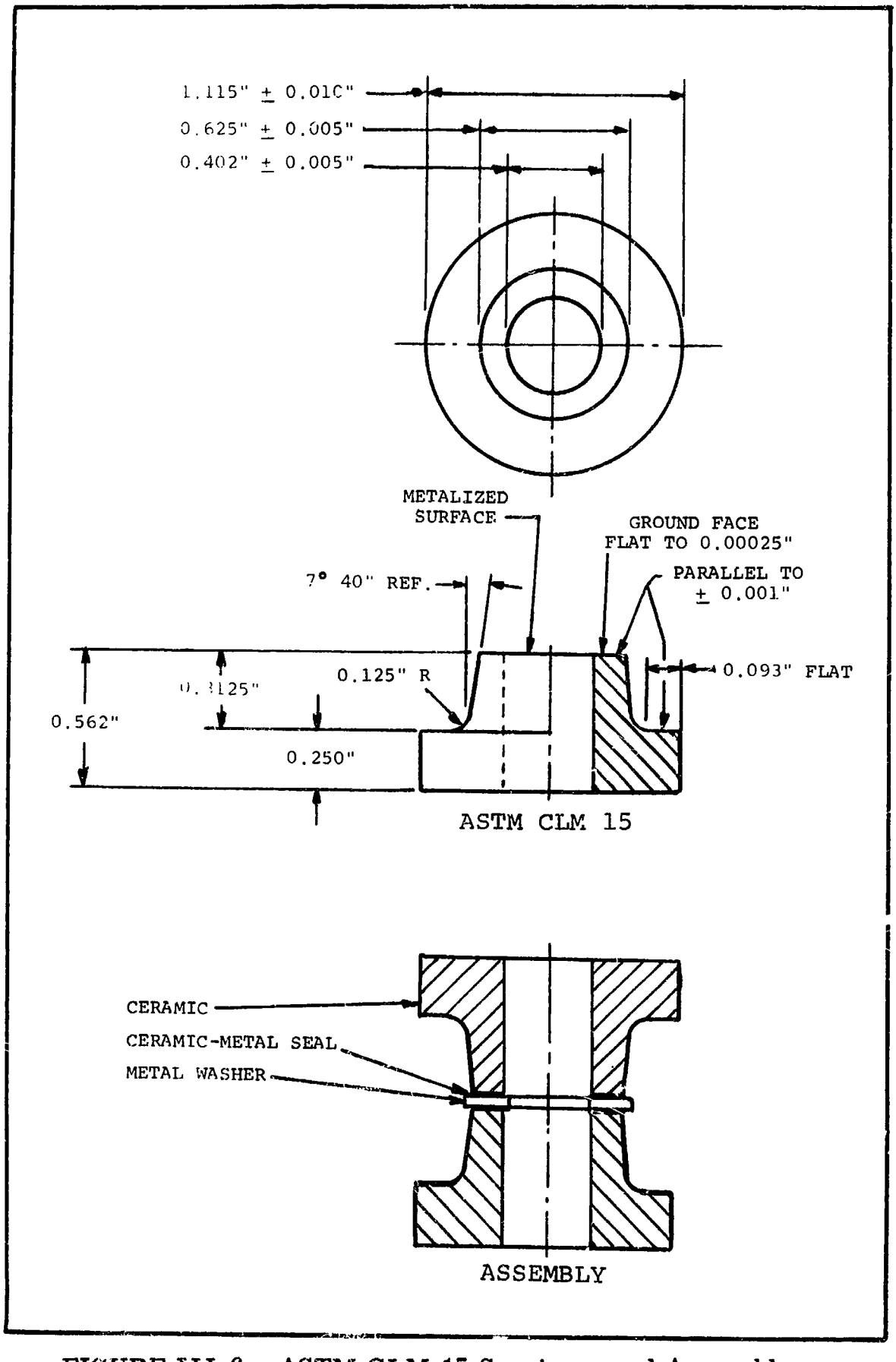


FIGURE III-6. ASTM CLM 15 Specimen and Assembly

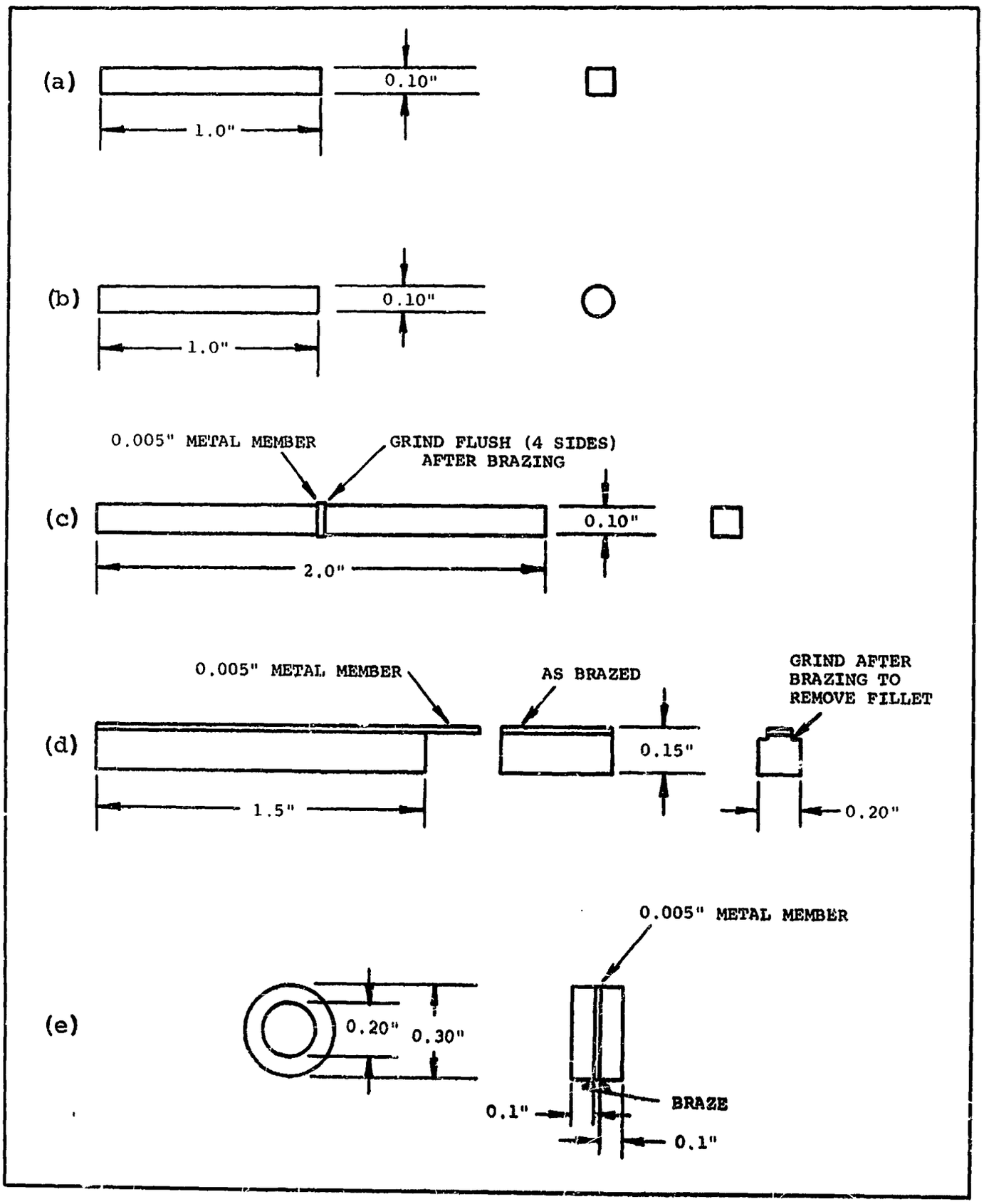


FIGURE III-7. Test Assembly Geometries

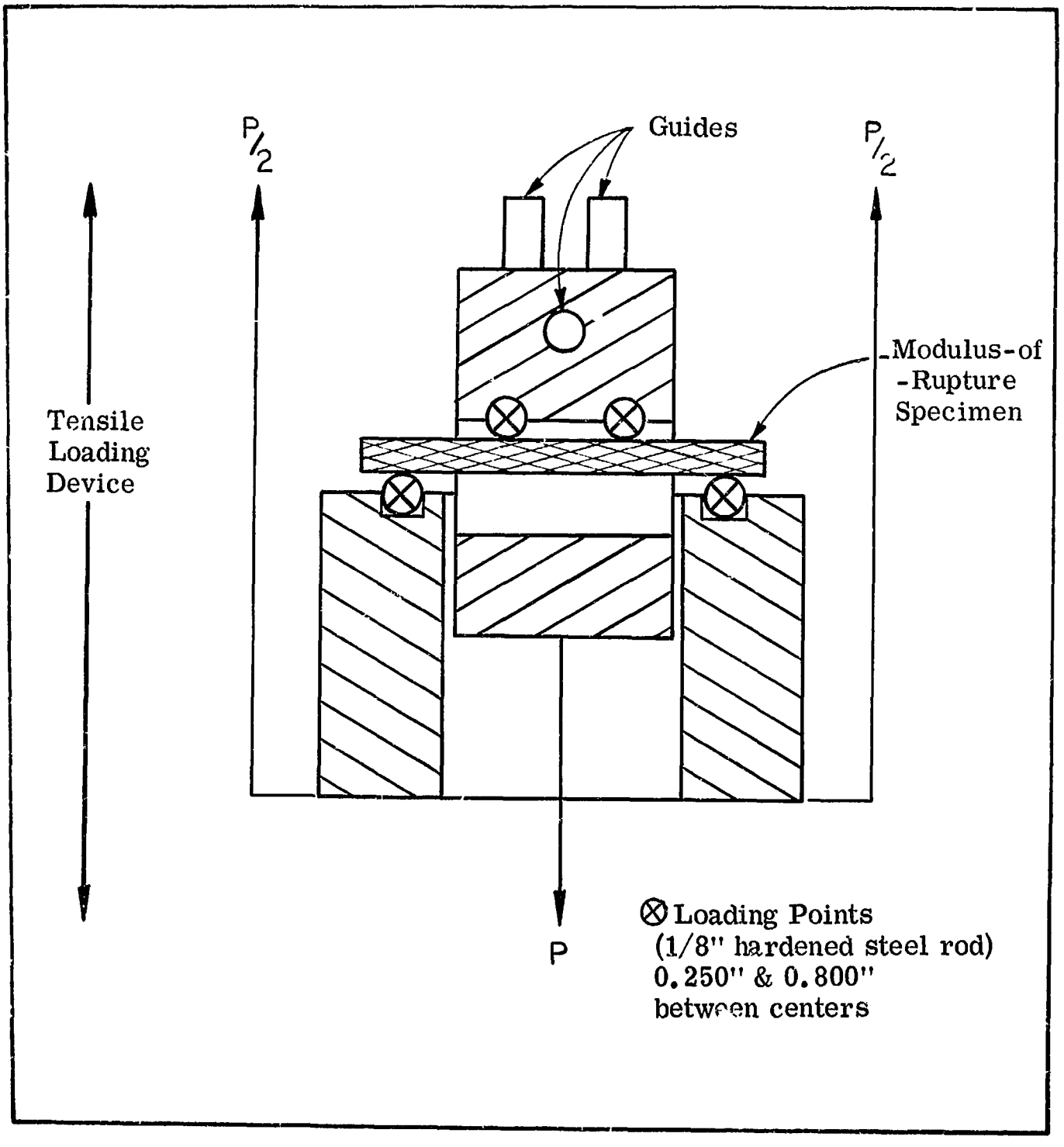
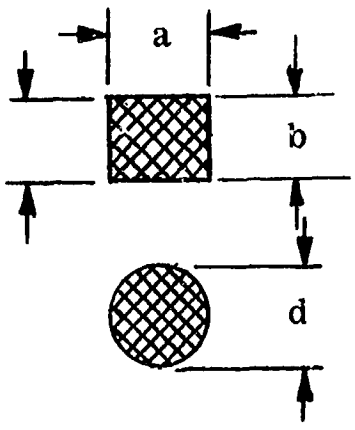
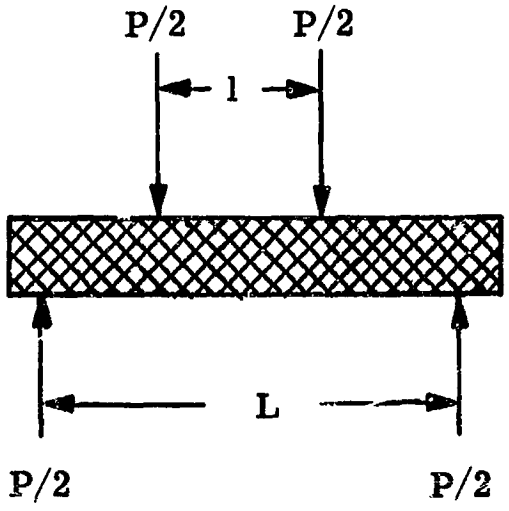


FIGURE III-8. Four Point Loading Fixture

TABLE III-9. Formulas for Computing Modulus-of-Rupture (Flexural Strength)

Cross Section	Formula
	M of R $= \frac{3(L-1)P}{2ab^2}$ M of R $= \frac{8(L-1)P}{\pi d^3}$

Symbol	Units
M of R	psi
a, b, d	inches
P	pounds
L, l	inches



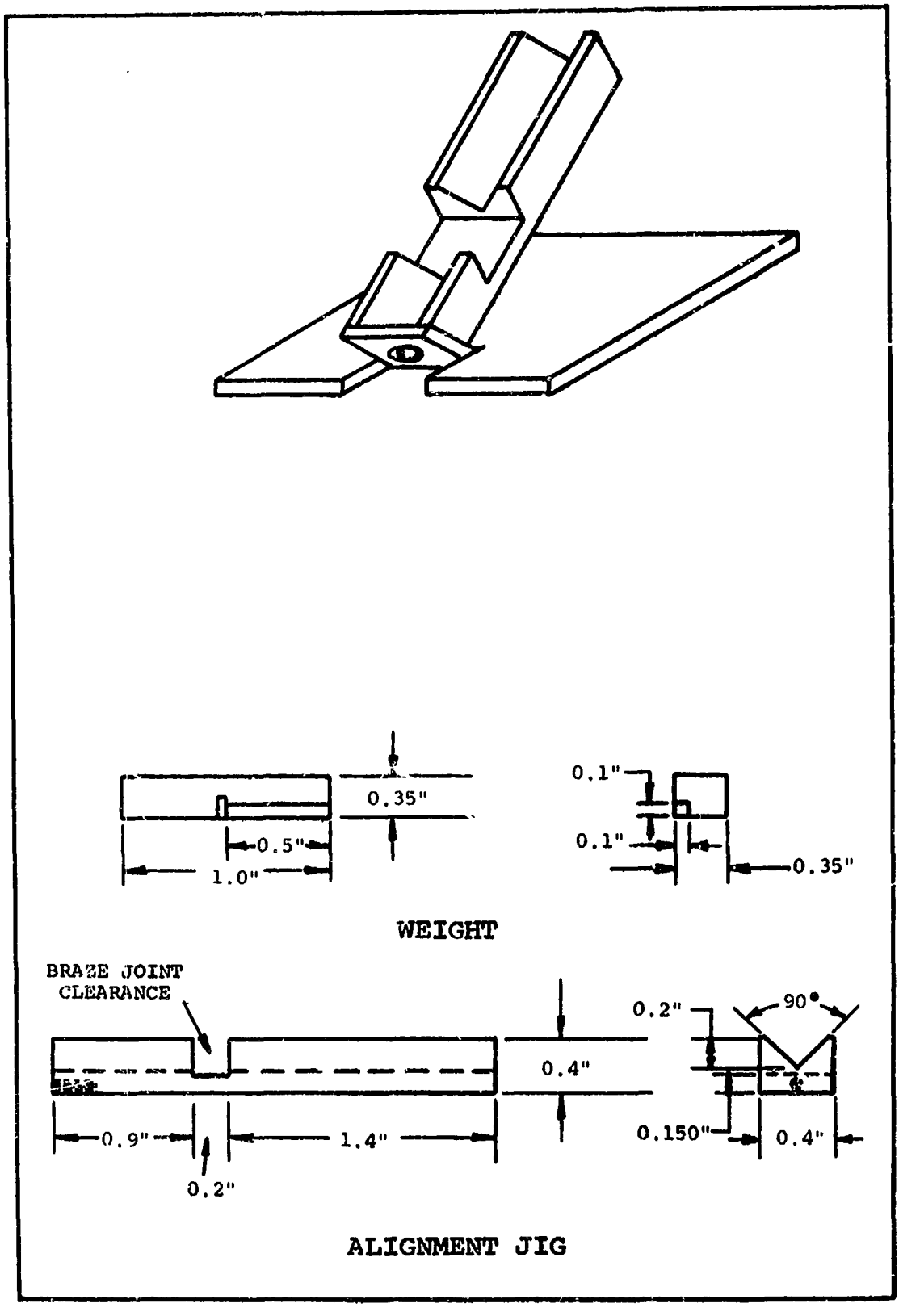


FIGURE III-9. Modulus-of-Rupture Brazing Fixture

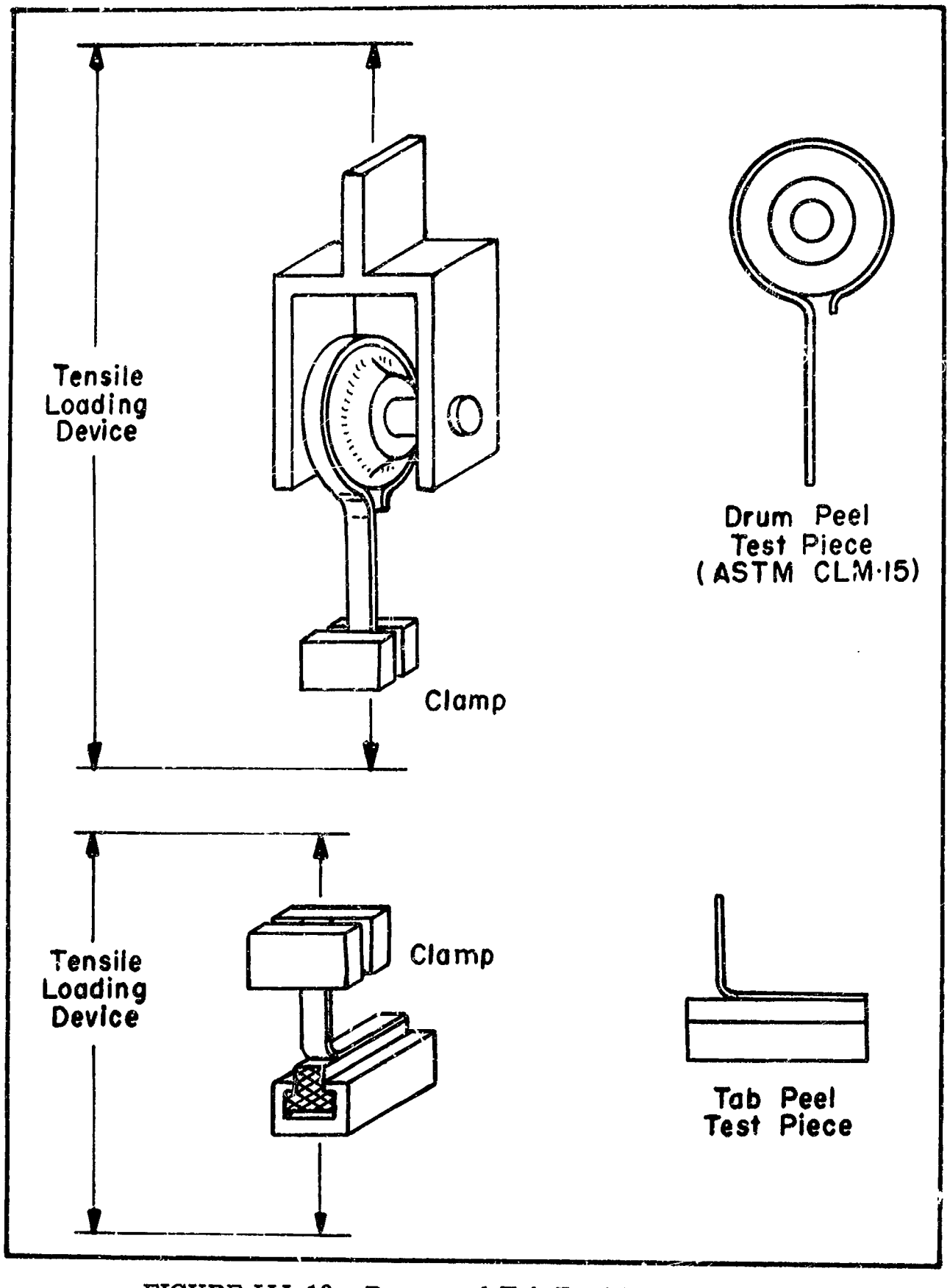


FIGURE III-10. Drum and Tab Peel Test Fixtures

a. MODULUS-OF-RUPTURE AND CLM 15 TENSILE TEST OF CERAMIC-TO-METAL SEALS

Two alumina ceramics and two braze alloys were used in preparing specimens. Each ceramic and braze combination was compared for the corresponding M-of-R and tensile strength groups. In all four cases, the M-of-R groups had significantly higher mean strength values than the tensile strength groups. The ratio of the means of the CLM 15 seal tensile strength to the modulus-of-rupture for the brazed alumina bodies was 0.31 for pure copper braze alloy and 0.21 for the nickel alloy (Coast Alloy 52).

b. TAB PEEL AND DRUM PEEL TESTS

The correlation between the drum peel test (CLM 15 periphery) and the tab peel test was made using the same braze systems as cited in part (a) above. Results of a comparison between drum peel and tab peel test for ceramic to metal seals indicate no significant differences between the tests. Both tests perform their function nearly identically.

c. EFFECT OF POST-BRAZING SAMPLE PREPARATION ON MODULUS-OF-RUPTURE

Modulus-of-rupture assemblies were brazed using alumina ceramics as in part (a) above. The brazed specimens were divided into four groups and treated as follows: (1) minimum preparation, excess metal member trimmed sufficiently to permit location in the test fixture; (2) excess metal member trimmed, sample hand ground to remove braze fillets; (3) metal member trimmed, hand ground to remove braze fillets, and surface ground on support and bearing sides of the sample; (4) maximum preparation, all sides surface ground flat and parallel.

An analysis of variance performed on the modulus-of-rupture data indicate no significant difference. Sample preparation may be minimized.

3. Leak Test Procedure for Ceramic-Metal Assemblies

a. CALIBRATION.

A certified leak obtained from the ALO Standards laboratory, Sandia Base, Albuquerque, New Mexico, is utilized to calibrate SC4 leaks (VEECO) as secondary standards. The secondary standard is then used to check the sensitivity of leak detectors daily. Records are logged daily. The specific calibration routine varies somewhat with the type and model of leak detector. Normal sensitivities range from 1 to 3 x 10^{-10} standard cc/sec and remain quite stable over a period of several weeks.

b. PROCEDURE.

1) Use a leak detector which is checked daily for sensitivity. Sensitivity must be better than 5 x 10^{-10} standard cc/sec, as determined with the certified leak.

2) Place sample on manifold connection and evacuate.

3) Place bag or inverted jar to completely cover sample and manifold connection.

4) Fill bag or jar with helium to purge out all air; continue helium flow through next step.

5) Leak detector output meter must indicate no leak on most sensitive scale.

D. ALKALI METAL LOADING AND EXPOSURE PROCEDURE

1. Capsule Loading Facility

The columbium-1% zirconium capsule fabrication and the loading of these capsules with test assemblies and with potassium and NaK was carried out in a vacuum purge dry box with appropriate accessories and modifications. The system is shown schematically in Figure III-11. Photographs of the system are shown in Figures III-12 and III-13.

The dry box is a General Technology Model Mark 5A modified to accommodate the special accessories for loading. A modified evacuation system consists of a 15 cfm Welch forepump Model No. 1397B and a 400 liters/sec oil diffusion pump with a Freon 12 cooled chevron baffle to limit backstreaming. Associated vacuum manifolding permits pumping out the entry vestibule and glove ports. The same pumping system is used to evacuate the loaded capsules prior to sealing. An ionization gage and power supply to monitor vacuum are included.

The argon atmosphere is supplied from a liquid argon reservoir and boiler capable of generating 50 cfm of gas. The argon is passed through a U.S. Dynamics Model HXV-20 Automatic Gas Purification System capable of purifying 50 cfm of the argon to less than one ppm of H_2O or O_2. Short connectors between the purification system and the dry box are made of stainless steel with SwageLok fittings.

Effluent argon from the dry box is monitored for H_2O and O_2 with a Beckman Hygromite and Model 80 Oxygen trace analyzer respectively. A dual track recorder provides a permanent record of dry box conditions during capsule loading.

The loading procedure carried out in this facility by the sequence outlined in the following section has resulted in pumped down vacuums of less than 1×10^{-5} torr and an argon working atmospheres of less than 5 ppm O_2 and H_2O combined.

