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WADC TECHNICAL REPORT 54-173
PART 2

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ALUMINA-BASE CERMETS

CHARLES A. HAUCK
EARNEST W. DEADWYLER
THOMAS S. SHEVLIN

THE OHIO STATE UNIVERSITY RESEARCH FOUNDATION

MARCH 1956

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WRIGHT AIR DEVELOPMENT CENTER

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MARCH 1956

AERONAUTICAL RESEARCH LABORATORY
CONTRACT AF 33(616)-472
TASK 70634
PROJECT 7350

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report was prepared by Charles A. Hauck, Earnest W. Deadwyler, and Thomas S. Shevlin of the Ohio State University Research Foundation, Columbus, Ohio under the general supervision of J. O. Everhart, Supervisor of Ceramic Research. The report summarizes work during the period 1 April 1954 to 1 February 1955 in which research was continued on alumina-base cermets. This work is being continued and further reports will be issued as warranted by the progress.

This work was accomplished under Contract AF 33(616)-472. It was administered under the direction of the Aeronautical Research Laboratory with Murray A. Schwartz as project engineer, and is identified under Task No. 70634, "High Temperature and High Stress Characteristics of Ceramics and Cermets", Project No. 7350, "Ceramic and Cermet Materials."

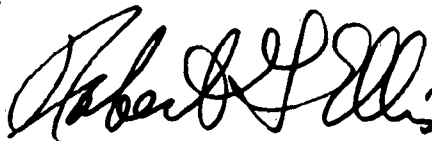
ABSTRACT

Studies were conducted on five new alumina-base cermets, the metal content ranging from 50 to 95% by volume. Alloys investigated include: 80Ni-20Cr, 65Co-30Cr-5Mo, 66Ni-18Cr-16Fe, 76Cr-24Ti, and an 18-8 type stainless steel. Details include fabrication techniques and test results. Wettability studies of alumina by various alloys are described. Flame holder test segments were fabricated and the techniques described.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



ROBERT G. ELLIS, Lt. Colonel, USAF,
Asst. Chief, Aeronautical Research Laboratory
Directorate of Research

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I. INTRODUCTION

The major portion of the work described in this report is concerned with the development of alumina-base cermets in which the Al_2O_3 comprises 50% and less of the volume of the cermet. The object of this study is the development of cermet compositions that will possess a high level of impact resistance in addition to adequate high-temperature strength and resistance to oxidation.

The alloys chosen for the study are of a somewhat ductile nature and are the same as, or similar to, alloys that have been developed for high-temperature use. The cermet compositions being investigated contain as the metal phases the alloys: 80Ni-20Cr, 65Co-30Cr-5Mo, 66Ni-18Cr-16Fe, 76Cr-24Ti, and an 18-8 type stainless steel. These alloys were combined with Al_2O_3 in five proportions ranging from 50 to 100 volume percent of metal.

At the outset of this investigation it was planned to prepare specimens of the compositions and evaluate them with respect to impact strength and high-temperature properties. After a preliminary firing study on all the compositions, it became evident that the problem of devising a method of sintering dense specimens was greater than anticipated. This was particularly the case with the compositions containing the larger amounts of Al_2O_3 .

Much of the subsequent effort was directed toward a means of producing nonporous cermet specimens suitable for testing.

The problem of obtaining a dense mixture of Al_2O_3 and metal is recognized as being a function of the wettability of the Al_2O_3 by the metal. Previous studies¹ on the wetting of Al_2O_3 by the metals Cr, Co, Ni, and Fe have shown that there is no inherent tendency for any of these metals to wet alumina but that it may be possible to effect a bond between the two phases through thermochemical equilibria involving the formation of an intermediate compound between the Al_2O_3 and the oxide of the metal phase. Later studies on Cr- Al_2O_3 compositions² have indicated that bonding takes place through a slight oxidation of the Cr and the subsequent solid solution of the Cr_2O_3 in the Al_2O_3 . However, in the case of the chromium-bearing alloys being used in the present investigation, the mechanism of bonding is complicated by the widely different oxidation potentials of the constituent metals in the alloys and also because of sintering temperature limitations caused by the relatively low melting temperature of the alloys.

A major portion of the present effort has been expended on methods of producing nonporous specimens by modifications of both the ceramic and metal phases, sintering conditions, and batch preparation.