Hot trapping of alkali metal is started about 30 hours before loading in order to allow 24 hours at 1400°F and four hours to cool from 1400°F to 300°F. During this time the potassium transfer line is evacuated and heated to approximately 300°F, and the dry box is evacuated to a vacuum of better than 1×10^{-5} torr. The capsules, test pieces and all items which will contact the alkali

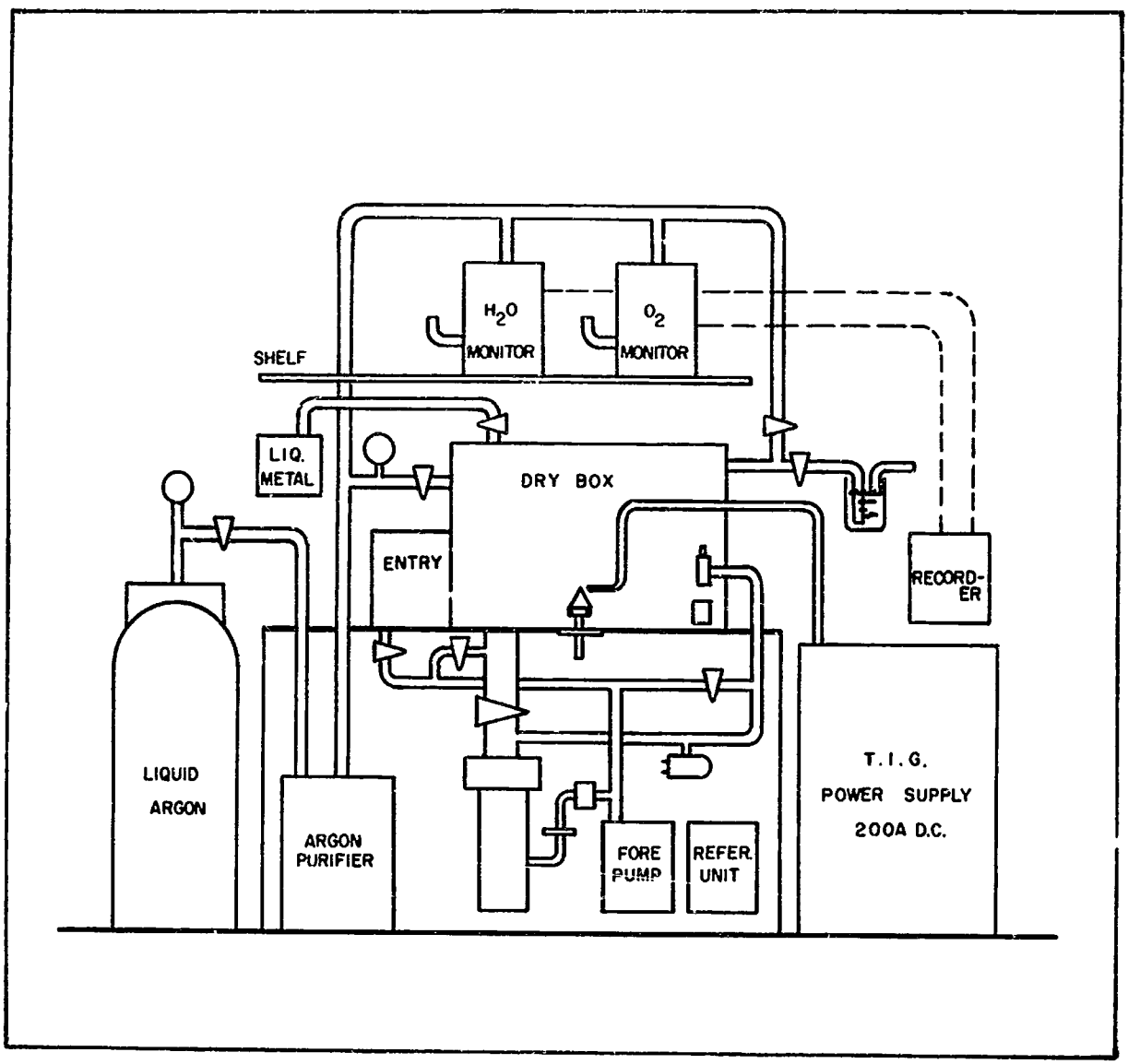


FIGURE III-11. Schematic of Capsule Welding and Loading Facility

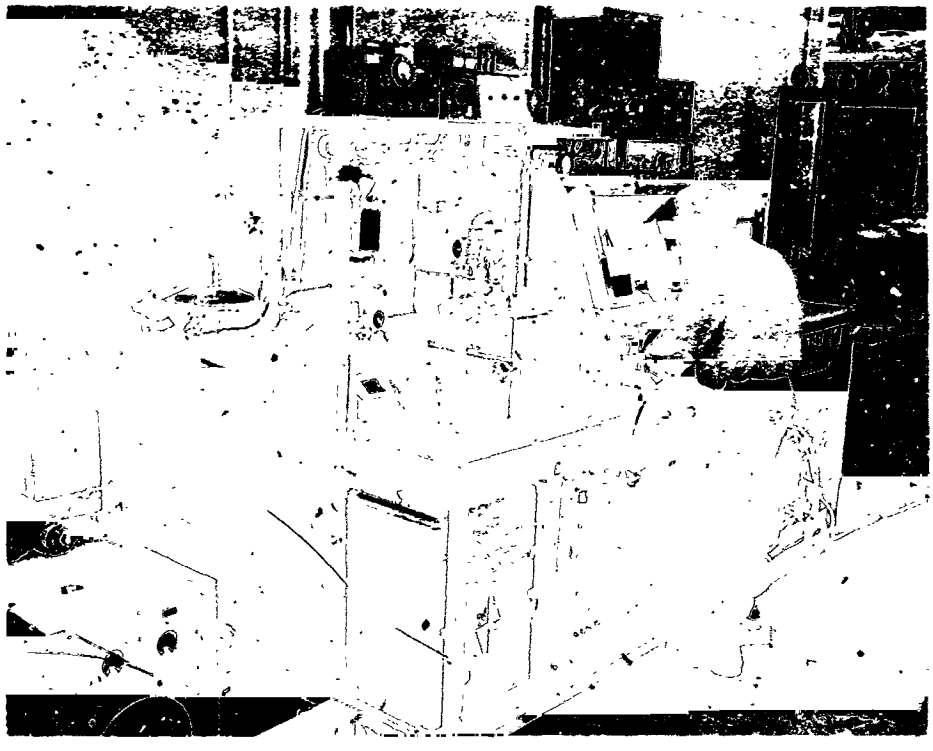


FIGURE III-12. Dry Box Facility

FIGURE III-13. Right End of Dry Box Showing Anti-Diffusion Traps

metal are vacuum fired to 1400°F (Vacuum less than 5×10^{-6} torr) and allowed to cool overnight. The chamber is back-filled with helium before parts are transferred to the dry box. Immediately prior to loading, the dry box is pumped down and back-filled several times with purified argon. The vacuum-fired parts are passed into the box through the vacuum purge entry vestibule. About 100 cc of potassium (or NaK) is used to clean the transfer line between the hot-trap container and the dry box prior to capsule loading. This potassium, which is stored in an open beaker during the capsule loading, may be used as an empirical test of the dry box atmosphere.

The oxygen and moisture monitors indicated average of five to ten ppm of the contaminants combined during one loading operation(a). Potassium in the beaker remained bright and clean for thirty minutes. A blue film covered the surface after 20 hours. The argon in the dry box was nearly stagnant in spite of a 25 cfh flow rate. In a subsequent loading, the moisture and oxygen were monitored at a total of five ppm in the dry box effluent gas(b). The argon flow rate during this loading was 15 cfh. Potassium in an open beaker remained bright and clean for twenty hours.

Thus, even though capsules are open to the dry box environment for 10 to 20 minutes before sealing, the alkali metal in the bottom of a capsule is not subjected to continuous exposure to contaminants because of the relatively stagnant environment.

Other dry box facility details are given at appropriate points in the loading procedure.

2. Capsule Loading Sequence

   a. The dry box is pumped down and back-filled with purified argon several times. Oxygen and moisture levels in the effluent argon must be below 10 ppm, combined.

(a) Subsequent analysis of potassium in test capsules indicated 21 and 26 ppm oxygen content.

(b) Subsequent analysis of alkali metal in three test capsules indicated less than 10 ppm oxygen.

b. All capsule tops, capsule bottoms, and test pieces (which had been vacuum outgassed at 1400°F, cooled in vacuum and helium back-filled) are loaded into the dry box just prior to the K loading sequence.

c. In the dry box, a capsule bottom, with test pieces removed is placed into the rotary turntable welding jig and heated by drawing a low energy arc with the TIG welding torch.

d. One cc of potassium is forced from the extruder (Figure III-14) and cut off with a clean stainless steel blade; then transferred into the capsule with clean, stainless-steel tweezers.

Note a: When loading NaK, a one cc stainless-steel bucket is used to transfer material from a beaker to the capsule.

Note b: The potassium or NaK are hot-trapped in Mine Safety Appliance zirconium-loaded hot-trap containers for 24 hours at 1400°F immediately prior to loading. The liquid alkali metal is transferred from the hot-trap container in the dry box receptacle by means of the valving arrangement shown in Figure III-15. This plumbing is vacuum outgassed and flushed with alkali metal before transferring the material which is to be used in capsule loading.

e. The capsule is then reheated with a low-energy arc to insure that the potassium melts and flows to the bottom of the capsule.

f. The Cb-1Zr rack is then placed into the capsule. Test samples are then positioned in the capsule. The rack is used to keep specimens in an alkali metal vapor environment.

g. The capsule is then lowered into the welding jig. The capsule top with pump-out tabulation is placed on the

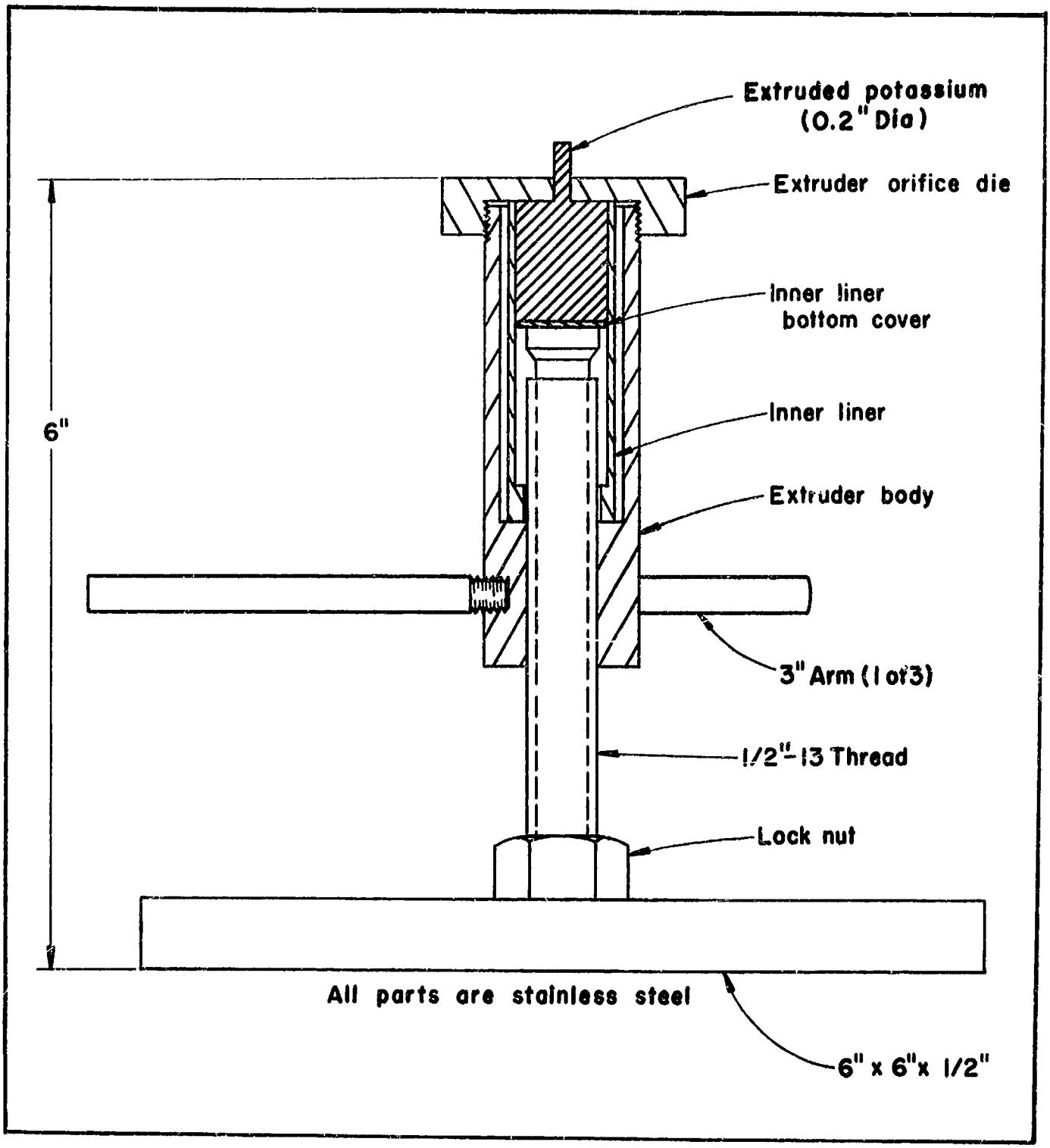


FIGURE III-14. Stainless Steel Potassium Extruder

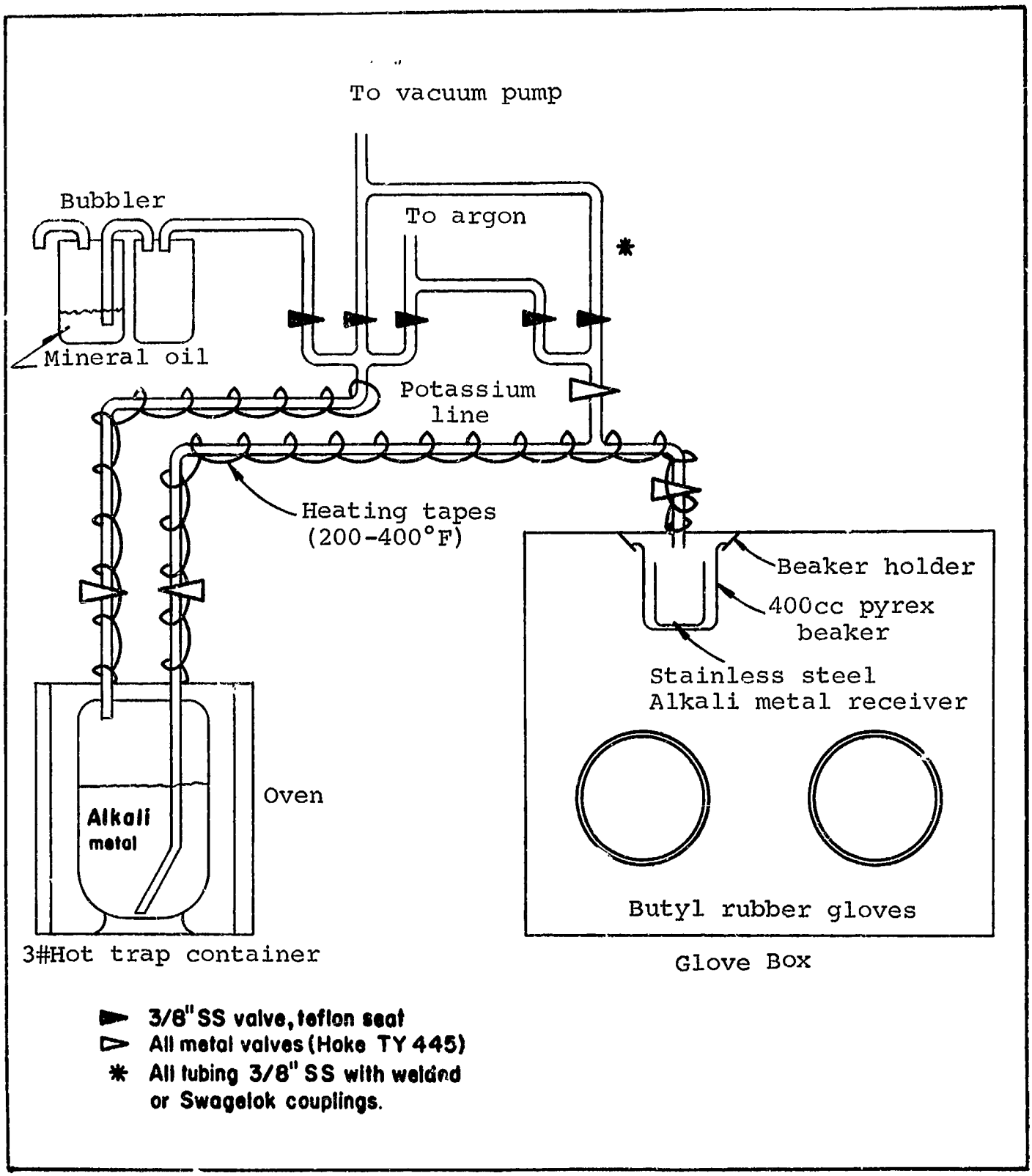


FIGURE III-15. Schematic of Alkali Liquid Metal Transfer System

open capsule and the weld area preheated with a low energy arc to vaporize any residual potassium (or NaK)[c].

h. The top is welded in place; the capsule removed from the welding jig, and visually inspected.

i. The welded capsule is then placed in a water-cooled copper chill block before evacuation and final sealing (to lower the vapor pressure of the potassium).

j. While the capsule is cooling, a second capsule is taken through steps c through i.

k. By the time the second capsule is placed into the chill block, the first capsule has cooled sufficiently for evacuation.

l. The cool capsule is then attached, with its flare fitting, to the vacuum manifold (Figure III-16) and evacuated to approximately 5×10^{-5} torr as indicated on an ionization gage. The gage is located about 20 inches from capsule fitting.

m. The evacuated capsule is then "nipped off" with hydraulically operated jaws and placed back in the welding fixture where in nip-off edge is TIG welded. (Nipped-off capsule tabulations proved to be leak-tight before welding.)

n. The second capsule has been cooled and is taken through steps l and m.

o. The two capsules are then set aside and the entire procedure repeated for the next two.

p. The alkali metal purity test capsules are loaded in the same manner and are identified with the associated batch of capsules. The purity test capsules are heated to 1000°F or 1600°F in vacuum before analyses to desorb interior surface occluded gases.

(c) Monitors indicated a sharp drop in oxygen and moisture levels after vaporizing potassium in the weld area.

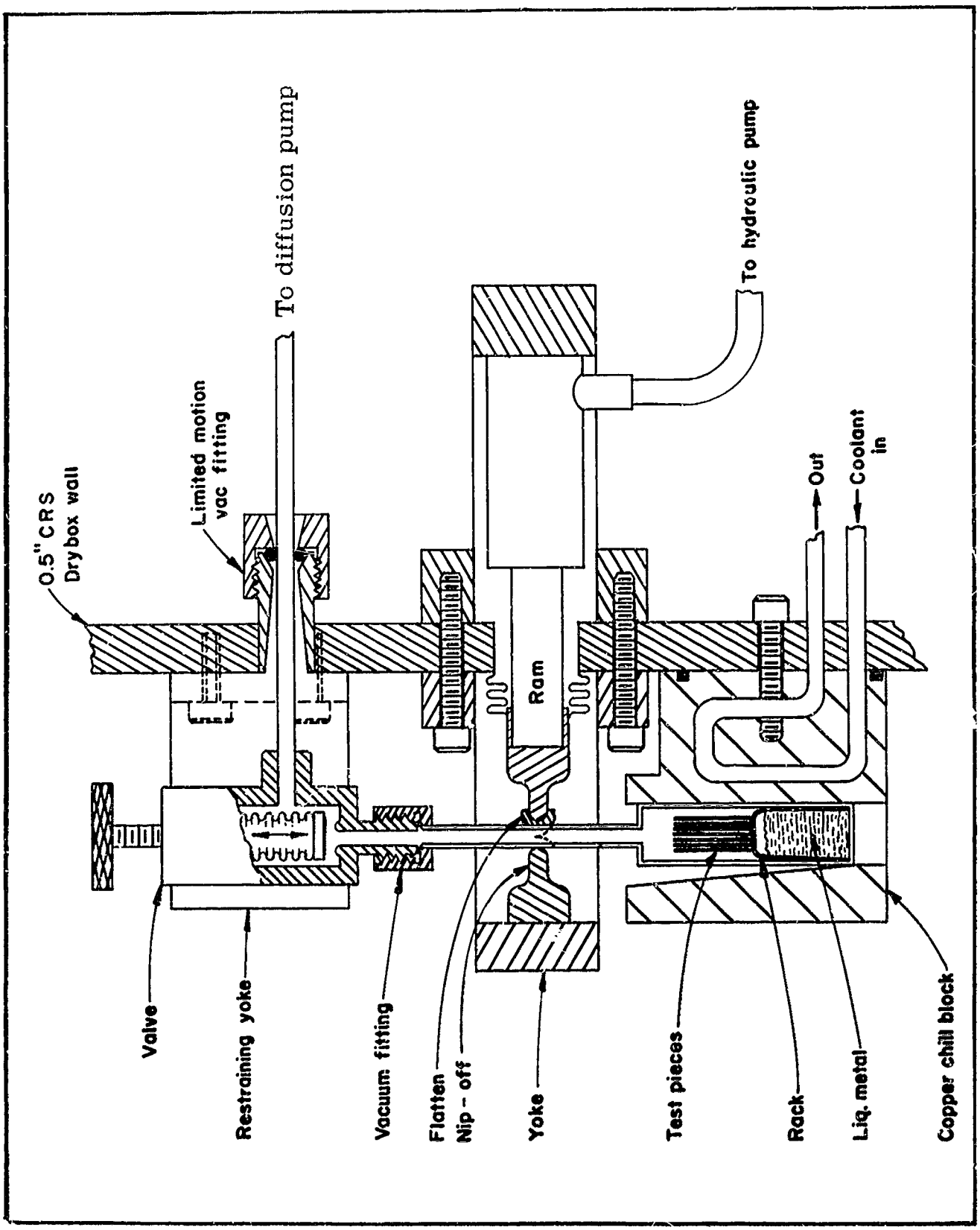


FIGURE III-16. Schematic of Capsule Evacuation and Nip-Off System

After the loading sequence, the capsules are removed from the dry box. Top welds and tubulation nip-off welds are annealed by heating the top portions of the capsules to 2200°F for one hour in a vacuum induction furnace; while the bottom portion of the capsules rest in a water-cooled chill block. Radiation shielding is provided in the set up between the furnace and chill block. The potassium is kept below the 1600°F exposure test temperature. Vacuum is maintained on the 10^{-6} scale.

The capsules are then ready for elevated temperature exposure in the vacuum furnace.

3. Alkali Metal Exposure

After loading and annealing, the capsules are wrapped successively with 0.001 inch thick titanium and tantalum foil to limit oxygen pickup by the capsule. The capsules are then placed in the tantalum element vacuum furnace (Figure III-17) and reach the exposure temperature (1600°F or 1000°F) within approximately two hours. Temperature is monitored by two Pt-Pt + 10 percent Rh thermocouples placed respectively one inch from the top and one inch from the bottom, and inside of two dummy capsules in the furnace. At approximately 1600°F, the top thermocouple indicates 23°F higher than the bottom. Since there is supersaturated potassium vapor in the capsules, any excess potassium remains in the relatively cool bottom of the capsule. The vapor pressure is thus set by the temperature at the capsule bottom. The lower thermocouple is being used to set the furnace temperature at 1600°F with potassium pressure of approximately 2.3 atmospheres.

Furnace temperature was controlled manually and was observed to vary ± 10°F from the control point, with excursions to as much as ± 30°F. The vacuum system for the furnace described above is shown in Figures III-18 and III-19. The pumping system is capable of an ultimate vacuum of less than 1 x 10^{-6} torr. It consists of a Welch Model 1397B, 15 cfm forepump, a 400 liter/second oil diffusion pump, a Freon 12 chilled chevron baffle, a constant level liquid nitrogen trap, and a manifold with ionization gage vacuum monitor. Provisions are made for dry nitrogen letdown between furnace cycles. Potassium monitors which consist of two electrical terminals separated by 0.250 inch thick ceramic are included. In the event of a capsule leak, the released vapor

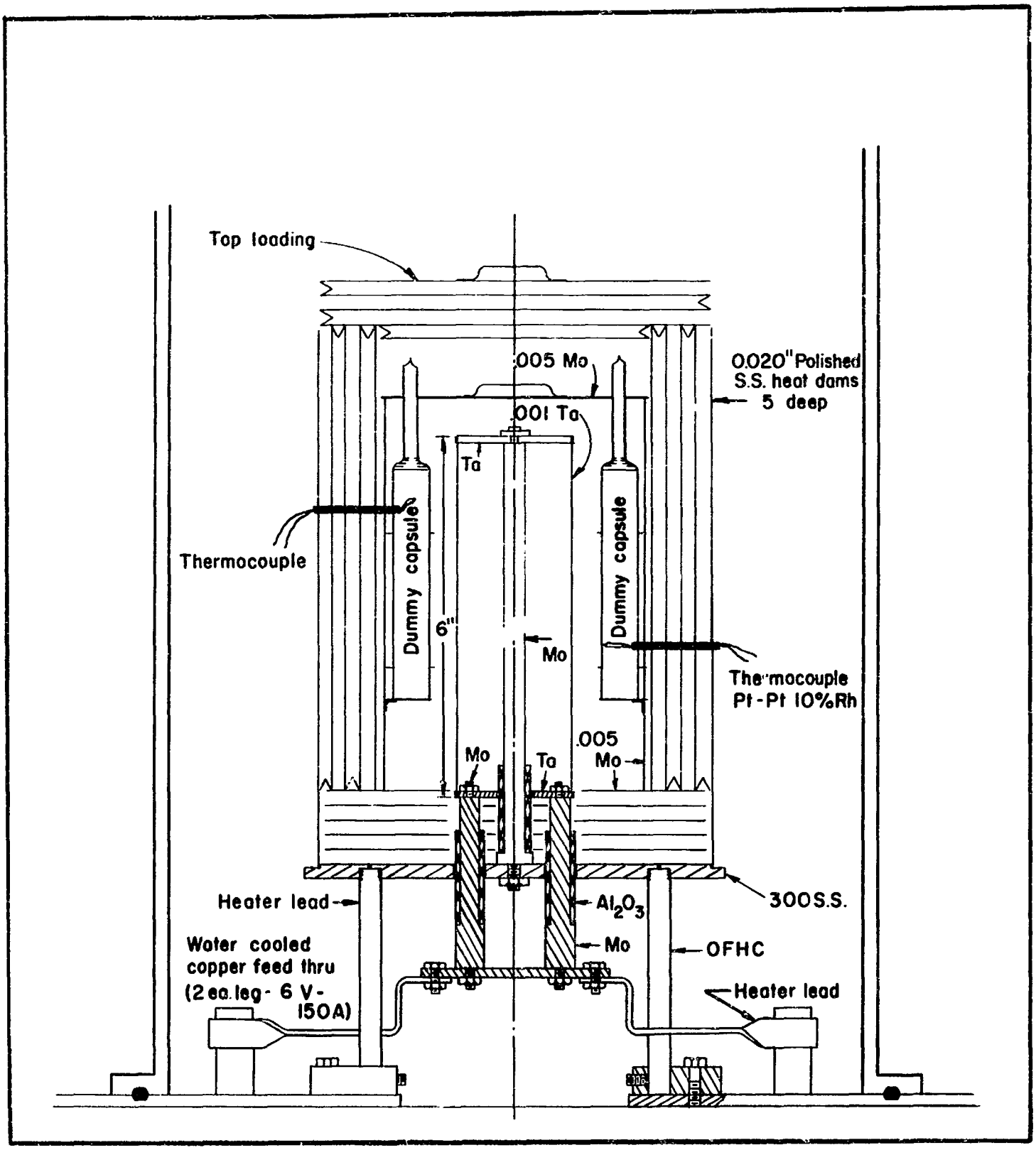


FIGURE III-17. Tantalum Element Furnace

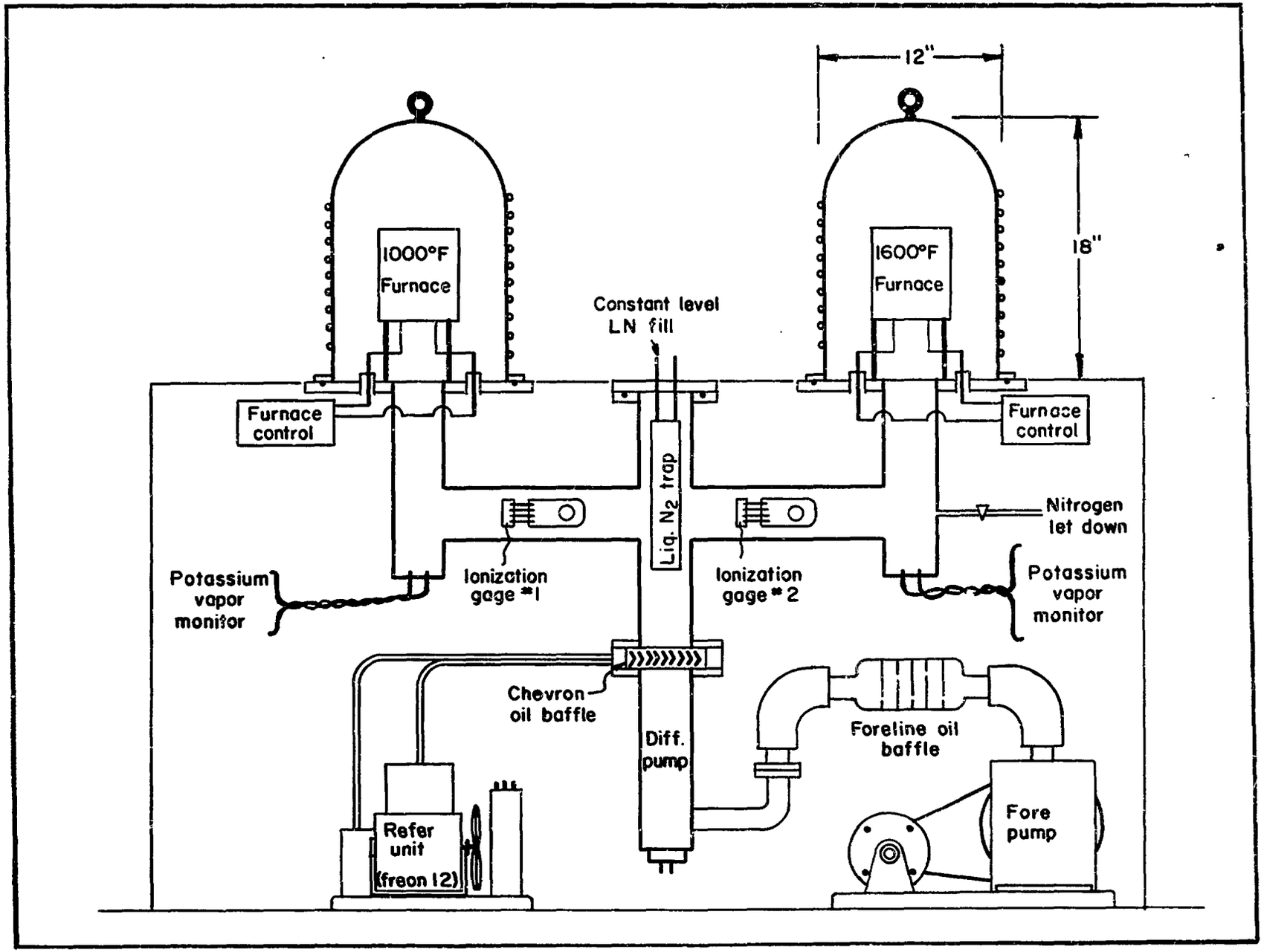


FIGURE III-18. Schematic of Environmental Test Facility

FIGURE III-19. Dual Vacuum Furnace

condenses on the ceramic and lowers the resistance between the terminals.

A Philips ionization gage which was used to monitor vacuum in the described vacuum system normally indicated 5×10^{-6} torr after one hour at temperature and decreased to 1×10^{-6} after 24 hours. After 200 hours, it indicated less than 1×10^{-6} torr. While the pressure in the vacuum furnace was of course higher than that indicated by the gage, it was low enough that no significant oxygen permeation of the foil-wrapped test capsules would take place (LB 184). A running log of temperature and vacuum was kept on the furnaces during test runs.

SECTION IV

BORE SEAL MATERIALS PROPERTIES

This section presents bore seal material properties in tabular form. They are arranged in order of thermophysical, mechanical, and compatibility properties. Graphic presentation of properties as a function of temperature follows the tabular summary. The numbering system for this section is as follows:

Section	A.	Ceramic Member	1.	99.8% Beryllia
			2.	99.8% Alumina
	B.	Metal Member	1.	Columbium Base Alloy D-43
			2.	Tantalum Base Alloy T-111
			3.	Columbium Base Alloy Cb-1Zr

The figures are numbered in consecutive numerical sequence for the entire section. References are given on each summary sheet and curve crediting the source of data. NAS3-4162 is the reference for the data obtained on this program.

BORE SEAL MATERIALS PROPERTIES SUMMARY

A. CERAMIC MEMBER

1. Beryllia, 99.8 percent.

Availability: Beryllia, 99.8 percent, is available commercially in various pressed shapes, including tubes. For bore seal applications, bodies made by the isostatic press method are preferred.

Composition: (weight percent)

99.8%	BeO	
0.0150%	Al	Approximate upper limits
0.0100%	Fe	
0.0100%	Si	
0.0080%	Ca	
0.1%	MgO	

Other elements - Ag, Cu, Cr, Mn, Mo, Na, Ni, Zn, less than 30 ppm
B, Cd, Co, K, Li, Pb, less than 10 ppm.

I. Thermophysical Properties

A.	Density (77°F)	0.105 lb/cu inch	2.90 g/cc
	Theoretical density		3.008 g/cc
B.	Melting Point (°F)		4620 ± 40°F

C. Specific Heat (LB 165)

Temperature (°F)	Btu/lb-°F
77	0.25
500	0.30
900	0.36
1600	0.46

D. Thermal Conductivity (LB 165)

Temperature (°F)	Btu-ft / ft^2-hr-°F
77	140
500	72
900	39
1600	20

E. Thermal Expansion (LB 165)

Temperature Range (°F)	Inch/inch-°F
77-500	3.5×10^{-6}
500-1000	4.5×10^{-6}
1000-1600	5.1×10^{-6}
1600-2600	7.0×10^{-6}

F. Electrical Resistivity (LB 165)

Temperature (°F)	Ohm-cm
77	
500	1×10^{13}
900	4×10^{10}
1600	3×10^{8}

G. Porosity Gas-tight

II. Mechanical Properties

A. Poisson's Ratio (77°F) 0.290 LB 165, LB 179

(1830°F) 0.38 LB 179

B. Flexural Strength (LB 165)

Temperature (°F)	Psi
77	32 000
500	32 000
900	35 000
1600	36 500
2600	27 000

C. Modulus of Elasticity (LB 192)

Temperature (°F)	Psi
77	53×10^6
500	52.5×10^6
900	52.0×10^6
1600	51.0×10^6
2600	44.0×10^6

D. Compressive Strength (LB 192)

Temperature (°F)	Psi
77	200 000
500	145 000
900	130 000
1600	38 000

III. Compatibility Properties

1. Alkali Metal - Excellent corrosion resistance in alkali metals including lithium to 1500°F.

2. Nuclear Radiation Resistance

No major damage encountered 10^{20} neutrons/cm^2 except for some loss in thermal conductivity. Slight damage may be removed by annealing at 1800 to 2700°F.

Figure IV-1. Specific Heat - 99.8% Beryllia

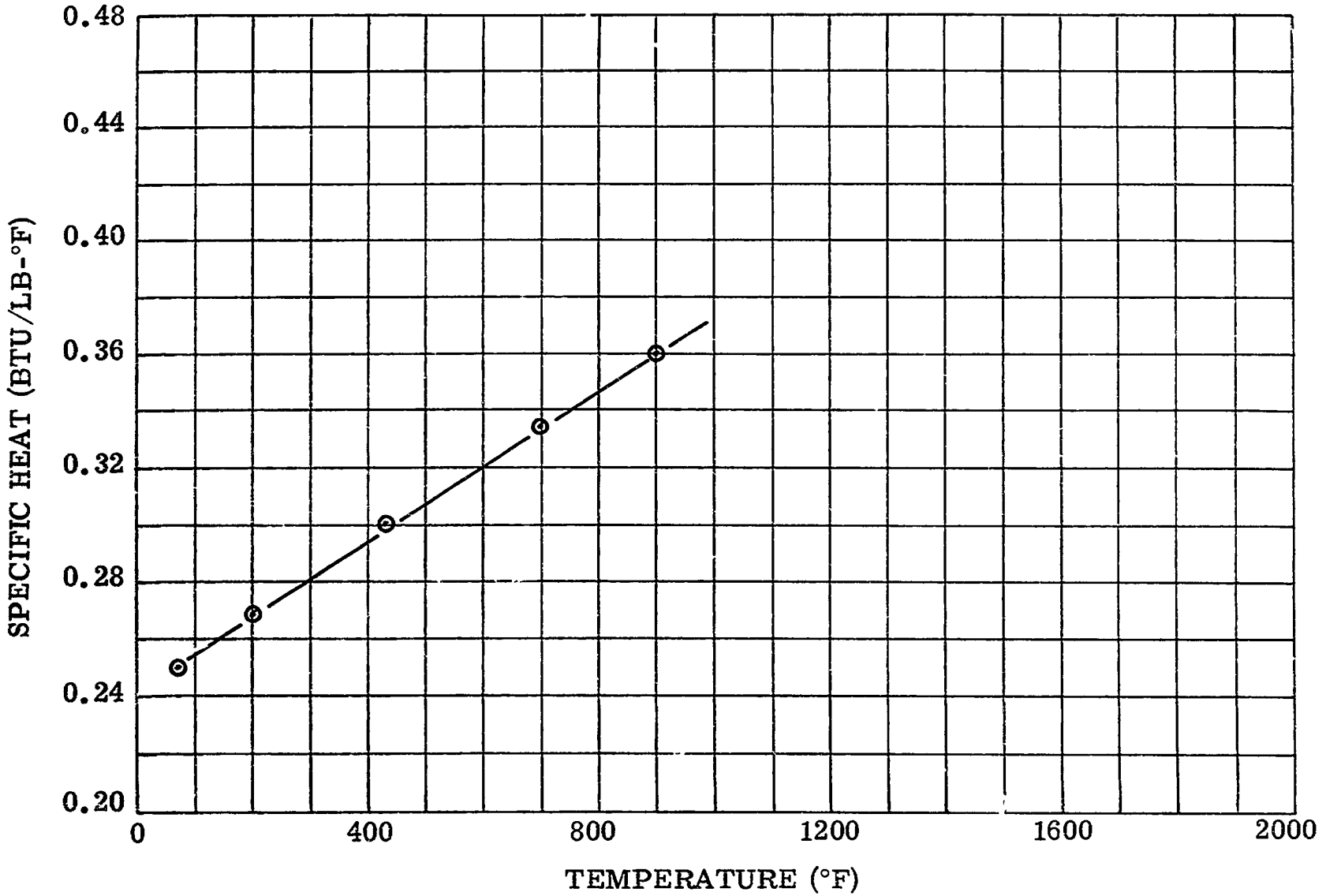


FIGURE IV-1. Specific Heat of 99.8% Beryllia Body, Density 2.90. (Reference: LB 190)

Figure IV-2. Thermal Conductivity - 99.8% Beryllia

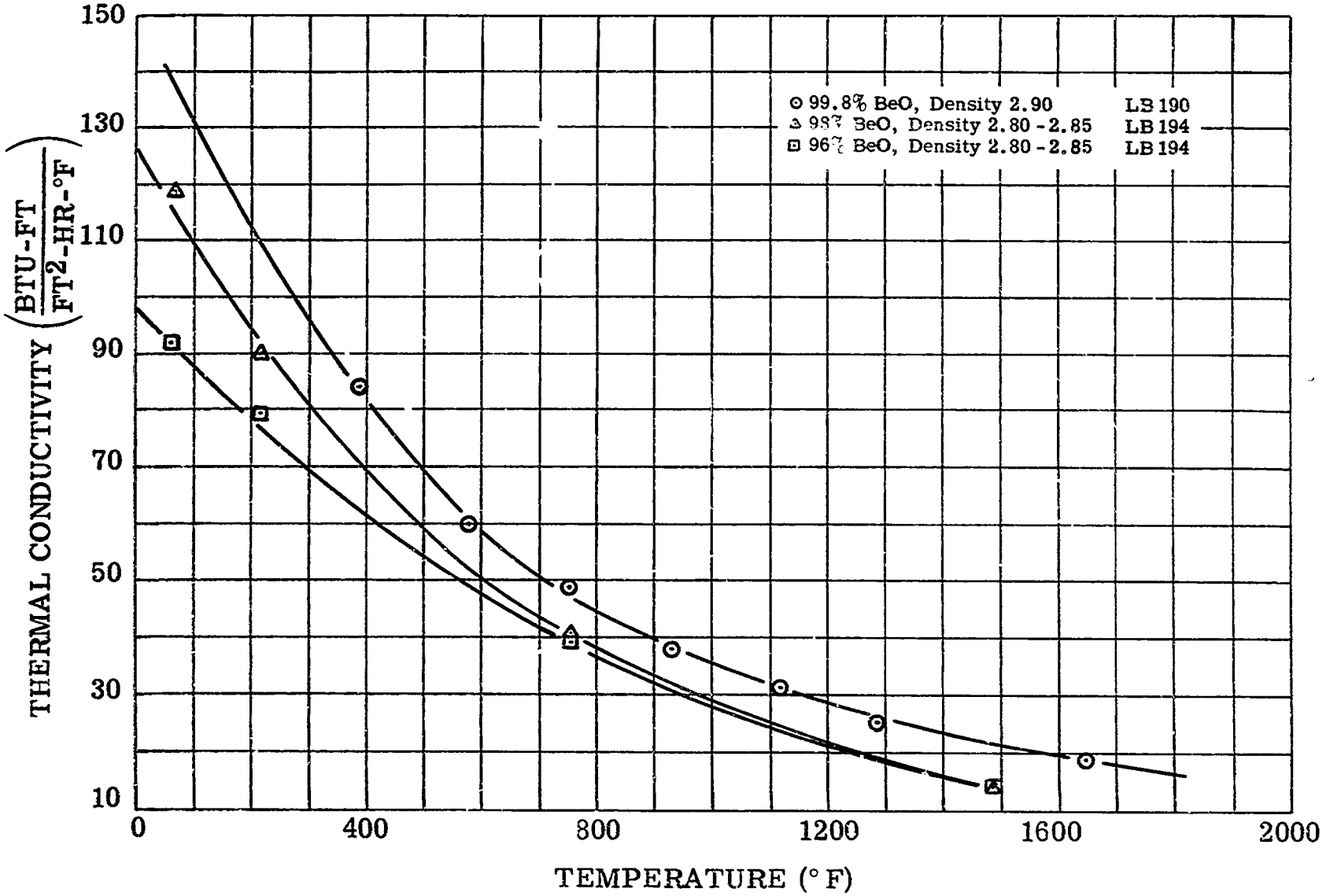


FIGURE IV-2. Thermal Conductivity of Beryllia Bodies

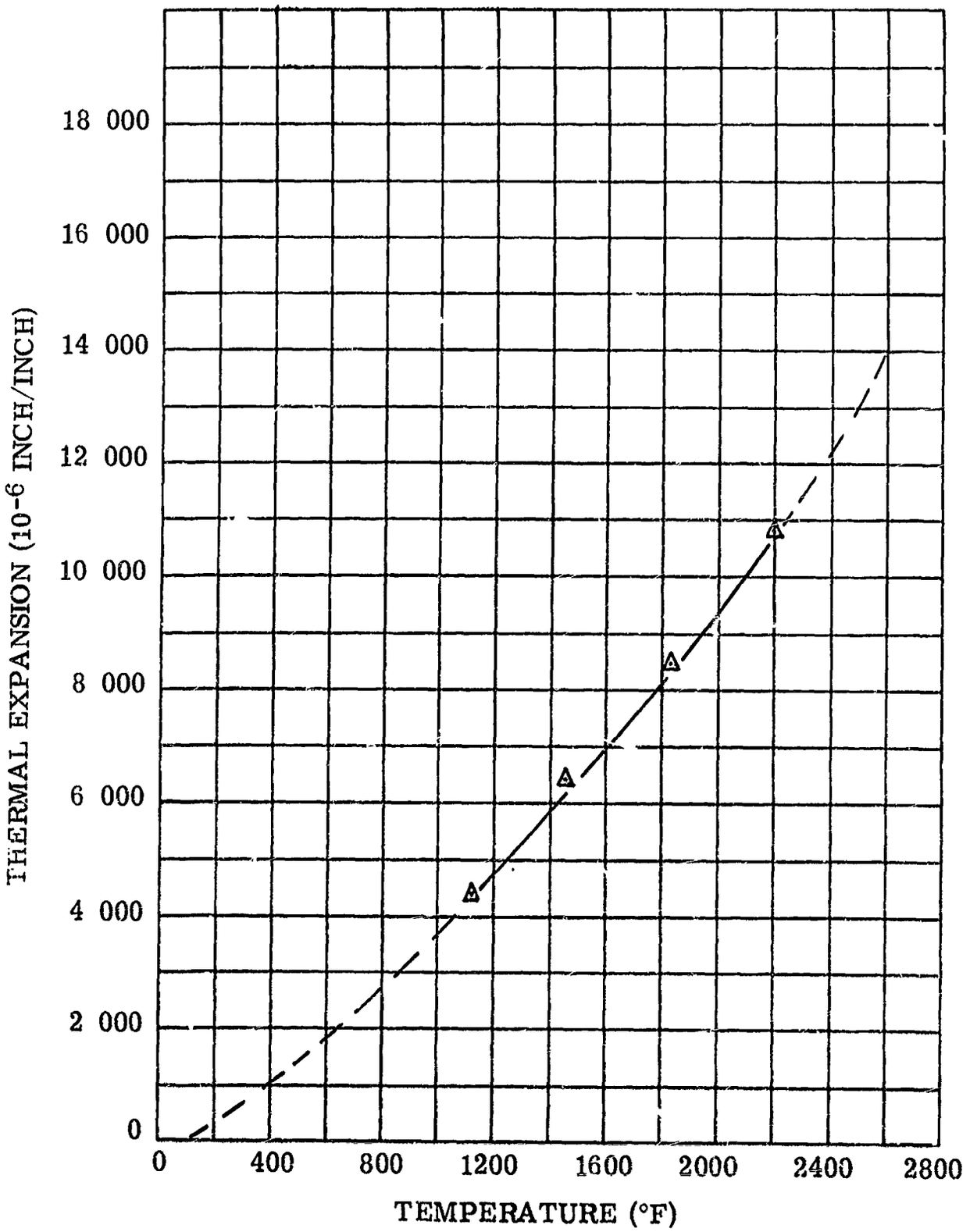


FIGURE IV-3. Thermal Expansion of 99.8% Beryllia Body, Density 2.90. (Reference: LB 165)

Figure IV-3. Thermal Expansion - 99.8% Beryllia

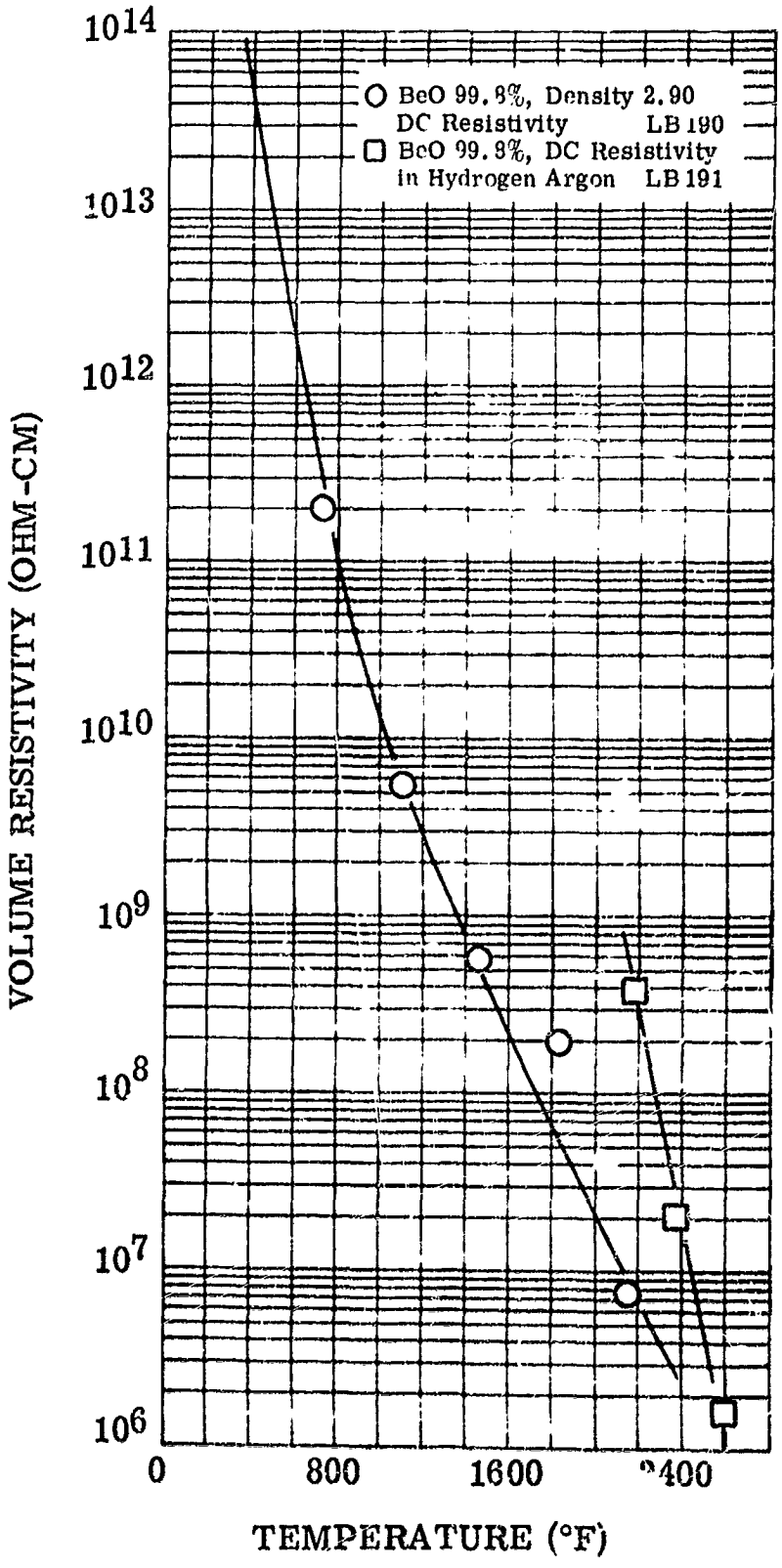


FIGURE IV-4. Volume Resistivity of 99.8% Beryllia Body, Density 2.90.

Figure IV-4. Volume Resistivity - 99.8% Beryllia

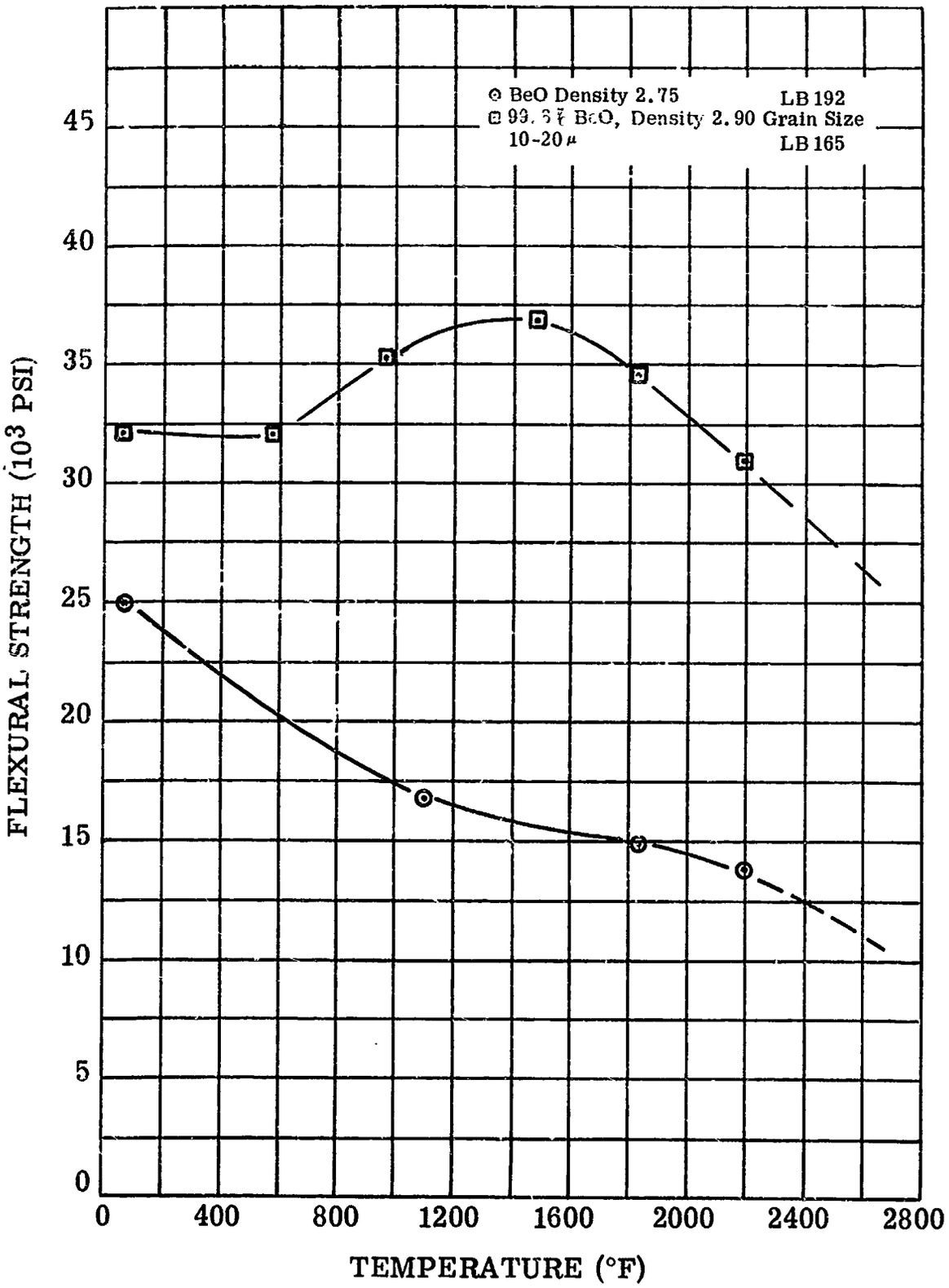


FIGURE IV-5. Flexural Strength of 99.8% Beryllia Body

Figure IV-5. Flexural Strength - 99.8% Beryllia

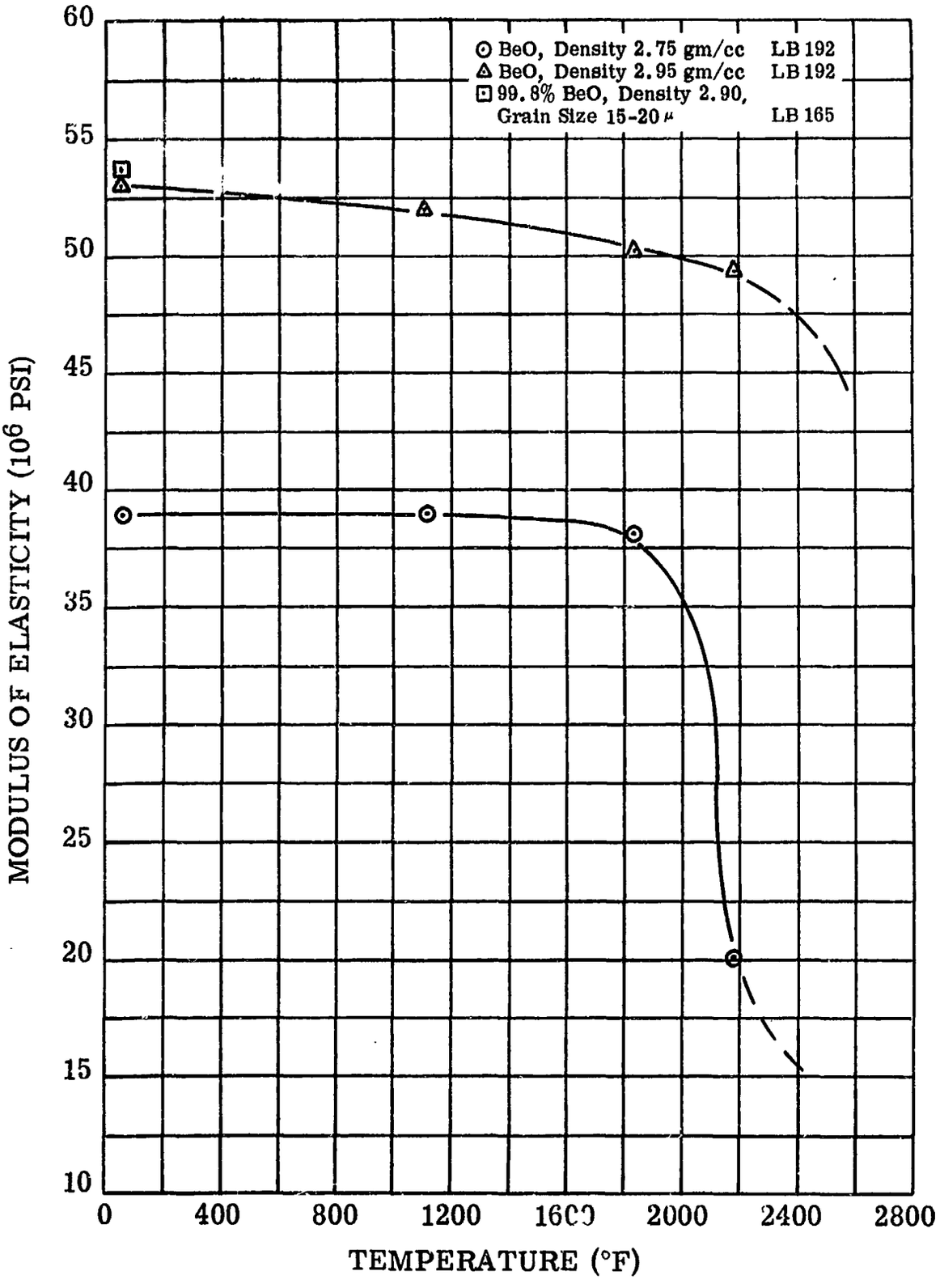


FIGURE IV-6. Modulus of Elasticity 99.8% Beryllia Body

Figure IV-6. Modulus of Elasticity - 99.8% Beryllia

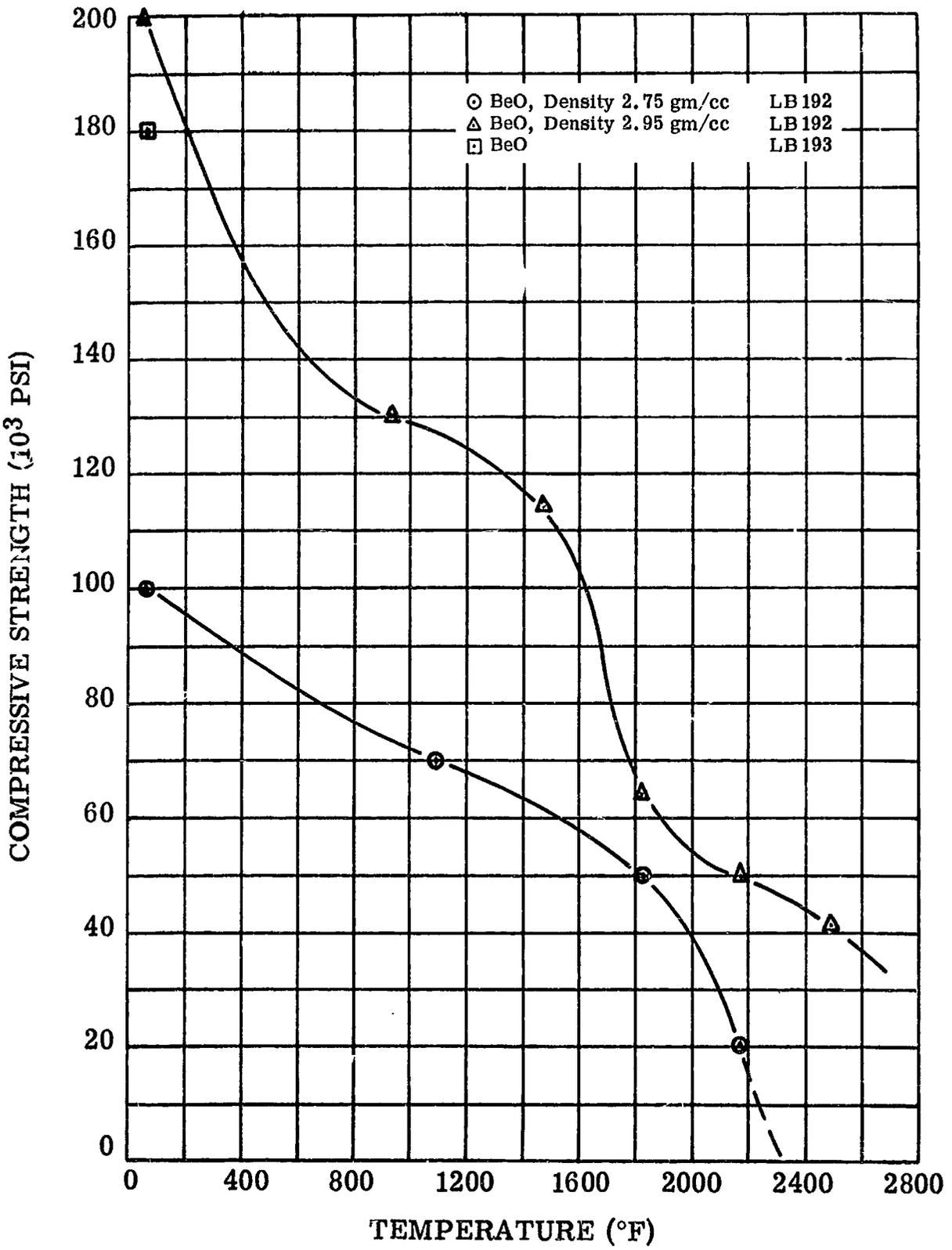


FIGURE IV-7. Compressive Strength 99.8% Beryllia Body

Figure IV-7. Compressive Strength - 99.8% Beryllia

2. Alumina, 99.8 percent Al_2O_3, 0.25 percent MgO.

Availability: This composition of alumina is available commercially from General Electric Company in various shapes and is identified as Lucalox.

Composition: (Weight)

	99.8% Al_2O_3
0.15 -	0.25% MgO
0.002 -	0.04% CaO
	0.05% SiO_2

I. Thermophysical Properties

A. Density (77°F) 0.144 lb/cu inch 3.98 gm/cc (LB 189)

B. Melting Point (°F) 3700

C. Specific Heat[(a)]

Temperature (°F)	Btu/lb-°F
77	0.32

D. Thermal Conductivity (LB 189)

Temperature (°F)	Btu-ft / ft^2-hr-°F
77	24.0
500	8.5
900	5.2
1600	3.0

E. Thermal Expansion (LB 189)

Temperature Range (°F)	Inch/inch-°F
77 - 500	3.5×10^{-6}
500 - 1000	4.6×10^{-6}
1000 - 1600	5.3×10^{-6}
1600 - 2600	6.0×10^{-6}

(a) General Electric Research Laboratory

F. Electrical Resistivity (LB 189)

Temperature (°F)	Frequency	Ohm-cm
77	DC	1.0×10^{14}
500	DC	2.8×10^{13}
900	DC	1.5×10^{12}
1600	DC	1.5×10^{7}

G. Porosity — Gas tight

II. Mechanical Properties

A. Poisson's Ratio (77°F) — 0.205 (LB 189)

B. Flexural Strength (Vacuum) (LB 187)

Temperature (°F)	Psi
77	61 000
500	57 500
900	56 500
1600	52 000
2600	32 000

C. Modulus of Elasticity (LB 187)

Temperature (°F)	Psi
77	56.0×10^{6}
500	55.5×10^{6}
900	54.0×10^{6}
1600	52.0×10^{6}

D. Compressive Strength (LB 187)

Temperature (°F)	Psi
77	300 000
2880	36 000

III. Compatibility Properties

1. Alkali Metal - Good corrosion resistance in potassium at 1600°F for 500 hours.

2. Nuclear Radiation Resistance

No significant change in electrical resistivity is anticipated when exposed to flux levels of 2×10^{20} fast neutrons/cm^2. An integrated fast neutron flux of 10^{19} to 10^{20} neutrons/cm^2, however, will result in some deterioration of the physical properties of this alumina body.

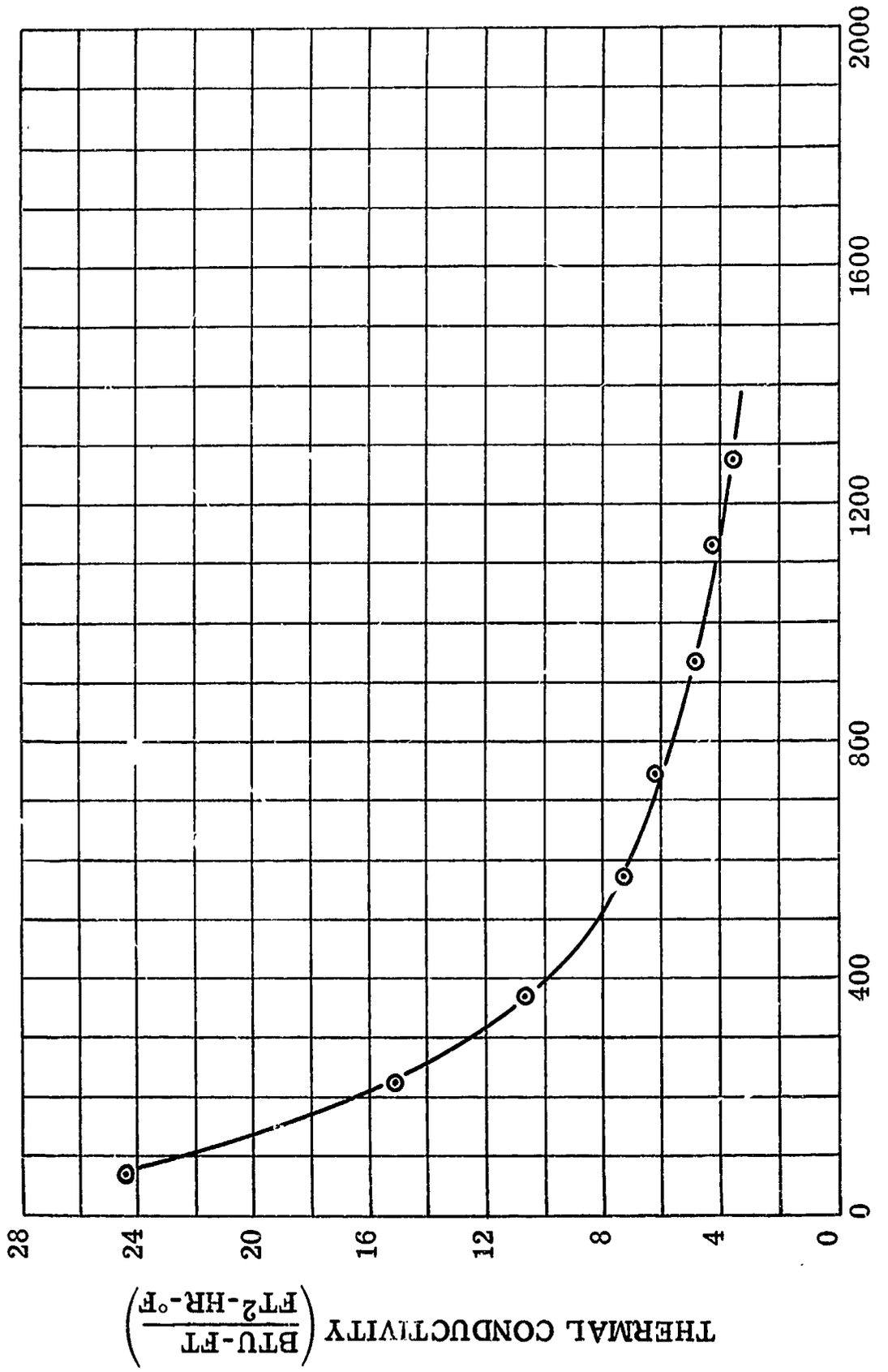


FIGURE IV-8. Thermal Conductivity of 99.8% Alumina, 0.25 MgO, Density 3.98 (Reference: LB 189)

Figure IV-8. Thermal Conductivity - 99.8% Alumina, 0.25 MgO

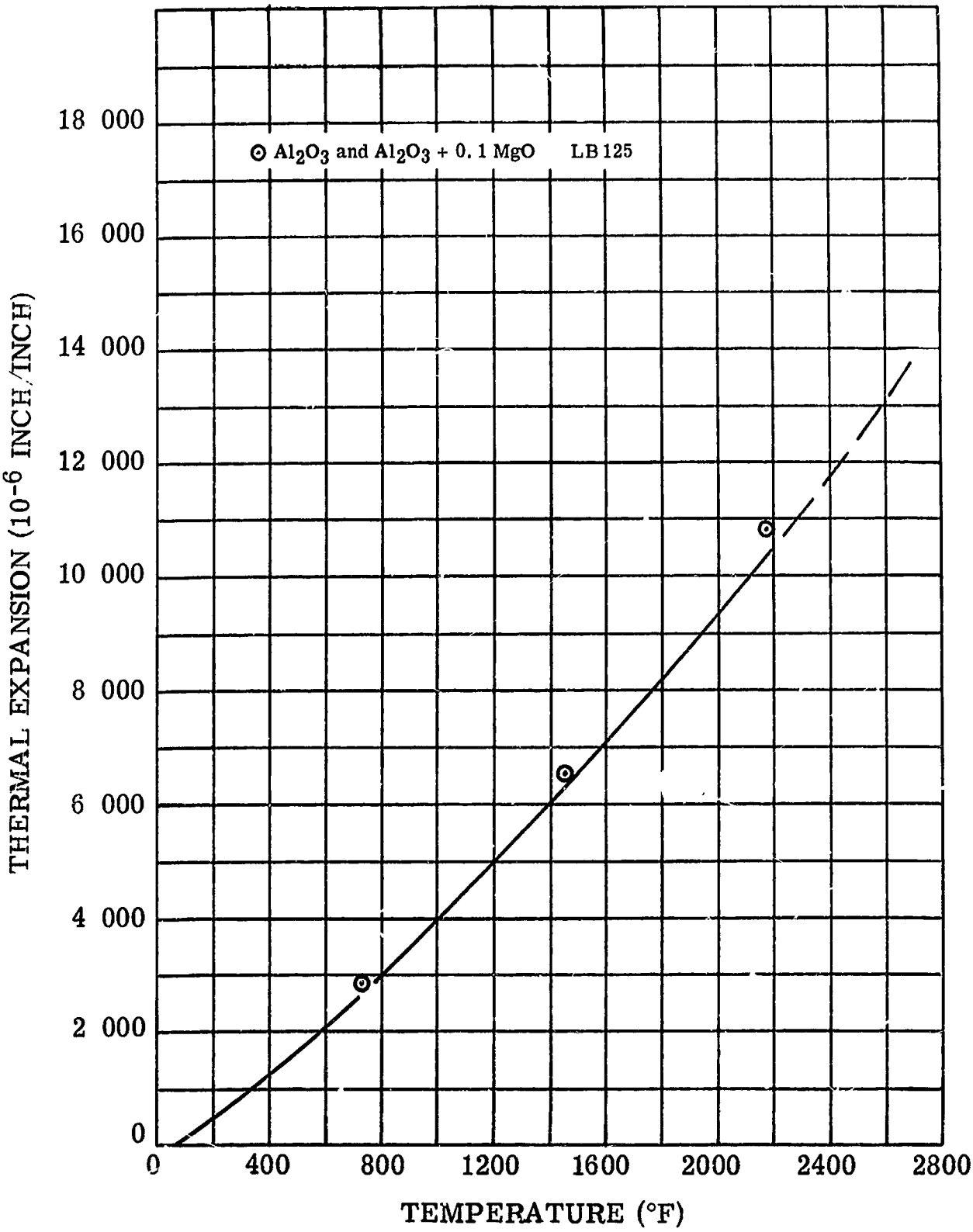


FIGURE IV-9. Thermal Expansion of 99.8% Alumina, 0.25 MgO, Density 3.98 (Curve Reference: LB 189)

Figure IV-9. Thermal Expansion - 99.8%, 0.25 MgO

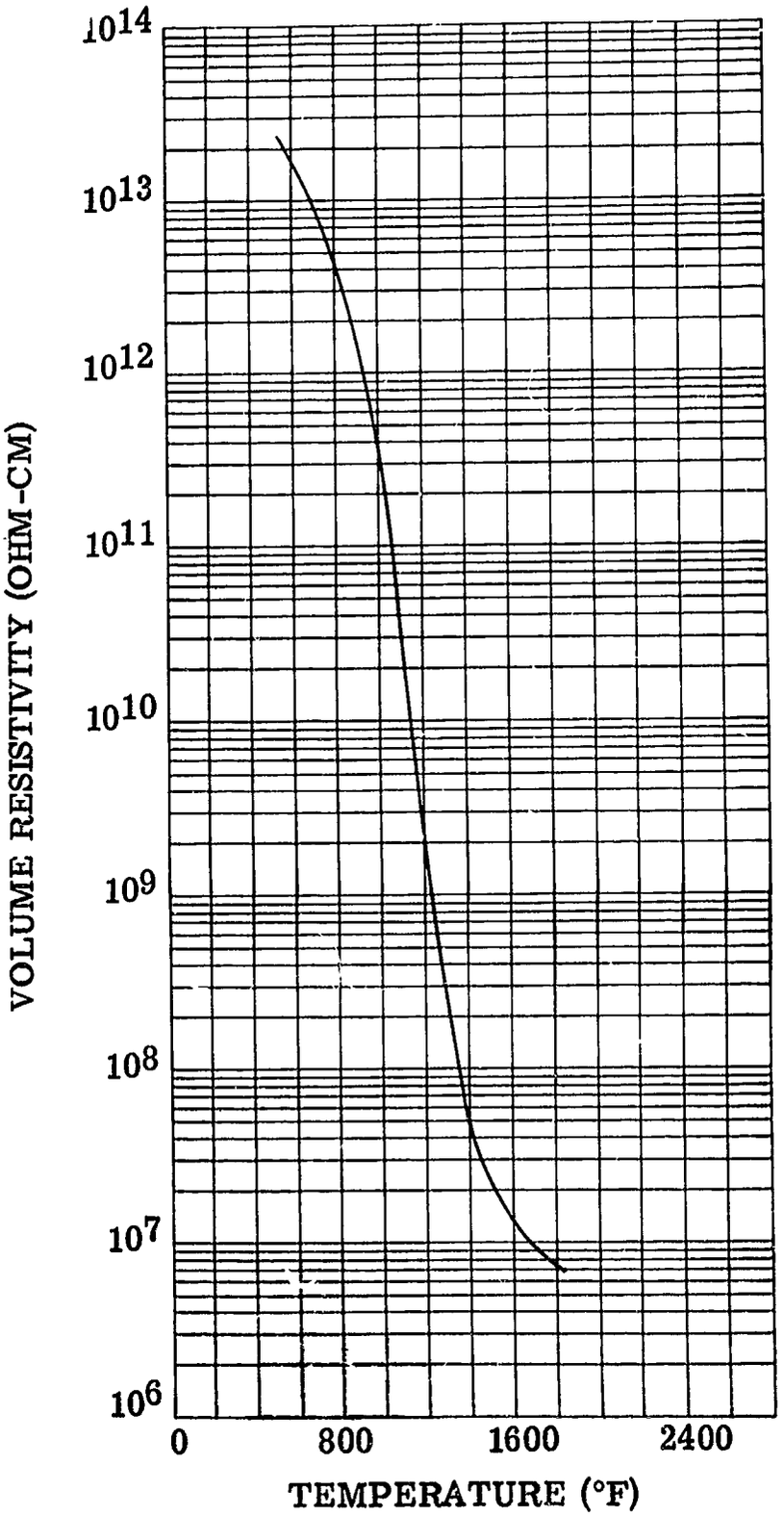


FIGURE IV-10. Volume Resistivity of 99.8% Alumina, 0.25 MgO, Density 3.98 (Reference: LB 189)

Figure IV-10. Volume Resistivity - 99.8% Alumina, 0.25 MgO

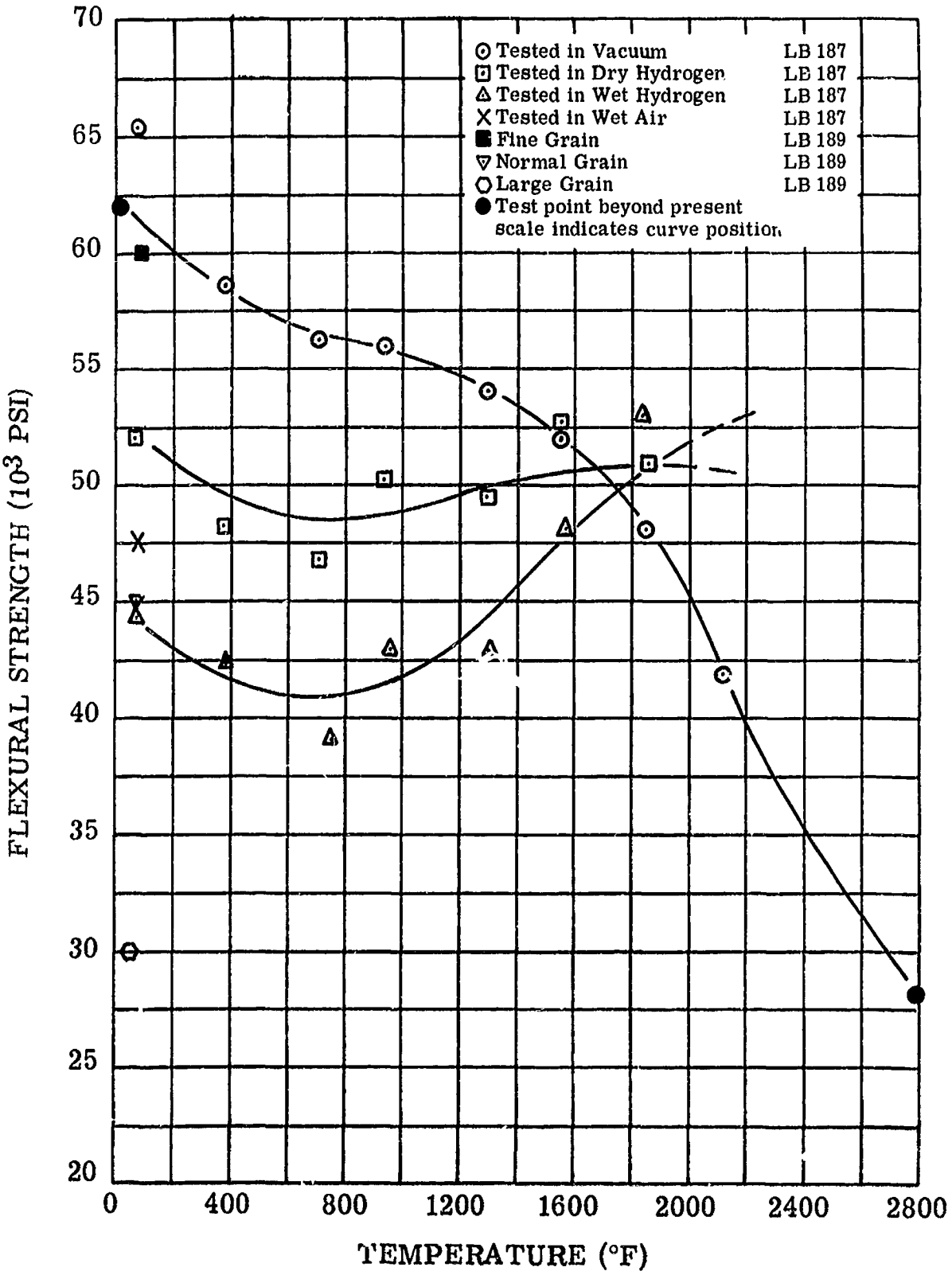


FIGURE IV-11. Flexural Strength of 99.8% Alumina, 0.25 MgO, Density 3.98

Figure IV-11. Flexural Strength - 99.8% Alumina, 0.25 MgO

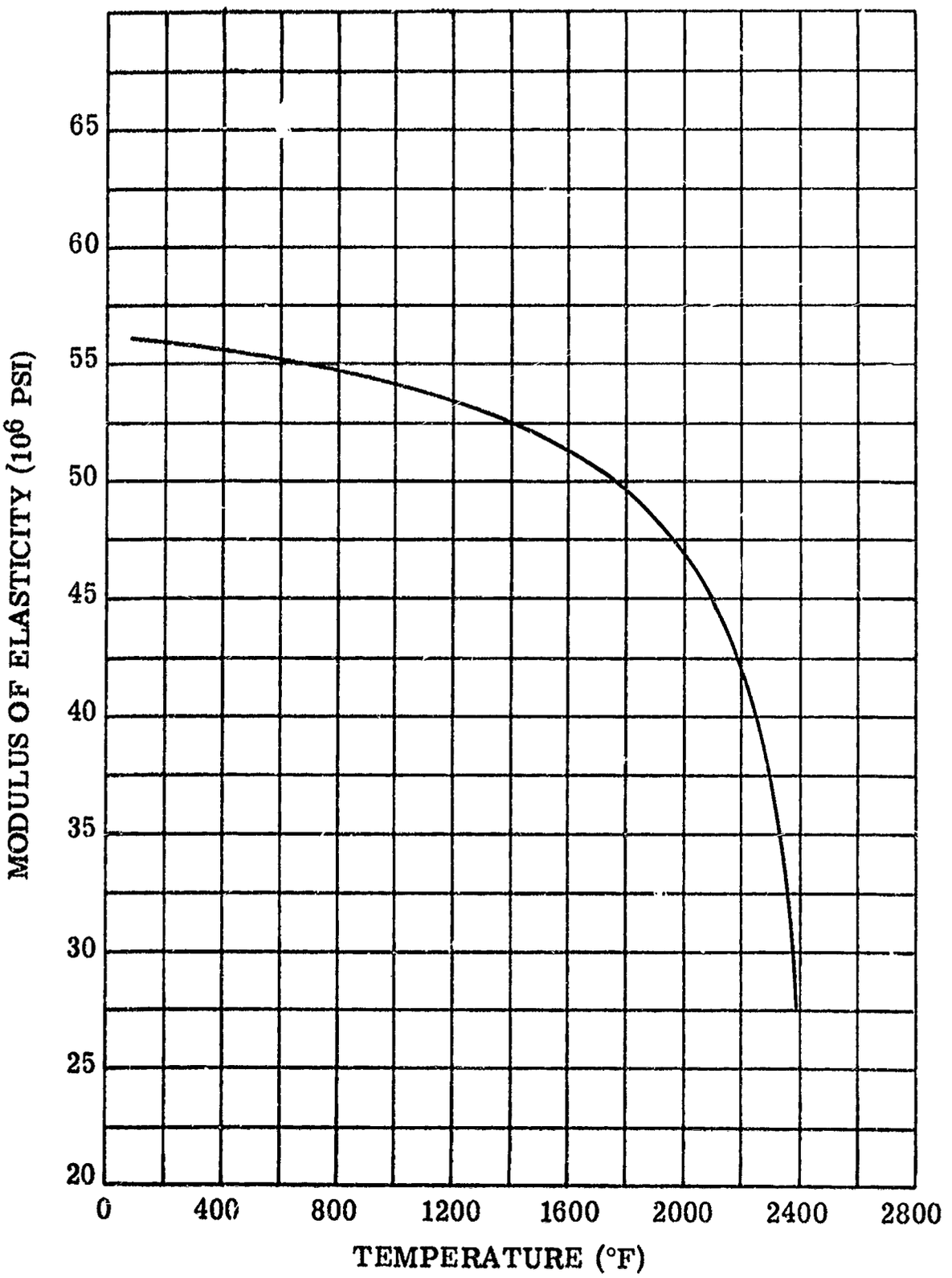


FIGURE IV-12. Modulus of Elasticity of 99.8% Alumina, 0.25 MgO, Density 3.98 (Reference: Interpolated from LB 187 using LB 189)

Figure IV-12. Modulus of Elasticity - 99.8% Alumina, 0.25 MgO

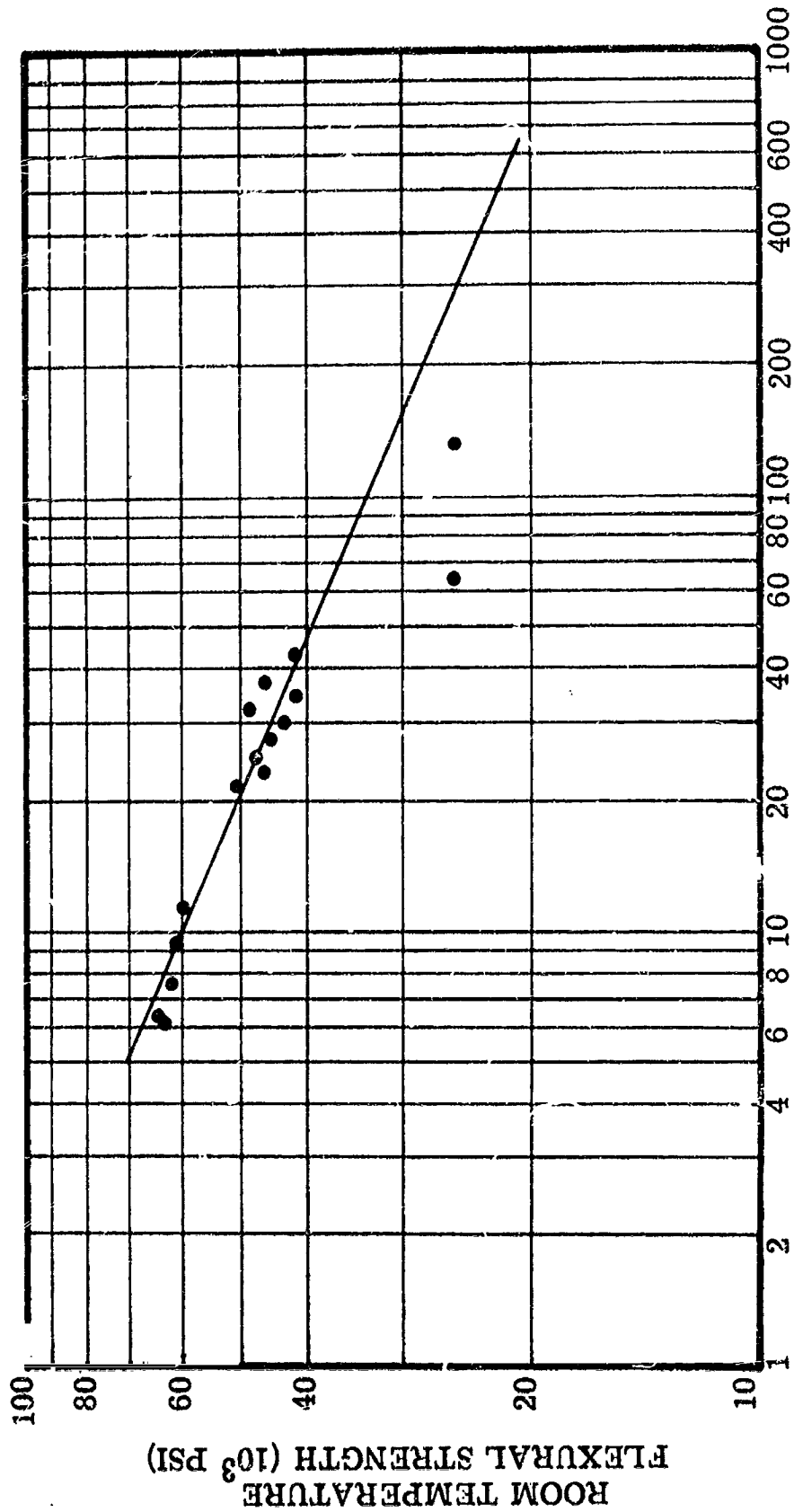


FIGURE IV-13. Room Temperature Flexural Strength of 99.8% Alumina, 0.25 MgO, Density 3.98, as a Function of Surface Grain Size. (Reference: LB 187)

Figure IV-13. Flexural Strength Versus Grain Size - 99.8% Alumina, 0.25 MgO

BORE SEAL MATERIALS PROPERTIES SUMMARY

B. METAL MEMBER

1. Columbium Base Alloy D-43 (Cb-10W-1Zr-0.1C).

Availability: Commercial; in the form of sheet, strip, plate, bar and tubing.

Composition:
9.0 - 11.0% Tungsten
0.75 - 1.25% Zirconium
0.08 - 0.12% Carbon
0.010% max Oxygen
0.010% max Hydrogen
0.0075% max Nitrogen
remainder - Columbium

I. Thermophysical Properties

A. Density (77°F) 0.326 lb/in^3 9.02 gm/cc

B. Melting Point (°F) 4500

C. Specific Heat (LB 132)

Temperature (°F)	Btu/lb-°F
77	0.052
500	0.061
900	0.069
1600	0.081

D. Thermal Conductivity (LB 132)

Temperature (°F)	Btu-ft / ft^2-hr-°F
77	34.8
500	35.0
900	35.2
1600	35.5

E. Thermal Expansion (LB 132)

Temperature Range (°F)	Inch/inch-°F
77 - 500	2.23×10^{-6}
500 - 1000	3.56×10^{-6}
1000 - 1600	4.00×10^{-6}
1600 - 2600	5.25×10^{-6}

F. Electrical Resistivity

Temperature (°F)	Ohm-cm
77	19×10^{-6}
500	30×10^{-6}
900	39×10^{-6}
1600	53×10^{-6}

II. Mechanical Properties

A. Poisson's Ratio (77°F) 0.332

B. Tensile Properties [b] (LB 132)

		Psi
1. At 77°F		
a.	0.2 percent offset yield strength	76 000
b.	Ultimate strength	86 000
c.	Modulus of elasticity	16.2×10^6
2. At 500°F		
a.	0.2 percent offset yield strength	55 500
b.	Ultimate strength	69 500
c.	Modulus of elasticity	16.4×10^6

(b) The D-43 sheet was annealed for one hour in vacuum at 2600°F. The following strain rates were used for tensile tests: To 1500°F: 0.005 in/in.-min. to yield, 0.05 in/in.-min. to fracture, above 1500°F: 0.05 in/in.-min. throughout.

3. At 900°F

		Psi
a.	0.2 percent offset yield strength	54 000
b.	Ultimate strength	69 000
c.	Modulus of elasticity	16.4×10^6

4. At 1600°F

a.	0.2 percent offset yield strength	50 500
b.	Ultimate strength	59 000
c.	Modulus of elasticity	16.4×10^6

C. Creep Properties

Curves of existing creep data on sheet material are shown in Figure II-6. Long-time creep properties in high vacuum are currently being determined on NASA Contract NAS3-2545.

III. Compatibility Properties

1. Alkali Metal - Has excellent stability in alkali metals. Essentially no attack after 10,000 hours in potassium at 2000°F. Determined on NASA Contract 3-2140. Report to be published.

Figure IV-14. Specific Heat - D-43 Alloy

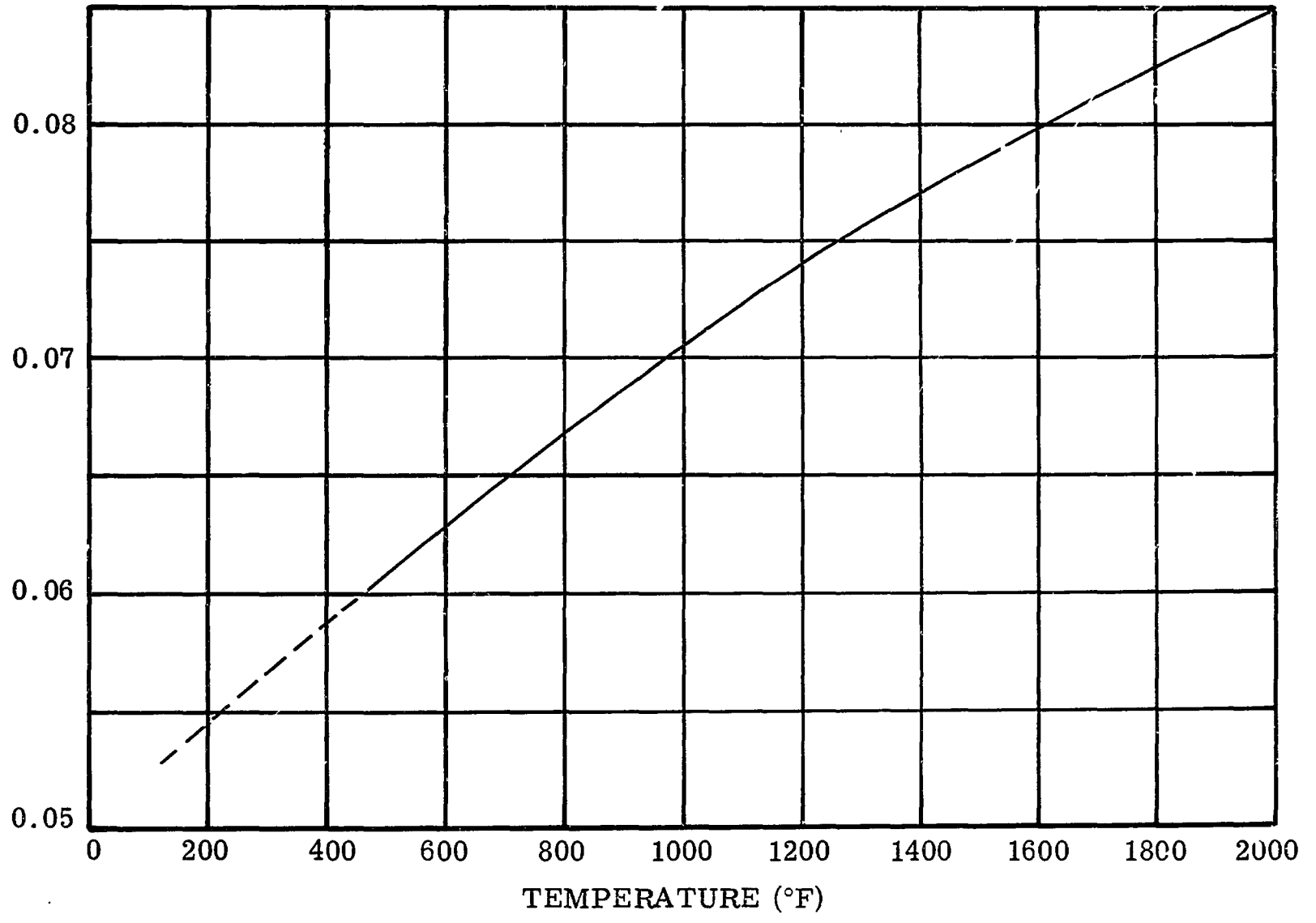


FIGURE IV-14. Specific Heat of D-43, Columbium Base Alloy. Elevated Temperature Vacuum at 10^{-5} torr. (Reference: LB 132)

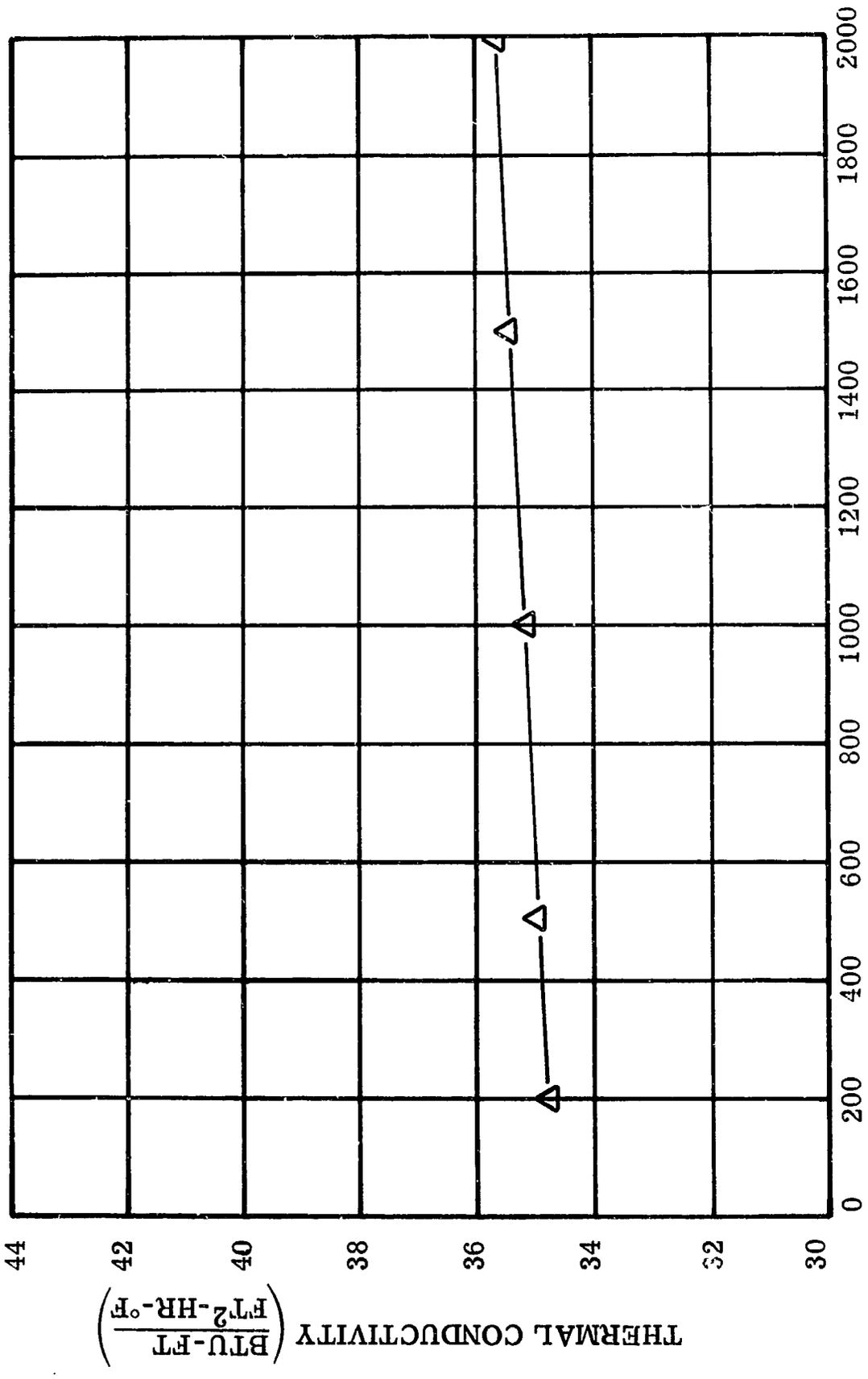


FIGURE IV-15. Thermal Conductivity of D-43 Columbium Base Alloy. Elevated Temperature Vacuum at 10-5 torr. (Reference: LB 132)

Figure IV-15. Thermal Conductivity - D-43 Alloy

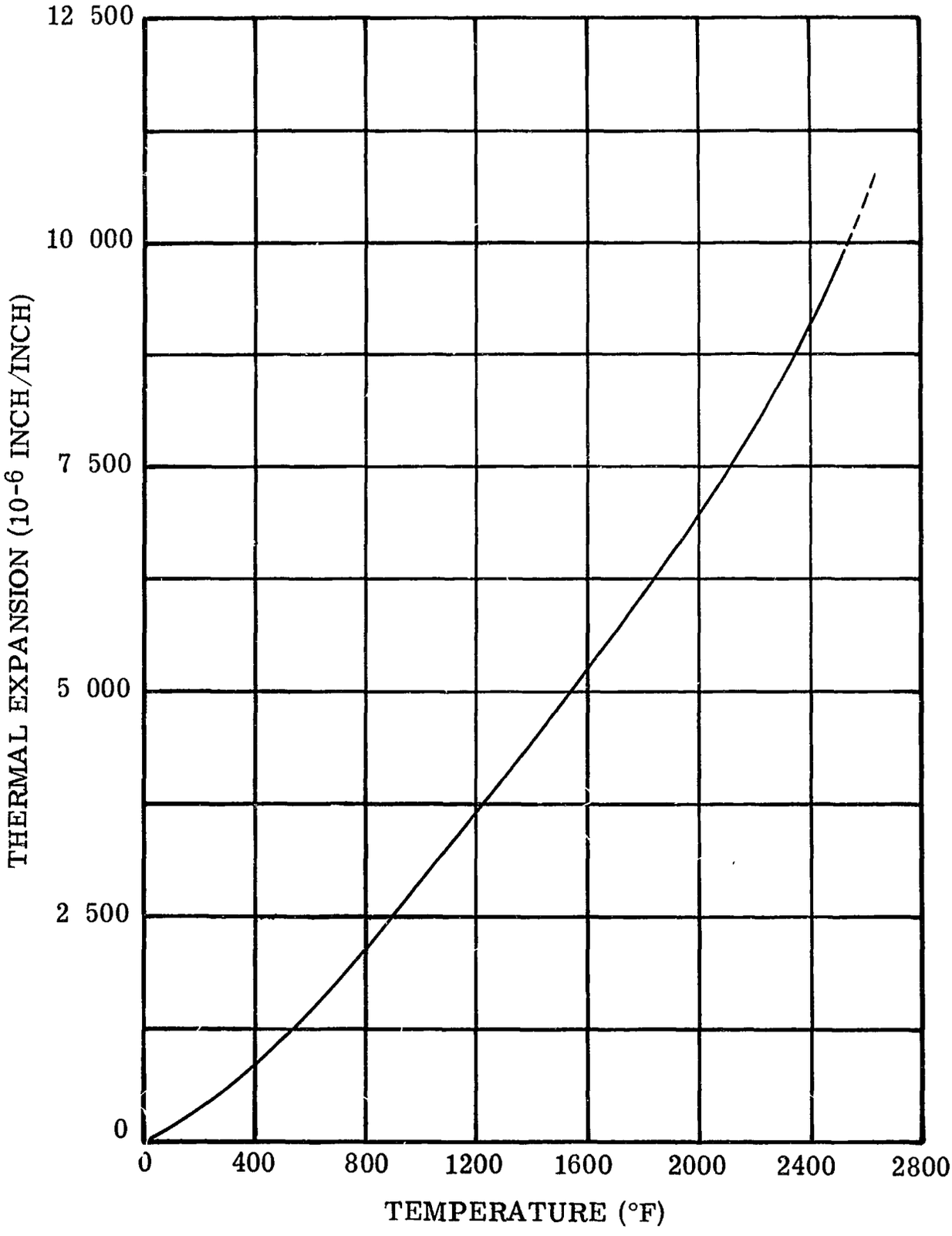


FIGURE IV-16. Thermal Expansion of D-43 Columbium Base Alloy on 0.030 Inch Sheet. Tested in Vacuum of 10^{-5} torr at Elevated Temperature. (Reference: LB 132)

Figure IV-16. Thermal Expansion - D-43 Alloy

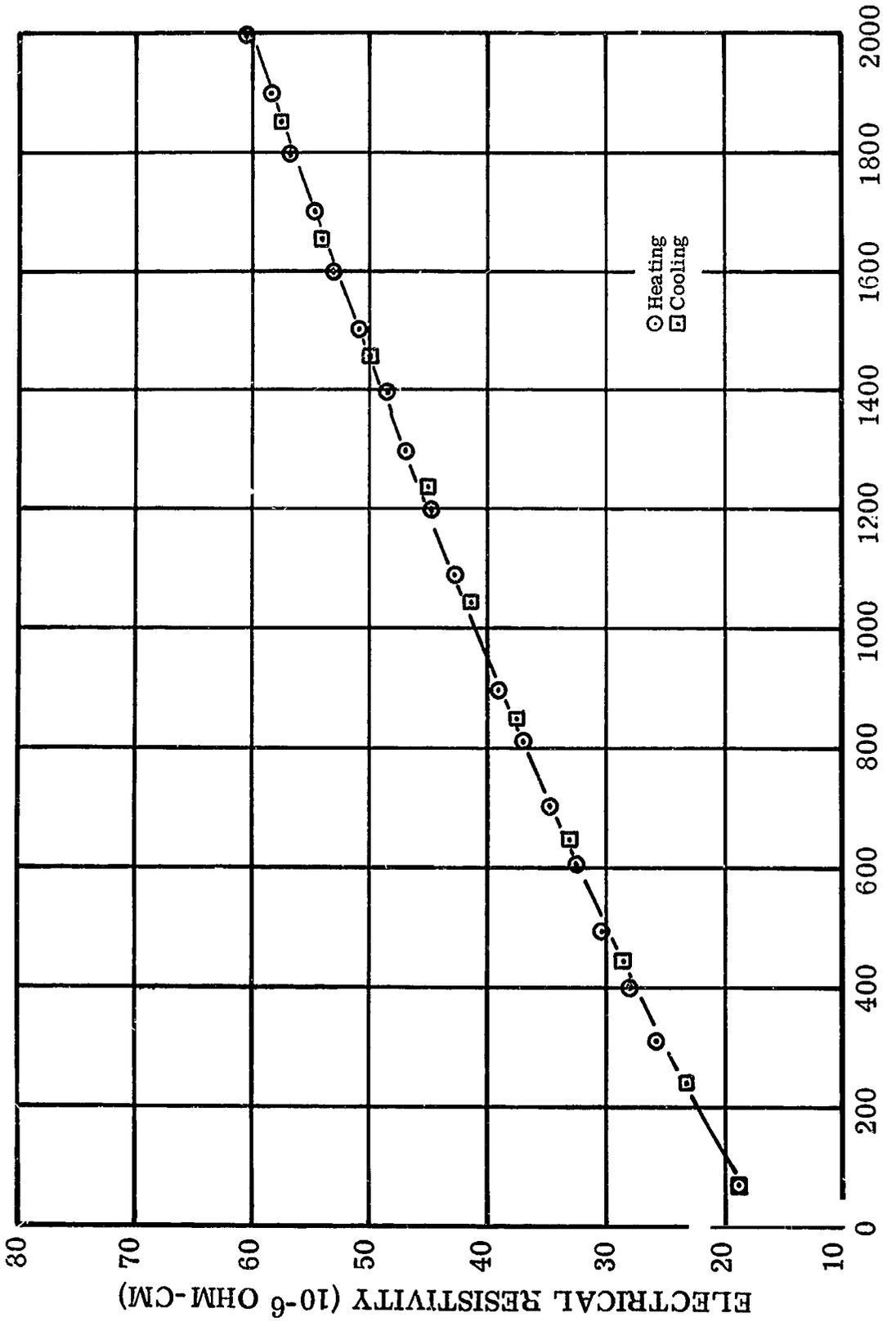


FIGURE IV-17. Electrical Resistivity of D-43 Columbium Base Alloy in Vacuum at 10^{-5} torr. (Reference: NAS3-4162)

Figure IV-17. Electrical Resistivity - D-43 Alloy

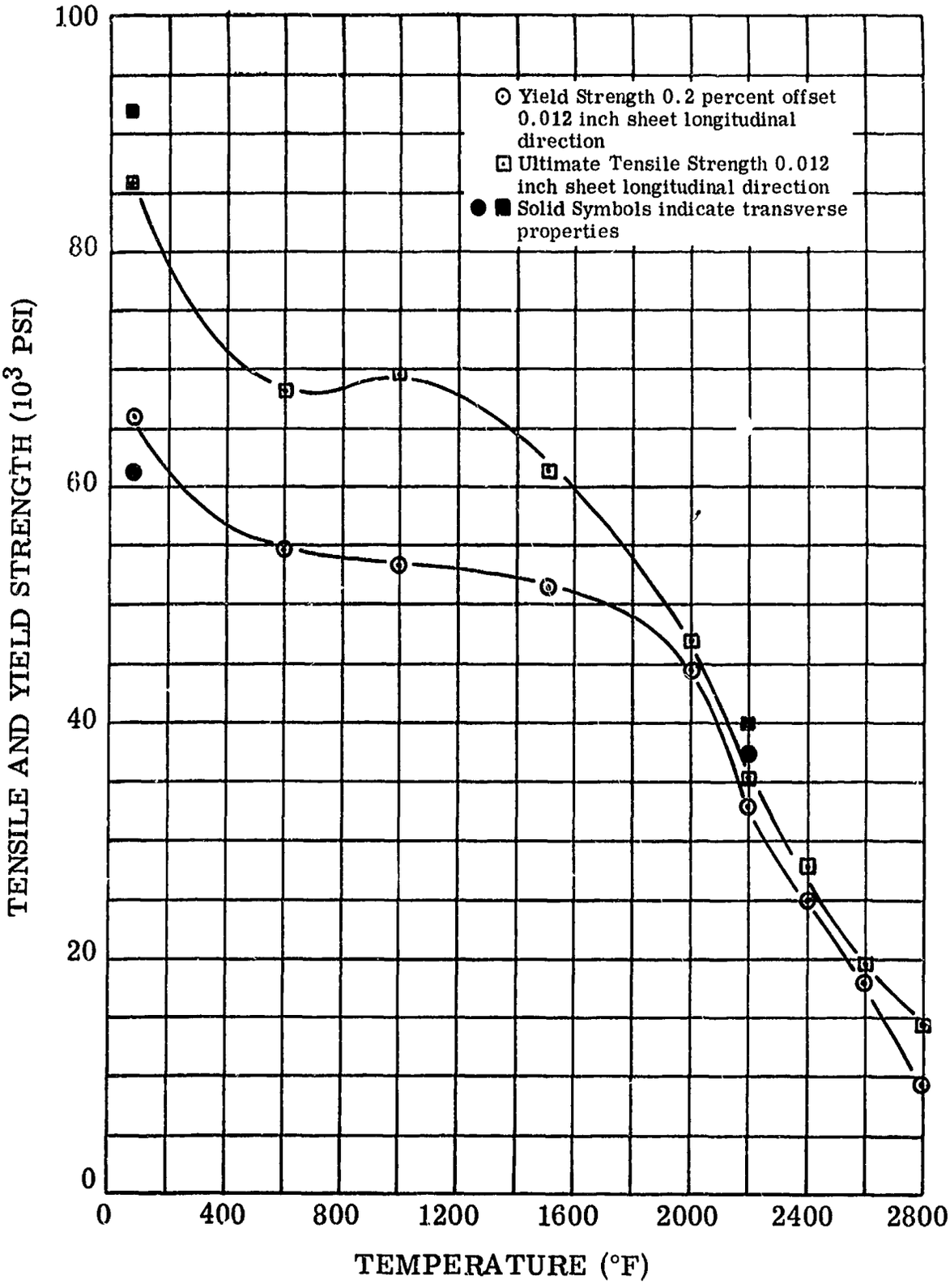


FIGURE IV-18. Tensile and 0.2 Percent Offset Yield Strength of D-43 Columbium Base Alloy - 0.012 Inch Thick Sheet: Elevated Temperature Vacuum at 10^{-5} torr. (Reference: LB 132)

Figure IV-18. Tensile and Yield Strength - D-43 Alloy

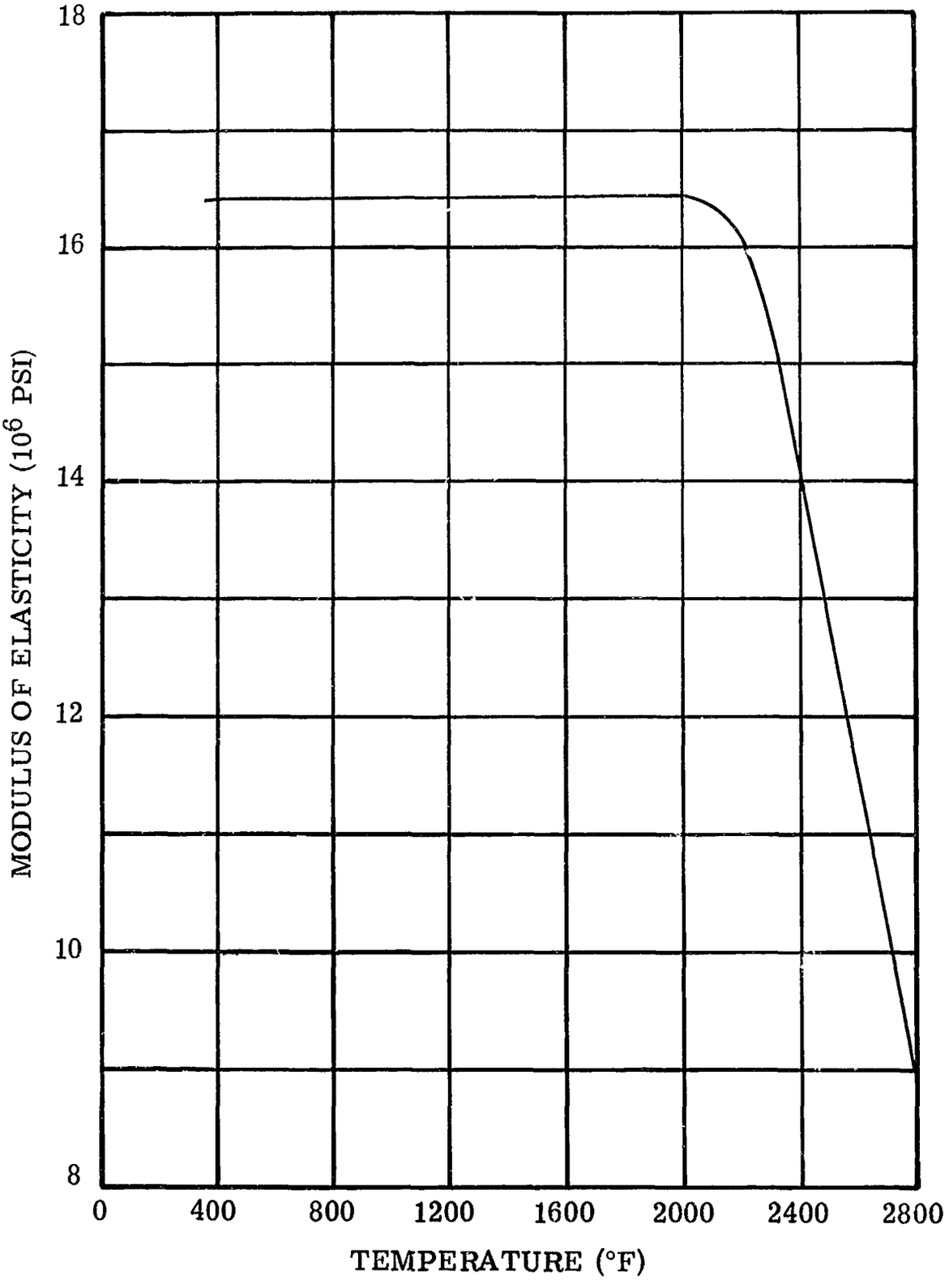


FIGURE IV-19. Modulus of Elasticity of D-43 Columbium Base Alloy. Elevated Temperature Vacuum at 10^{-5} torr. (Reference: LB 132)

Figure IV-19. Modulus of Elasticity - D-43 Alloy

2. Tantalum Base Alloy T-111 (Ta, 8W, 2Hf alloy).

Availability: Commercial; in the form of sheet, strip, plate, bar, tubing, and wire.

Composition:
7.0 - 9.0% Tungsten
1.8 - 2.4% Hafnium
0.010% Oxygen nominal
0.005% Nitrogen nominal
0.005% Carbon nominal
remainder - Tantalum

I. Thermophysical Properties

A. Density (77°F) 0.604 lb/in^3 16.72 g/cc (LB 88)

B. Melting Point (°F) 5400 (LB 88)

C. Specific Heat

Temperature (°F)	Btu/lb-°F
77	0.023
500	0.023
900	0.023
1600	0.063

D. Thermal Conductivity

Temperature (°F)	Btu-ft / ft^2-hr-°F
77	26.8
500	27.5
900	28.5
1600	30.2

E. Thermal Expansion (LB 88)

Temperature Range (°F)	Inch/inch-°F
77 - 500	3.73×10^{-6}
500 - 1000	3.74×10^{-6}
1000 - 1600	3.75×10^{-6}
1600 - 2600	4.2×10^{-6}

F. Electrical Resistivity (LB 88)

Temperature (°F)	Ohm-cm
77	22×10^{-6}
500	34×10^{-6}
900	43.5×10^{-6}
1600	57×10^{-6}

II. Mechanical Properties

A. Poisson's Ratio (77°F) 0.3 estimated

B. Tensile Properties (LB 162)

	Psi	
1. At 77°F		
a. 0.2 percent offset yield strength(c)	145 000	
b. Ultimate strength(c)	175 000	
c. Modulus of elasticity	26×10^6	(LB 186)
2. At 500°F		
a. 0.2 percent offset yield strength(c)	117 500	
b. Ultimate strength(c)	120 000	
c. Modulus of elasticity	25.2×10^6	(LB 186)
3. At 900°F		
a. 0.2 percent offset yield strength(c)	110 000	
b. Ultimate strength(c)	115 000	
c. Modulus of elasticity	24.36×10^6	(LB 186)
4. At 1600°F		
a. 0.2 percent offset yield strength(c)	88 000	
b. Ultimate strength(c)	107 500	
c. Modulus of elasticity	23.0×10^6	(LB 186)

(c) Tantalum Base Alloy, T-111, oxygen, nitrogen, and carbon less than 0.003 percent and 0.001 percent respectively, cold rolled 90 percent and stress relieved for one hour at 2000°F. A strain rate of 0.005 in./in.-min to yield and then 0.05 in./in.-min thereafter.

C. Creep Properties

Curves of existing creep data on sheet material are shown in Figure II-6. Long-time creep properties in high vacuum are being determined on NASA Contract NAS3-2545.

III. Compatibility Properties

1. Alkali Metal

Excellent tested long-time stability in alkali metals. In potassium 2000 hours to 2000° F. In cesium 2000 hours to 2600°F.

Figure IV-20. Specific Heat - T-111 Alloy

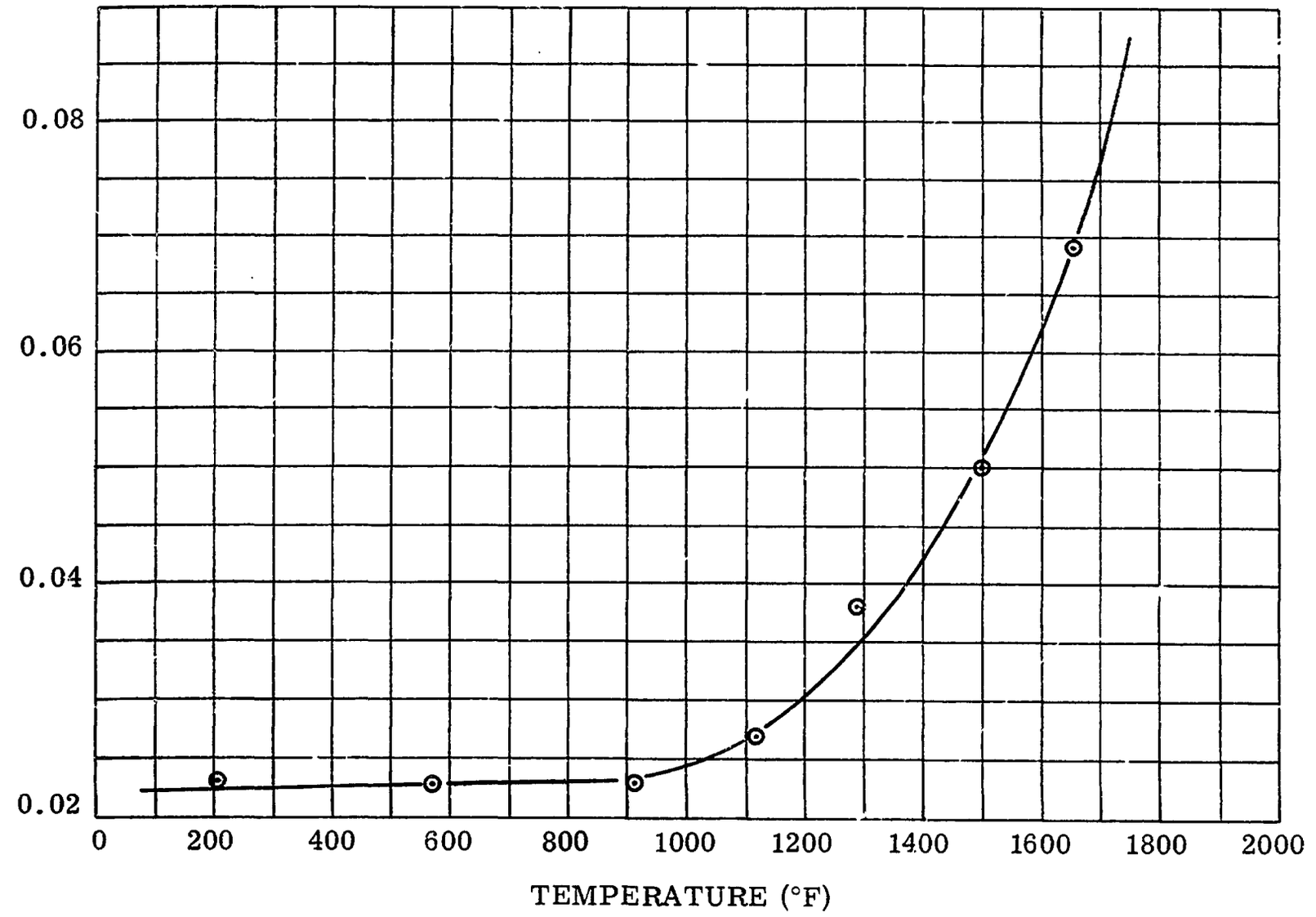


FIGURE IV-20. Specific Heat of T-111 Tantalum Base Alloy. Tested in Vacuum at 5×10^{-5} torr. (Reference: NAS 3-4162)

Figure IV-21. Thermal Conductivity - T-111 Alloy

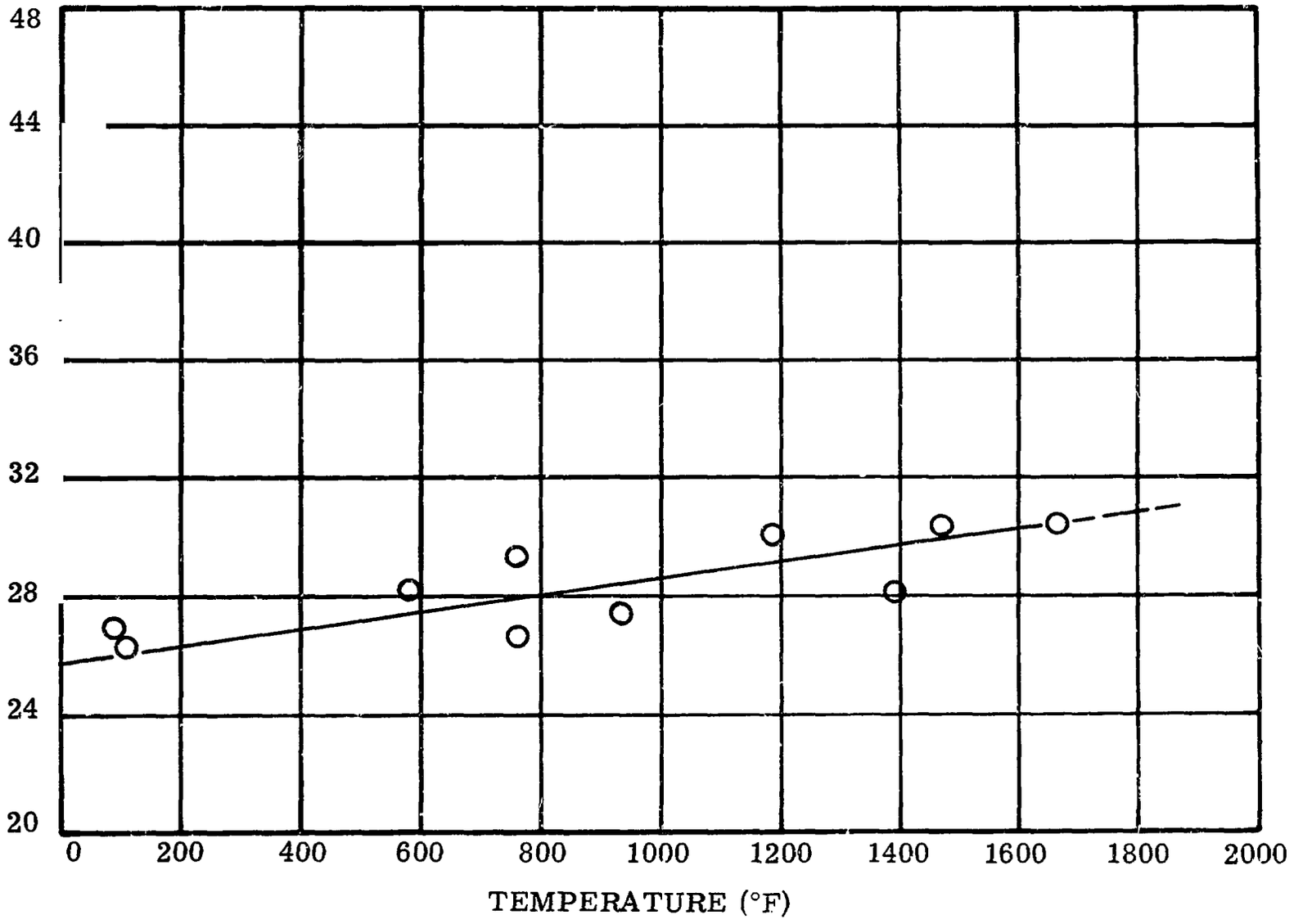


FIGURE IV-21. Thermal Conductivity of T-111 Tantalum Base Alloy in Vacuum 5×10^{-6} torr. (Reference: NAS 3-4162)

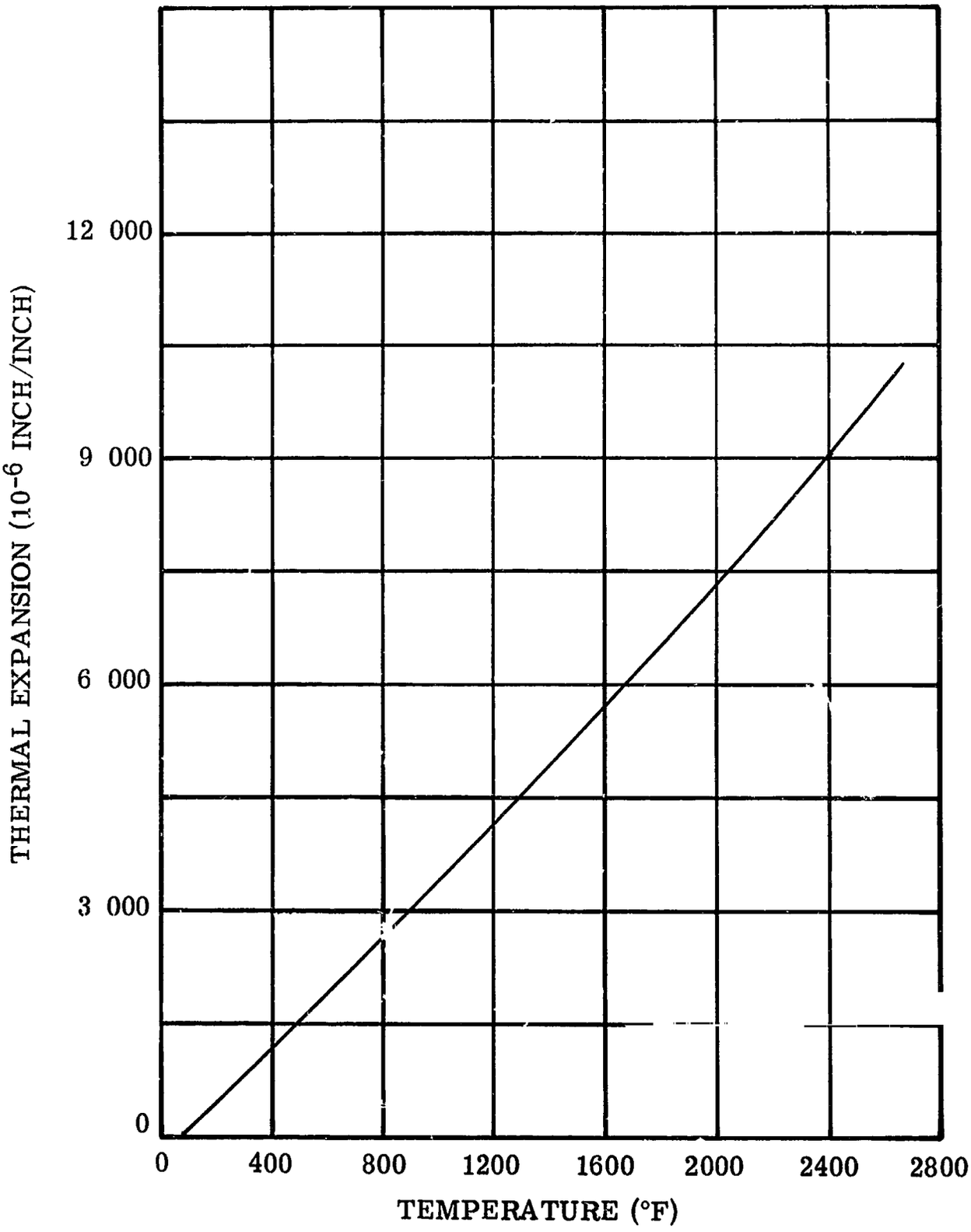


FIGURE IV-22. Thermal Expansion of T-111 Tantalum Base Alloy in Vacuum at 5×10^{-6} torr. (Reference: LB 88)

Figure IV-22. Thermal Expansion - T-111 Alloy

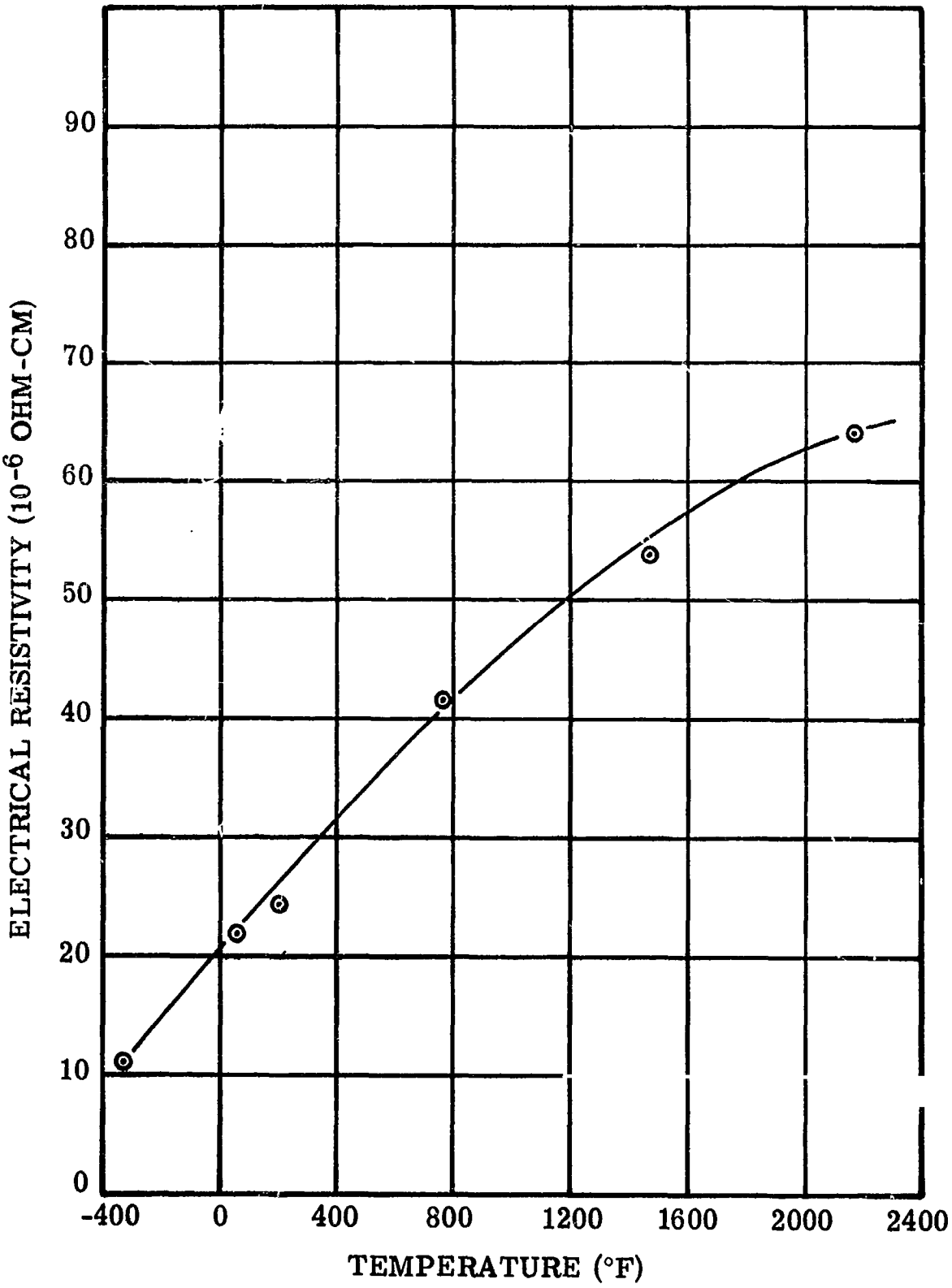


FIGURE IV-23. Electrical Resistivity of Recrystallized T-111 Tantalum Base Alloy in Vacuum at 5×10^{-6} torr at Elevated Temperatures. (Reference: LB 88)

Figure IV-23. Electrical Resistivity - T-111 Alloy

Figure IV-24. Tensile Strength - T-111 Alloy

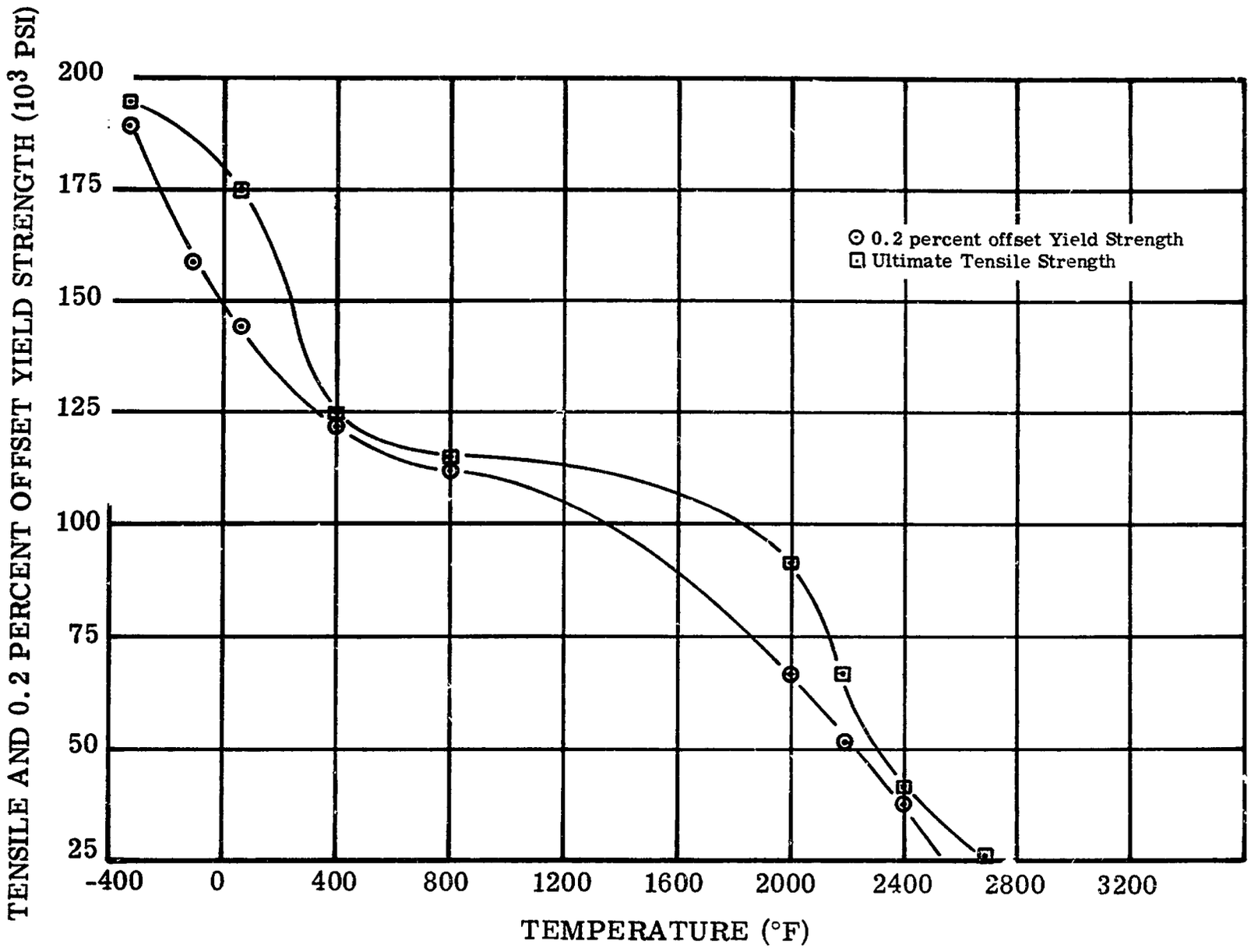


FIGURE IV-24. Tensile Strength of T-111 Tantalum Base Alloy Low Interstitial Grade Sheet Cold Rolled 90%, Stress Relieved 1 Hour at 2000°F. Elevated Temperature Tests in Vacuum at Less Than 5 x 10^{-5} torr. Strain Rate of 0.005 in/in-min. Through 0.2% Yield Strength, 0.05 in/in.-min, Thereafter. Above 800°F 0.04-0.06 in/in-min. (Reference: LB 162)

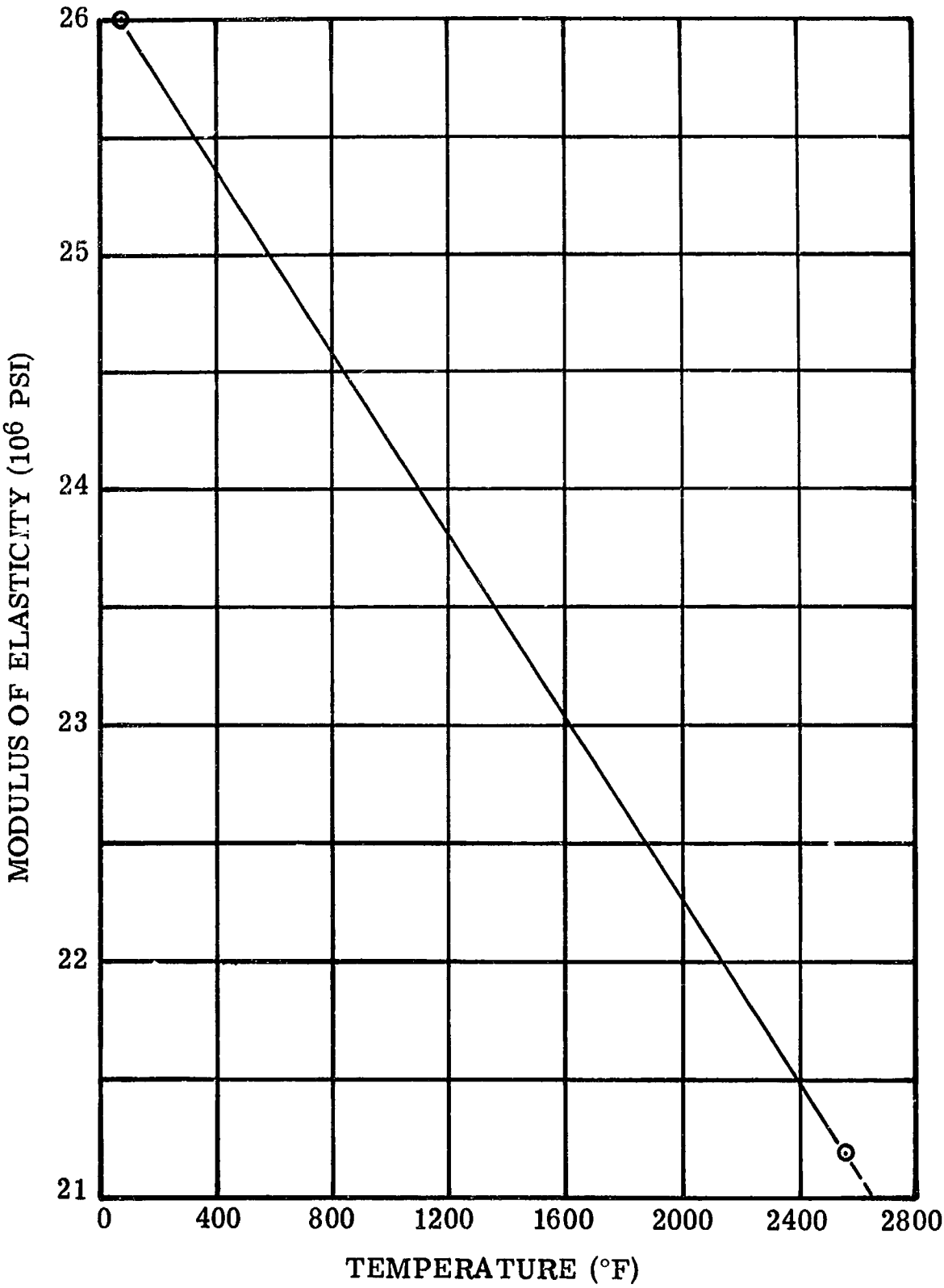


FIGURE IV-25. Modulus of Elasticity of T-111 Tantalum Base Alloy in Vacuum 5×10^{-6} torr. (Reference: LB 186)

Figure IV-25. Modulus of Elasticity - T-111 Alloy

3. Columbium Base Alloy, Cb-1Zr.

Availability: Commercial; in the form of sheet, strip, plate, bar and tubing.

Composition:
0.8 - 1.2% Zirconium
0.010% max Carbon
0.030% max Oxygen
0.0015% max Hydrogen
0.030% max Nitrogen

I. Thermophysical Properties

A. Density (77°F) 0.31 lb/in^3 8.58 gm/cc

B. Melting Point (°F) 4375 (LB 164)

C. Specific Heat

Temperature (°F)	Btu/lb-°F
77	0.052 estimated

D. Thermal Conductivity

Temperature (°F)	Btu-ft / ft^2-hr-°F
77	25.0
500	29.3
900	31.4
1600	34.5

E. Thermal Expansion (LB 91)

Temperature Range (°F)	Inch/inch-°F
77 - 500	3.54×10^{-6}
500 - 1000	3.54×10^{-6}
1000 - 1600	3.54×10^{-6}
1600 - 2600	3.54×10^{-6}

F. Electrical Resistivity

Temperature (°F)	Ohm-cm
77	17.3×10^{-6}
500	26×10^{-6}
900	34×10^{-6}
1600	48×10^{-6}

II. Mechanical Properties

A. Poisson's Ratio (77°F)

B. Tensile Properties [d] (LB 164)

		Psi
1.	At 77°F	
a.	0.2 percent offset yield strength	35,500
b.	Ultimate strength	48,000
c.	Modulus of elasticity	11.5×10^6
2.	At 500°F	
a.	0.2 percent offset yield strength	33,750
b.	Ultimate strength	47,000
c.	Modulus of elasticity	9.8×10^6
3.	At 900°F	
a.	0.2 percent offset yield strength	32,500
b.	Ultimate strength	44,500
c.	Modulus of elasticity	8.5×10^6
4.	At 1600°F	
a.	0.2 percent offset yield strength	27,500
b.	Ultimate strength	34,500
c.	Modulus of elasticity	5.9×10^6

(d) Cb-1Zr Recrystallized sheet, one hour at 2200°F, tested in vacuum.

C. Creep Properties

Curves of existing creep data on sheet material are shown in Figure II-6. Long-time creep properties in high vacuum are currently being determined on NASA Contract NAS3-2545.

III. Compatibility Properties

1. Alkali Metal

Columbium Base Alloy Cb-1Zr has excellent stability in alkali metals. Essentially no attack after 10,000 hours in potassium at 2000° F. Determined on NASA Contract NAS3-2140. Report to be published.

2. Nuclear Radiation Resistance

Figure IV-26. Thermal Conductivity - Cb-1Zr

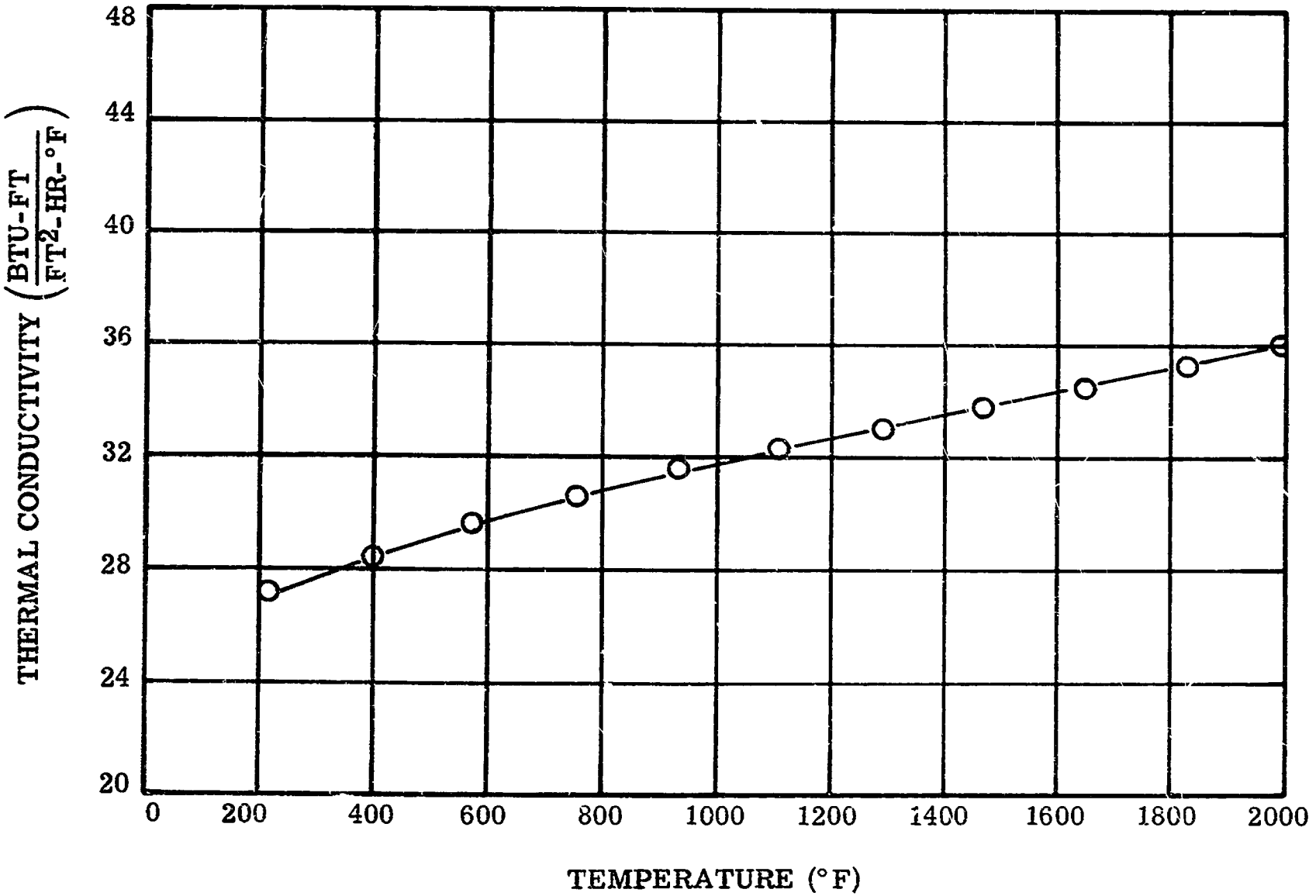


FIGURE IV-26. Thermal Conductivity of Cb-1Zr Columbium Base Alloy. (Reference: Thermal Conductivity and Electrical Resistivity of Potassium and Niobium -1 Zirconium, H. W. Deem and J. Matolich, Jr., Battelle Report 4673-T6, April 1963).

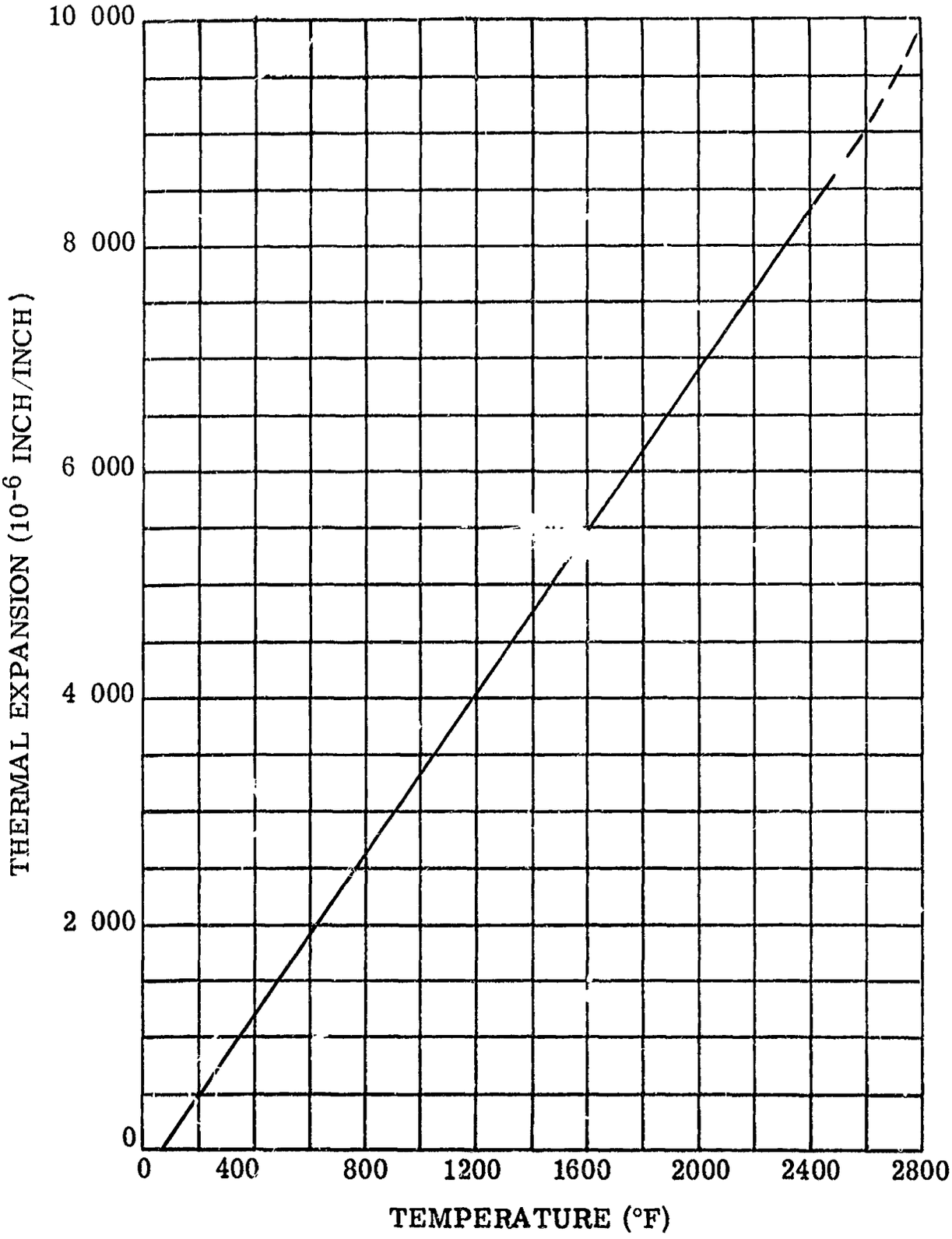


FIGURE IV-27. Thermal Expansion of Cb-1Zr Columbium Base Alloy. (Reference: LB 91)

Figure IV-27. Thermal Expansion - Cb-1Zr, Columbium Base Alloy

Figure IV-28. Electrical Resistivity - Cb-1Zr

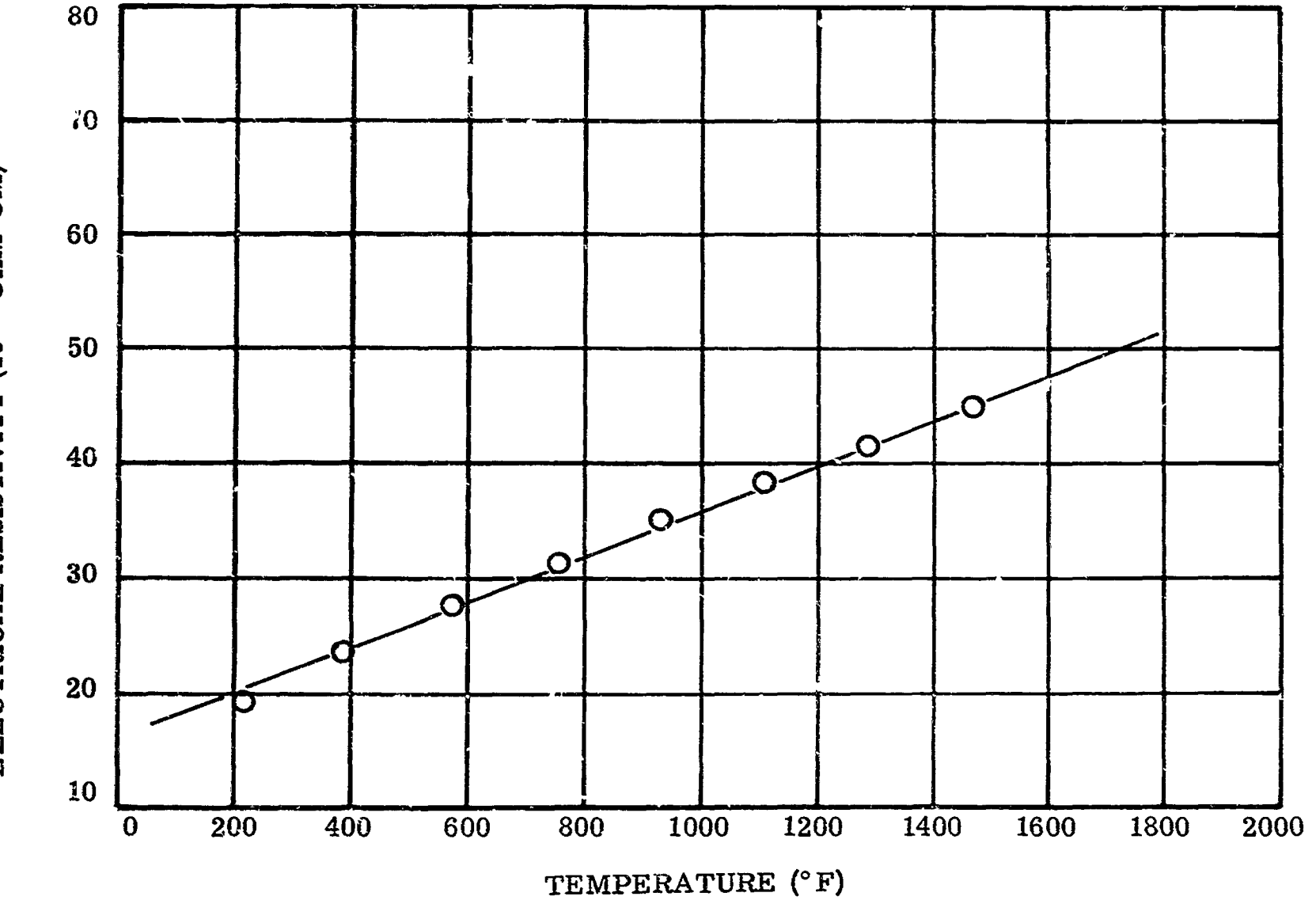


FIGURE IV-28. Electrical Resistivity of Cb-1Zr Columbium Base Alloy. (Reference: Thermal Conductivity and Electrical Resistivity of Potassium and Niobium -1 Zirconium; H. W. Deem and J. Matolich, Jr., Battelle Report 4673-T6, April 1963).

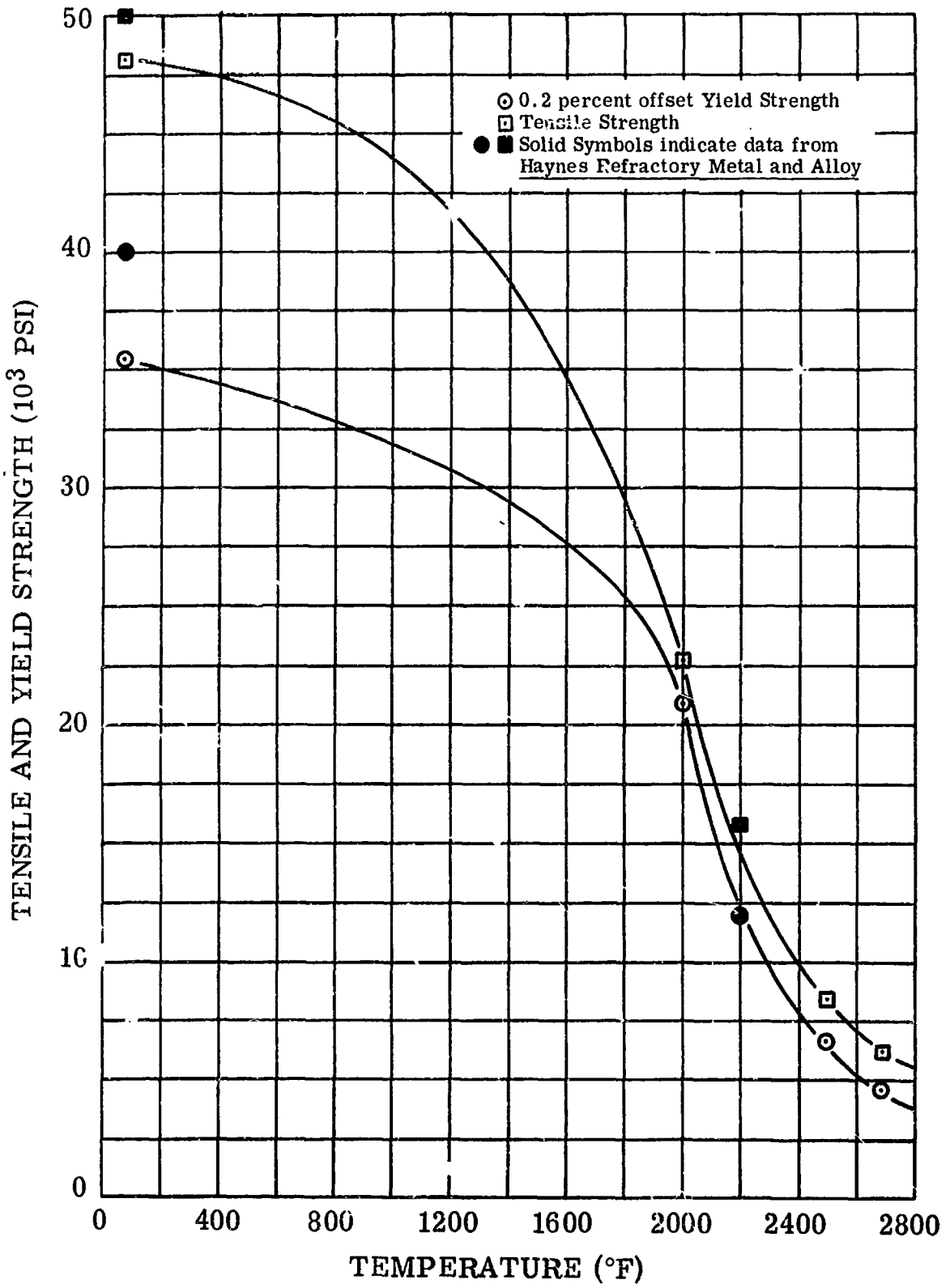


FIGURE IV-29. Tensile and Yield Strength of Cb-1Zr Columbium Base Alloy in the Recrystallized Condition. (Reference: LB: 164)

Figure IV-29. Tensile and Yield Strength - Cb-1Zr

Figure IV-30. Modulus of Elasticity - Cb-1Zr

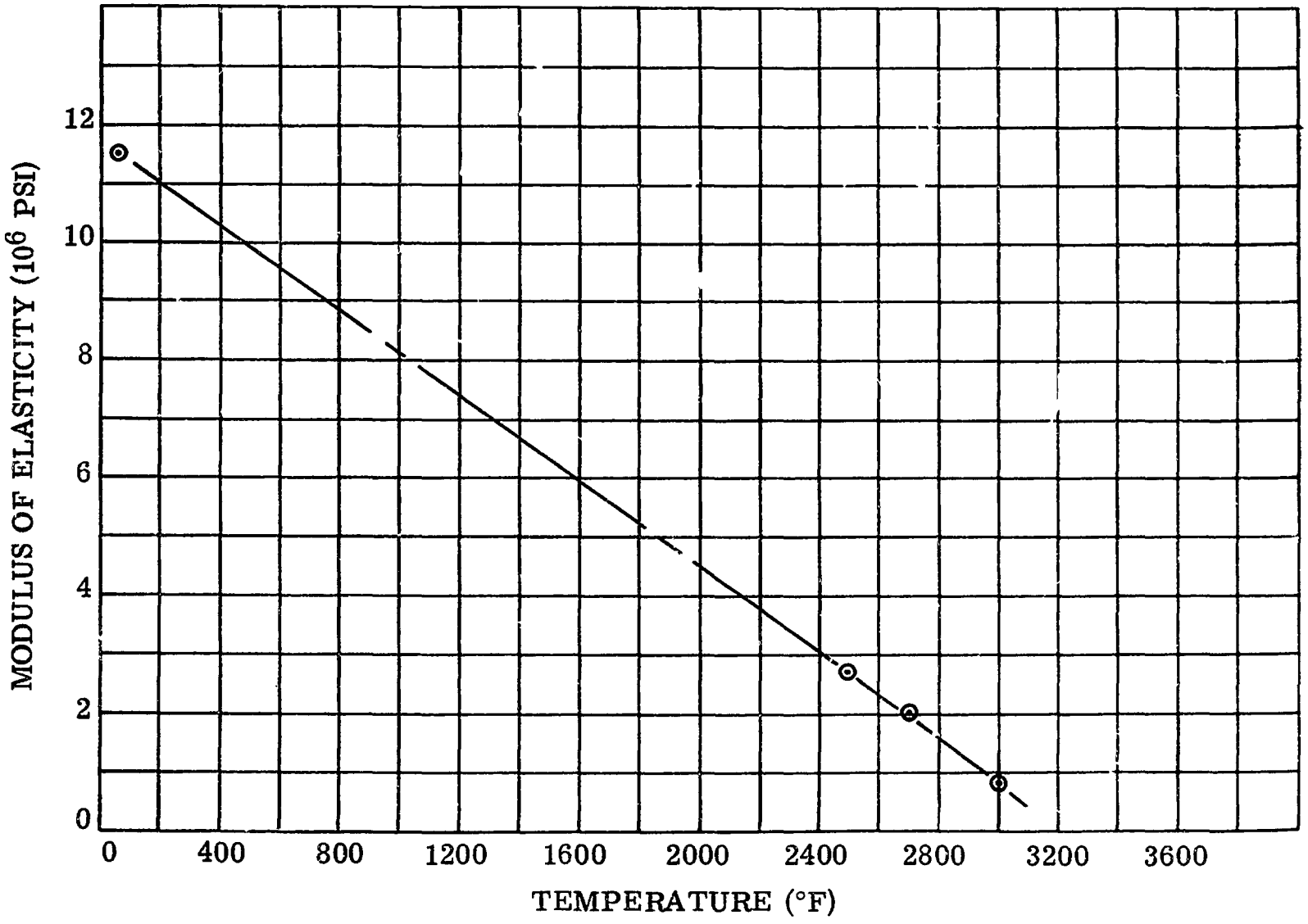


FIGURE IV-30. Modulus of Elasticity of Cb-1Zr Columbium Base Alloy. (Reference: LB 164)

SECTION V

CONCLUSIONS

1. The practicality of handling alkali metals in a high-quality vacuum-purge chamber to achieve low impurity levels in loaded capsules was demonstrated. Oxygen levels of less than 10 ppm were attained. This is the present limit of detectability by the mercury amalgamation and titration method of separation and analysis.

2. Of the ceramics which are commercially available in sizes larger than four inches in diameter, 99.8 percent BeO (Thermalox 998-Brush) is superior for alkali metal systems. There was no significant degradation detected after 500 hours at 1600°F in potassium vapor.

3. One hundred percent Al_2O_3 (Sapphire-Linde) and 99.8 percent Al_2O_3 (Lucalox-G.E.) were similarly unaffected by 500 hours exposure to potassium vapor at 1600°F.

4. Low silica (nominally less than 0.1 percent) ceramics such as Ei3-3W alumina are unsuitable at 1600°F in potassium vapor and 1000°F in NaK. The silica content of ceramic bodies for test purposes or bore seal construction must be closely controlled.

5. The (99.8 percent BeO)-(56Zr-28V-16Ti)-(Cb-1Zr) seal system represents the best of the 1600°F potassium vapor exposed systems and was also satisfactory after 500 hours in lithium at 1000°F.

6. The three active brazing alloys evaluated in depth on this program alloy rapidly with the Cb-1Zr metal member, with the result that brazing time-temperature variations tended to over-shadow the effects of the 500 hour exposure to alkali metal vapors.

7. The modulus-of-rupture and tab-peel assemblies are capable of resolving ceramic-to-metal seal strength variables as readily as the CLM 15 tensile test and drum peel test assemblies.

8. Work on tungsten base metalizing paints with thermodynamically stable secondary phases showed these compositions to be excessively brittle after sintering.

9. The susceptibility of thin-film molybdenum metalizing to solution by the nickel-base braze alloy through imperfections in the iron barrier layer has redirected the interest in this approach towards the use of thin-film metalizing as a wetting aid for active alloy brazes to ceramic. Initial tests indicated unusually high tensile strength.

10. Electroformed nickel seals between columbium 1 percent zirconium alloy and metalized ceramics, although vacuum tight, exhibited inadequate strength for load bearing applications.

APPENDIX A

BIBLIOGRAPHY

Appendix A presents a bibliography abstracted during the literature search phase of the program.

The literature search was divided into several categories; namely, ceramic gap liner materials, metal member, ceramic to metal joining techniques and brazing materials, and alkali metal corrosion technology with respect to these materials. Information was sought on physical and mechanical properties of candidate materials along with fabricability.

A division of effort on the literature survey was made. Eitel-McCullough covered the ceramic and seal materials and sealing techniques with supplements by Westinghouse. Westinghouse conducted the survey on metal end materials. The appraisal of alkali metal compatibility with bore seal materials was covered in a joint effort.

Dr. Walter H. Kohl was acting consultant with Eitel-McCullough and directed the Eimac Literature study. The literature surveyed included:

Battelle Technical Review	1961-1963
Applied Science and Technology Indexes	1958-1963
Engineering Indexes	1958-1963
ASTIA Indexes	1959-1963
Nuclear Science Abstracts	1955-1963
NASA Indexes	1958-1963
Index to Publications of the American Ceramic Society	1922-1955 and 1958-1963
Literature and Book Index of the National Association of Corrosion Engineers	1945-1961

ASM Review of Metal Literature	1960-1962
Werkstoffe and Korrosion	1961-1963

In addition to the journals and abstract bulletins listed above, the following publications were selectively reviewed.

Atomkern-Energie
Aerospace Engineering
British Ceramic Society Journal
Berichte der Deutschen Keramischen Gesellschaft
Corrosion Abstracts
Corrosion et Anticorrosion (French)
Corrosion Science
Corrosion Technology
Journal of Nuclear Energy
Journal of Nuclear Metallurgy
Liquid Metals Technology Abstract Bulletin (M.S.A. Res. Corp.)
Metallurgical Abstracts
Nukleonick (German)

The following information centers were contacted.

Thermophysical Properties Research Center (TPRC)
Electronic Properties Information Center (EPIC)
Mechanical Properties Data Center (MPDC)
Defense Metals Information Center (DMIC)
DDC Telephone Rapid Search Service

Five producers of refractory metal alloys being considered in the bore seal program were contacted to obtain current information on commercially available columbium alloys. Information obtained consisted primarily of specifications, with some data received from their technical departments. Supplier information on columbium-1% zirconium was especially meager in regard to physical properties as a function of temperature. Personal contacts were made with a number of firms now investigating various phases of ceramic-to-metal seal technology.

The bibliography was prepared for IBM punched cards. It differs from the conventional practice of presenting references, and is of added value because of the additional information which it provides. Titles of papers often deceived the reader; therefore, a "key word" or "descriptor" was defined for each reference. A code number at the end of the reference alerts the reader to the type of property information available. The code selected is as follows:

0 Not applicable to this study, but considered of sufficient general interest to warrant reporting.
1 Mechanical properties other than creep and fatigue.
2 Creep
3 Fatigue, combined loading
4 Welding, joining, fabricability
5 Magnetic properties
6 Thermo-physical properties other than electrical.
7 Electrical properties
8 Compatibility, environmental, other than liquid metal.
9 Compatibility, with liquid metal.

The punched card format required three 80 column cards to complete the reference. The format used in printing follows:

Line			
1	Bibliographic Sheet No.	Material Name of Descriptor Title Periodical, Report or Book References	Author Property Information

The property information code prints in column 70-79 of the third line and allows a standard card sorter to be used when a search for specific properties is initiated. The cards can also be computer programmed if a more complicated search is required. The second letter of the Bibliographic Sheet Number indicates the type material to which the reference pertains: LB being bore seal materials.

Three printouts are presented in this Appendix: one listing the references in numerical sequence; a second listing authors in alphabetic order; and a third listing the key words in alphabetic order.

LB1 LITHIUM,CORROSION BY HOFFMAN E E
CORROSION OF MATERIALS BY LITHIUM AT ELEVATED TEMP
ORNL 2924OUC25 OAK RIDGE NATIONAL LAB 1961 9

LB2 BERYLLIA BUDNIKOV P P BELYAYEV R A
SYSTEMS WITH BERYLLIUM OXIDE AND THEIR USE IN TECHNOLOGY
RUSSIAN PERIODICAL FTD621712 ASTIA 299870 18PP MAR 13,1963 0

LB3 SODIUM REED E L
STABILITY OF REFRACTORIES IN LIQUID METALS
JOURNAL OF AMER CERAMIC SOC V37(3)P 146-53 MARCH 1954 9

LB4 COLUMBIUM ALLOYS METCALFE A C
JOINING REFRACTORY METAL FOILS
QUAR PROG RPT 2 CONTRACT AF33(657)9442 PROJ 651G JUNE 11,1963 4

LB5 SEALS FLOYD J R
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BULLETIN AMERICAN CERAMIC SOC V42 P65-70 FEB 1963 8

LB6 SEALS PINCUS A G
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LB7 ALUMINA ANGELIDES PETER
RELIEF POLISHING OF HIGH ALUMINA CERAMICS FOR METALLOGRAPHIC STUDY
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JOURN OF AMERICAN CERAMICS SOC V45 P199 APRIL 1962 8

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CERAMIC PROPERTIES OF SAMARIUM OXIDE AND GADOLNIUM OXIDE
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LB13 EUROPIUM OXIDE CURTIS C E THARP A G
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JOURNAL AMERICAN CERAMIC SOCIETY 42(3) 151-56 MAR 1959 6

LB14 YTTRIUM OXIDE CURTIS C E
PROPERTIES OF YTTRIUM OXIDE CERAMICS
JOURNAL AMERICAN CERAMIC SOCIETY 40(8)274-78 AUG 1957 6

LB15 ALUMINA KINGERY W D
METAL CERAMIC INTERACTIONS II METAL OXIDE INTERFACE REACTION AT ELEV TEMP
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METAL CERAMIC INTERACTIONS IV ABSOLUTE MEASUREMENT OF METAL CERAMIC ENERGY
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LB17 BORIDES BRENAR L ET AL
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JOURNAL OF AMERICAN CERAMIC SOCIETY 36(6)173-79 JUNE 1951 0

LB18 COLUMBIUM PATTEE H E EVANS R M
BRAZING BONDING COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
DMIC BATTELLE MEMORIAL INSTITUTE MEMO 153 JULY 11,1962 8

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LB20 COLUMBIUM FOX C W GILLILAND R G
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LB22	MAGNESIA	FOX C W		
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	WELDING OF COLUMBIUM AND COLUMBIUM ALLOYS			
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LB28	SEALS	BENDER A		
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	REVIEW OF HIGH TEMPERATURE METAL TO CERAMIC SEALS			
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LB30	ALKALI METALS	AMATEAU M F		
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LB33 ALUMINA SHAFFER P T B WATTS R L
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LB35 SEALS ROBKIN V B
A METHOD FOR JOINING METAL TO CERAMIC
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LB39 RHENIUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB40 OSMIUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB41	CHROMIUM	TIETZ T E WILCOX B A WILSON J W	
	MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS		
	OTS PB 151855 JAN 30 1959		12 4 6 8
LB42	TANTALUM	TIETZ T E WILCOX B A WILSON J W	
	MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS		
	OTS PB 151855 JAN 30 1959		12 4 6 8
LB43	TUNGSTEN	TIETZ T E WILCOX B A WILSON J W	
	MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS		
	OTS PB 151855 JAN 30 1959		12 4 6 8
LB44	VANADIUM	TIETZ T E WILCOX B A WILSON J W	
	MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS		
	OTS PB 151855 JAN 30 1959		12 4 6 8
LB45	PLATINUM	DOUGLAS R W HOLDEN F C JAFFEE R I	
	HIGH TEMP PROP AND ALLOYING BEHAVIOR OF REFRACTORY PLATINUM METALS		
	OTS PB 161823 1961		12 678
LB46	PALLADIUM	DOUGLAS R W HOLDEN F C JAFFEE R I	
	HIGH TEMP PROP AND ALLOYING BEHAVIOR OF REFRACTORY PLATINUM METALS		
	OTS PB 161823 1961		12 678
LB47	RHODIUM	DOUGLAS R W HOLDEN F C JAFFEE R I	
	HIGH TEMP PROP AND ALLOYING BEHAVIOR OF REFRACTORY PLATINUM METALS		
	OTS PB 161823 1961		2 678
LB48	IRIDIUM	DOUGLAS R W HOLDEN F C JAFFEE R I	
	HIGH TEMP PROP AND ALLOYING BEHAVIOR OF REFRACTORY PLATINUM METALS		
	OTS PB 161823 1961		2 678
LB49	OSMIUM	DOUGLAS R W HOLDEN F C JAFFEE R I	
	HIGH TEMP PROP AND ALLOYING BEHAVIOR OF REFRACTORY PLATINUM METALS		
	OTS PB 161823 1961		6 8
LB50	RUTHENIUM	DOUGLAS R W HOLDEN F C JAFFEE R I	
	HIGH TEMP PROP AND ALLOYING BEHAVIOR OF REFRACTORY PLATINUM METALS		
	OTS PB 161823 1961		6 8

LB51 TUNGSTEN YOUNG W R
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LB52 BRAZING ALLOYS YOUNG W R
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BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALIUM TUNGSTEN GRAPHITE
BATTELLE MEMORIAL INSTITUTE DMIC MEMO 153 JULY 11 1962 4

LB55 TUNGSTEN PATTEE H E EVANS R M
BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
BATTELLE MEMORIAL INSTITUTE DMIC MEMO 153 JULY 11 1962 4

LB56 GRAPHITE PATTEE H E EVANS R M
BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
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LB57 BERYLLIA KINGERY W D
METAL CERAMIC INTERACTIONS II
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LB58 THORIA KINGERY W D
METAL CERAMIC INTERACTIONS II
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LB59 MAGNESIA KINGERY W D
METAL CERAMIC INTERACTIONS II
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LB60 TITANIA KINGERY W D
METAL CERAMIC INTERACTIONS II
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PROGRESS REPORT BRAZING OF CERAMICS
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LB62 ALUMINA LYNCH C T VAHLDIEK F W ROBINSON L B
GROWTH AND ANALYSIS OF ALUMINA WHISKERS
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LB63 WHISKERS LYNCH C T VAHLDIEK F W ROBINSON L B
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PLASTIC DEFORMATION OF CERAMIC-OXIDE SINGLE CRYSTALS II
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LB65 ALUMINA WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB66 SAPPHIRE WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB67 RUBY WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
JACS V42(5) P254-60 MAY 1959 6

LB68 MAGNESIA WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB69 THORIA WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB70 ZIRCONIA WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
JACS V42(5) P254-60 MAY 1959 6

LB71 SILICON CARBIDE WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB72 COLUMBIUM CARLSON R G MIKETTA D N FRANK R G
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EFFECTS OF LIQUID AND VAPOR CESIUM ON CONTAINMENT MATERIALS
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BIBLIOGRAPHY ON CORROSION BY LIQUID METALS
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SEAL AND INSULATOR PROBLEMS IN THERMIONIC CONVERTERS
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LB79 ALKALI METAL CESIUM BLOCK F G OGRADY J J
DEVELOPMENT OF AN AUXILIARY ELECTRODE THERMIONIC CONVERTER
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COMPARISON OF THE BRITTLE BEHAVIOR OF METALLIC AND NON-METALLIC MATERIALS
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DEVELOPMENT OF AUXILIARY ELECTRODE THERMIONIC CONVERTER
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RESISTANCE OF MATERIALS TO ATTACK BY LIQUID METALS
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DESIGN DATA STUDY FOR COATED COLUMBIUM ALLOYS
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NAVY BUWEP REPT BY WEST NOW62-0656-D DDC409896 JUNE 15 1963 1 4 67

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DEVELOPMENT OF PROCEDURES FOR SHAPE ROLLING COLUMBIUM ALLOYS
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DESIGN AND MANUFACTURING DEVELOPMENT OF LIGHTWEIGHT HEAT EXCHANGERS
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MEASUREMENTS OF THERMAL PROPERTIES
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MEASUREMENTS OF THERMAL PROPERTIES
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LB103 SAPPHIRE ARMSTRONG W M CHAKLADER A C CLARKE J F
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THERMAL EXPANSION OF MATERIALS AT -200 TO 0 DEG C
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THERMAL EXPANSION OF MATERIALS AT -200 TO 0 DEG C
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GROWTH OF BERYLLIA SINGLE CRYSTALS
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END POINT DENSITY OF HOT-PRESSED ALUMINA
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LB134 MOLYBDENUM LONG R A BANNING R D ET AL
INVESTIGATION OF EXOTHERMIC BRAZING OF REFRACTORY ALLOYS
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INVESTIGATION OF EXOTHERMIC BRAZING OF REFRACTORY ALLOYS
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LB139 SEALS DRING M L OF BENDIX CORP REDBANK N J
CERAMIC TO METAL SEALS FOR HIGH TEMPERATURE THERMIONIC CONVERTERS
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LB140 CERAMIC ADHESIVES PRATT D S SHOFFNER J E TURNER H C
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MECHANICAL AND ELECTRICAL PROPERTIES OF ALSIMAG CERAMICS
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GLASS MIGRATION MECHANISM OF CERAMIC TO METAL SEAL ADHERENCE
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GLASS TO METAL BONDING TEMP AND PRESSURE DEPENDENCE OF WETTABILITY
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CERAMIC MATERIALS FOR NUCLEAR THERMIONIC CONVERTERS
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LB146 ALUMINA STODDARD S P COWAN R E
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LB147 COLUMBIUM THOMPSON RAMO WOOLDRIDGE STAFF
DESIGN STUDY FOR COATED COLUMBIUM ALLOYS
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LB148 SEALS BRUNDIGE E L HANKS G S
CERAMIC TO METAL SEALS FOR HIGH TEMPERATURE OPERATION
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LB149 ALKALI METALS COOK W H
CORROSION RESISTANCE OF VARIOUS CERAMICS AND CERMETS IN LIQUID METALS
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LB150 ALUMINA COOK W H
CORROSION RESISTANCE OF VARIOUS CERAMICS AND CERMETS IN LIQUID METALS
ORNL 2391 JUNE 1960 9

LB151 BERYLLIA COOK W H
CORROSION RESISTANCE OF VARIOUS CERAMICS AND CERMETS IN LIQUID METALS
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LB152 RARE EARTH BODIES COOK W H
CORROSION RESISTANCE OF VARIOUS CERAMICS AND CERMETS IN LIQUID METALS
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LB153 THORIA COOK W H
CORROSION RESISTANCE OF VARIOUS CERAMICS AND CERMETS IN LIQUID METALS
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ELASTIC MODULI OF AL2O3 AND BEO TO 1200 C BY AN IMPROVED SONIC METHOD
COORS PORCELAIN CO APRIL 22, 1964 1

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ELASTIC MODULI OF AL2O3 AND BEO TO 1200 C BY AN IMPROVED SONIC METHOD
COORS PORCELAIN CO APRIL 22, 1964 1

LB156 COLUMBIUM 1 ZR JONES E S YOUNG W S
JOINING OF REFACTORY METALS BY BRAZING AND DIFFUSION BONDING
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LB157 COLUMBIUM F-48 JONES E S YOUNG W S
JOINING OF REFACTORY METALS BY BRAZING AND DIFFUSION BONDING
ASD TDR 63-88 UNDER CONTRACT 33(616)7484 TASK 735101 JAN 1963 4

LB158 TUNGSTEN JONES E S YOUNG W S
JOINING OF REFACTORY METALS BY BRAZING AND DIFFUSION BONDING
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DEVELOPMENT OF ALLOYS FOR BRAZING COLUMBIUM
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LB160 BRAZING ALLOYS FOX C W GILLILAND R G SLAUGHTER G M
DEVELOPMENT OF ALLOYS FOR BRAZING COLUMBIUM
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WELDING OF COLUMBIUM 1 ZIRCONIUM
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LB162 TANTALUM T-111 WESTINGHOUSE MATERIALS MANUFACTURING DIVISION STAFF
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LB167	BERYLLIA	KRIEGEL W W PALMOUR H III		
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LB168	MAGNESIA	KRIEGEL W W PALMOUR H III		
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LB169	ALUMINA	SPRIGGS R M MITCHELL J T VASILOS T		
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LB171	ALUMINA	COORS PORCELAIN CO STAFF		
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LB172	BERYLLIA	COORS PORCELAIN CO STAFF		
	COORS CERAMICS			
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LB173	ALUMINA	WESGO STAFF		
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TECHNICAL CERAMICS
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REFACTORY CERAMICS A MATERIAL SELECTION HANDBOOK
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LB181 COLUMBIUM ALLOY METCALFE, A.C., ET AL
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LB182 COLUMBIUM ALLOYS DUPONT STAFF
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UNPUBLISHED DATA WESTINGHOUSE ASTRONUCLEAR LABS FEB.1964 1 6

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STUDIES OF THE BRITTLE BEHAVIOR OF CERAMIC MATERIALS
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LB189 ALUMINA MATHESON R R
ADVANCEMENTS IN TECHNICAL CERAMICS
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LB190 BERYLLIA BRUSH BERYLLIUM CO
PROPERTIES OF HIGH PURITY BERYLLIA
COMMUNICATION R BROWN OF BRUSH TO NEFF AT W 7-17-64 12 67

LB191 BERYLLIA HAVELL R F
ELECTRICAL INSULATORS FOR VERY HIGH TEMPERATURES
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LB192 BERYLLIA STAFF BERYLLIUM CORP
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LB193 BERYLLIA STAFF NATIONAL BERYLLIA CORP
BERLOX
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LB200 SEALS BUYERS A G
CERAMIC METAL BONDING STABLE IN EXCESS OF 2248K
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LB201 BRAZING ALLOYS CANONICO D A SCHWARTZBART H
DEVELOPMENT OF OXIDATION AND LIQUID SODIUM RESISTANT BRAZING ALLOYS
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MATERIALS TESTED IN LITHIUM
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	SEAL AND INSULATOR PROBLEMS IN THERMIONIC CONVERTORS					
	SUMMARY REPT ARF2215-6 CONTRACT NONR-3341(00) 1962			48		
LB242	ALKALI METALS	LITMAN A P DISTEFANO J R				
	OXYGEN PARTITIONING IN POTASSIUM-OXYGEN REFRACTORY METAL SYSTEMS					
	12TH ANNUAL AEC CORROSION SYMPOSIUM MAY 20-22 1963					8
LB134	MOLYBDENUM	LONG R A BANNING R D ET AL				
	INVESTIGATION OF EXOTHERMIC BRAZING OF REFRACTORY ALLOYS					
	ASTIA AD298696 FEB 28 1963		0	4		8
LB136	COLUMBIUM	LONG R A BANNING R D ET AL				
	INVESTIGATION OF EXOTHERMIC BRAZING OF REFRACTORY ALLOYS					
	ASTIA AD298696 FEB 28 1963		0	4		8
LB135	TUNGSTEN	LONG R A BANNING R D ET AL				
	INVESTIGATION OF EXOTHERMIC BRAZING OF REFRACTORY ALLOYS					
	ASTIA AD298696 FEB 28 1963		0	4		8

LB235 ALKALI METALS LUNDEN A
RESISTIVITY OF VARIOUS MATERIALS TO ATTACK BY MOLTEN SALT AND METALS
CORROSION SCIENCE V1 NO 1 P62-4 AUGUST 1961 9

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GROWTH AND ANALYSIS OF ALUMINA WHISKERS
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GROWTH AND ANALYSIS OF ALUMINA WHISKERS
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FUNDAMENTALS OF LIQUID METAL CORROSION
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STUDY OF THE TETRABORIDES OF URANIUM AND THORIUM
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DETERMINATION OF CARBON IN SODIUM PROPERTIES OF MTLS EXPOSED TO 1200F NA
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LB116 ALUMINA MC CLELLAND J D ZEHMS E H
END POINT DENSITY OF HOT-PRESSED ALUMINA
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STATIC SODIUM CORROSION TESTS OF CERAMIC MATERIALS
KAPL-M-LRM-2 CONTRACT W-31-109-ENG-52 JULY 6 1951 9

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STATIC SODIUM CORROSION TESTS OF CERAMIC MATERIALS
KNOLLS ATOMIC POWER LAB KAPL-M-LRM-2 CONTW-31-109 JULY6 1951 8

LB217 CERAMICS MCCREIGHT L R
STATIC SODIUM CORROSION TESTS OF CERAMIC MATERIALS
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LB312 ALKALI METALS MCKEE M STEINMETZ H
SUMMARY OF LIQUID METALS ACTIVITIES AT UNITED NUCLEAR
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POLISHING AND ETCHING TECHNIQUES FOR DENSE ALUMINA
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JOINING REFRACTORY METAL FOILS
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DIFFUSION BONDING OF REFRACTORY METALS
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LB32 ALUMINA MILLS E J
THERMAL SHOCK RESISTANT PROPERTIES OF CERAMICS
FINAL RPT DA36-039SC85294 BATTELLE INSTITUTE AD262165 4-30-61 6

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DETERMINATION OF TRACES OF OXYGEN IN METALLIC SODIUM
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PROGRESS REPT ON DISSOLUTION AND SOLUBILITY OF METALS IN LITHIUM
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THE SYSTEM COPPER OXIDE ALUMINA
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LB126 MAGNESIA NIELSON T H LEIPOLD M H
THERMAL EXPANSION IN AIR OF CERAMIC OXIDES TO 2200 DEG C
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HIGH TEMP CIRCUIT FOR TESTING STABILITY, CORROSION RESIST OF CONST MTLS
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REACTION OF CONSTRUCTION MATERIALS WITH LIQUID METALS
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DECARBURIZATION OF IRON NICKEL COBALT GLASS SEALING ALLOY
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PROPERTIES OF PYROLYTIC GRAPHITE
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INVESTIGATION OF METAL CERAMIC COMPOSITIONS FOR HIGH TEMP APPLICATIONS
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BRAZING BONDING COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
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BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
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BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
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BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALIUM TUNGSTEN GRAPHITE
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BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
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BRAZING AND BONDING OF COLUMBIUM MOLYBDENUM TANTALUM TUNGSTEN GRAPHITE
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DISTILLATION METHOD FOR DETERMINATION OF SODIUM OXIDE IN NAK
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SELECTING HIGH TEMP BRAZING ALLOYS NI-BASE MATERIALS FOR SERVICE TO 2200F
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LIQUID METAL INVESTIGATION
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ALKALI METALS BOILING AND CONDENSING INVESTIGATIONS
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LIQUID METAL INVESTIGATIONS
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DEVELOPMENT OF ULTRA REFRACTORY MATERIALS
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DETERMINATING O2 IN NA AND NAK BY THE BUTYL BROMIDE METHOD
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IMPURITIES IN A LIQUID METAL COOLANT EFFECT ON FUEL ELEMENT CANNING MTLS
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LB252 COLUMBIUM SLAUGHTER G M
JOINING OF EXOTIC MATERIALS
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LB253 ALUMINA SLAUGHTER G M
JOINING OF EXOTIC MATERIALS
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JOINING OF EXOTIC MATERIALS
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DEVELOPMENT HIGH SENSITIVITY ANALYTICAL METHOD FOR OXYGEN IN SODIUM METAL
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LB292 SODIUM STEINMETZ H
EXPERIMENTAL DETERMINATION OF CONTAMINANTS IN SODIUM
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LB293 SODIUM STEINMETZ H MINUSHKIN B
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LB294 SODIUM STEINMETZ H
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LB305 LITHIUM SAX H I STEINMETZ H
DETERMINATION OF OXYGEN IN LITHIUM METAL
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LB145	SEALS	STODDARD S P COWAN R E			
	CERAMIC MATERIALS FOR NUCLEAR THERMIONIC CONVERTERS				
	PRIVATE COMMUNICATION LAMS MAY 1963			4	89
LB146	ALUMINA	STODDARD S P COWAN R E			
	CERAMIC MATERIALS FOR NUCLEAR THERMIONIC CONVERTERS				
	PRIVATE COMMUNICATION LAMS MAY 1963			4	
LB271	ALKALI METALS	STRAHL H			
	A DEVICE FOR CONTINUOUS DETECTION OF H2 IN NA				
	NAA SR 6986 CONT AT(11-1)GEN 8 MAY 31 1962				8
LB263	NAK	SUTHERLAND D SMITH A W			
	SOME PROPERTIES OF THIN OXIDE FILMS ON SODIUM POTASSIUM ALLOY SURFACES				
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LB131	TANTALUM	SYMOND J			
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LB229	ALKALI METALS	TARPINIAN M			
	EFFECT OF MOLTEN SODIUM ON THERMAL INSULATION SPECIMENS				
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LB230	CERAMIC MATERIALS	TARPINIAN M			
	EFFECT OF MOLTEN SODIUM ON THERMAL INSULATION SPECIMENS				
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THERMAL CONDUCTIVITY OF TITANIUM CARBIDE AT HIGH TEMP
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THE CONTAINMENT OF LIQUID-METAL FUELS AT 500 TO 1000 DEG C
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LB147 COLUMBIUM THOMPSON RAMO WOOLDRIDGE STAFF
DESIGN STUDY FOR COATED COLUMBIUM ALLOYS
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LB38 COLUMBIUM TIETZ T E WILCOX B A WILSON J W
MECHANICAL PROPERTIES AND OXIDATION RESISTANCE OF CERTAIN REFRAC METALS
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LB39 RHENIUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB40 OSMIUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB41 CHROMIUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB42 TANTALUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB43 TUNGSTEN TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB44 VANADIUM TIETZ T E WILCOX B A WILSON J W
MECH PROP AND OXIDATION RESISTANCE OF CERTAIN REFRACTORY METALS
OTS PB 151855 JAN 30 1959 12 4 6 8

LB240 LITHIUM TYZACK C LONGDON P B
THE OXIDATION OF LITHIUM
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COMPRESSIVE CREEP OF POLYCRYSTALLINE BERYLLIUM OXIDE
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PRESSURE SINTERING MECHANISMS AND MICROSTRUCTURES FOR ALUMINA AND MAGNESIA
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PRESSURE SINTERING MECHANISMS AND MICROSTRUCTURES FOR ALUMINA AND MAGNESIA
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LB65 ALUMINA WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB67 RUBY WACHTMAN J B LAIN D A
YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP
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LB68	MAGNESIA	WACHTMAN J B LAIN D A			
	YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP				
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LB69	THORIA	WACHTMAN J B LAIN D A			
	YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP				
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LB70	ZIRCONIA	WACHTMAN J B LAIN D A			
	YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP				
	JACS V42(5) P254-60 MAY 1959				6
LB71	SILICON CARBIDE	WACHTMAN J B LAIN D A			
	YOUNGS MODULUS OF VARIOUS REFRACTORY MATERIALS AS FUNCTION OF TEMP				
	JACS V42(5) P254-60 MAY 1959				6
LB164	COLUMBIUM ALLOYS	WAH CHANG STAFF			
	COLUMBIUM AND TANTALUM BASE ALLOYS FOR STRUCTURAL NUCLEAR APPLICATION				
	WAH CHANG TECHNICAL BROCHURE VI REV 1 MAY 1962			12	6
LB213	ALKALI METALS	WALLHELM C			
	PREVENTING CORROSION BY LIQUID METALS				
	MATERIALS IN DESIGN ENGRG V58 P97-9 NOV 1963				89
LB114	ALUMINA	WARSHAW S I NORTON F H			
	DEFORMATION BEHAVIOR OF POLYCRYSTALLINE ALUMINUM OXIDE				
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LB228	LITHIUM	WEATHERFORD W D JOHNSTON R K ET AL			
	CONTAMINATION EFFECTS ON LIQUID RUBIDIUM AND LIQUID LITHIUM SYSTEMS				
	SOUTHWEST RESEARCH INST FINAL REPT AF33(657)8657 1963				8
LB227	BERYLLIA	WELLS W M CLINE C F			
	THERMAL STRESS FRACTURE CHARACTERISTICS OF BEO				
	LAWRENCE RADIATION LAB UCRL-7430 JULY 15 1963			1	
LB173	ALUMINA	WESGO STAFF			
	ALUMINA CERAMICS				
	WESTERN GOLD AND PLATINUM BROCHURE C-115			1	67

LB162 TANTALUM T-111 WESTINGHOUSE MATERIALS MANUFACTURING DIVISION STAFF
T-111 TANTALUM BASE ALLOY
WESTINGHOUSE MMD TECHNICAL DATA MARCH 1963 12 67

LB186 TANTALUM ALLOY T-111 WESTINGHOUSE ANL STAFF
ELASTIC MODULUS AND THERMAL EXPANSION OF TANTALUM T-111 ALLOY
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LB313 SEALS STAFF
SPUR GENERATOR DEVEL PERIOD MAY-JULY 1964
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LB89 COLUMBIUM ALLOYS WHITE G K CORTES F R
DEVELOPMENT OF PROCEDURES FOR SHAPE ROLLING COLUMBIUM ALLOYS
ASTIA AD409982 UNDER AF33(657)10831 JUNE 1963 12 4 6

LB308 ALKALI METALS WHITE J C
ALKALI METAL ANALYTICAL PROGRAM AT ORNL DETERMINATION OF OXYGEN IN K
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LB272 ALKALI METALS WHITE J C
DETERMINATION OF OXYGEN IN SODIUM AND NAK BY DISTILLATION METHOD
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JOINING OF REFACTORY METALS BY BRAZING AND DIFFUSION BONDING
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