NOTE - This Technical Report was released by the author for publication in March 1955.

The results of the many and varied attempts to produce a dense body are for the most part negative. In some cases an improvement was noted, but a satisfactory method has not yet been devised.

It was found that a dense body could be sintered without resorting to composition modification where the cermet contains less than 15% by volume of Al_2O_3 . A limited amount of physical property data was obtained on the compositions containing stainless steel and small proportions of Al_2O_3 .

In cooperation with the National Bureau of Standards a project was undertaken to fabricate a number of flameholders. The cermet used for this purpose was the previously developed A-355.6-G composition containing 34.4% Al_2O_3 and 65.6% 80Cr-20Mo alloy. This cermet has demonstrated excellent thermal-shock resistance in tests on nozzle diaphragm blades and simplified afterburner test specimens. Therefore it may prove to be a satisfactory material in flameholder applications where thermal shock is of prime importance.

II. MATERIALS

The cermet compositions described in this report were compounded using the following materials:

Alumina: Corundum form, 0.05 maximum soda, Alcoa Grade T-61, milled to minus 25 microns, acid leached, furnished by the Champion Spark Plug Company.

Chromium: Electrolytic, 99% minimum chromium content, minus 325 mesh, furnished by Electro Metallurgical Company.

Stainless Steel: 18-8 type, 302 B, minus 325 mesh, furnished by Vanadium Alloys Steel Company.

80 nickel - 20 chromium alloy: minus 325 mesh, furnished by Metal Hydrides Inc.

Nickel: annealed, 98% minimum nickel content, minus 325 mesh, furnished by Charles Hardy Inc.

Chromium - Titanium alloy: 76% chromium 24% titanium, furnished by Metal Hydrides Inc.

Cobalt: 97.5% minimum cobalt content, minus 300 mesh, furnished by Charles Hardy Inc.

Iron: carbonyl, type E, 97.9% minimum iron content, minus 325 mesh, furnished by Charles Hardy Inc.

Molybdenum: hydrogen reduced, 99.75% minimum molybdenum content, minus 325 mesh, furnished by Charles Hardy Inc.

Titanium Carbide: 0.05 maximum carbon, minus 325 mesh, furnished by the Norton Company.

TiO₂: Heavy grade, 200 mesh, furnished by Titanium Alloys Manufacturing Division of the National Lead Company.

Silicon: Minus 100 mesh plus 325 mesh powder, furnished by Charles Hardy Inc.

Aluminum: Grade HA, minus 325 mesh, furnished by Charles Hardy Inc.

Cr₂O₃: Reagent grade, furnished by The Coleman and Bell Company.

Two types of binder-lubricants were used in the forming operations. At the beginning of the investigation Carbowax "4000," furnished by the Carbide and Carbon Chemicals Company, was used but was later abandoned in favor of camphor, U.S.P., furnished by DuPont.

III. COMPOSITIONS

A list of the compositions investigated under this contract is given in Table 1. The "21-.-" series of compositions was prepared originally using elemental metal powders but was later made with alloy powder when it became available. Elemental metal powders were employed in the "22-.-" and "23-.-" series of compositions. The "6-.-" and "20-.-" series contain alloyed metal powder. There was no development work done on the A-355.6-G composition during this period. This cermet, used for the fabrication of flameholders, was developed during a prior research period.

Table 1

Batch Compositions

Designation	Component	Weight	Volume %
A-65.1-G	T-61 Alumina	39	50
	76Cr-24Ti Alloy	61	50
A-67.2-G	T-61 Alumina	17.6	25
	76Cr-24Ti Alloy	82.4	75

Table 1 (continued)

Designation	Component	Weight %	Volume %
A-68.1-G	T-61 Alumina	8.7	13
	76Cr-24Ti Alloy	91.3	87
69.0-G	76Cr-24Ti Alloy	100	100
A-205.6-G	T-61 Alumina	33.6	50
	302B Stainless Steel	66.4	50
A-207.6-G	T-61 Alumina	14.4	25
	302B Stainless Steel	85.6	75
A-208.1-G	T-61 Alumina	8.2	15
	302B Stainless Steel	91.8	85
A-208.7-G	T-61 Alumina	2.6	5
	302B Stainless Steel	97.4	95
209.0-G	302B Stainless Steel	100	100
A-215.7-G	T-61 Alumina	32.3	50
	80Ni-20Cr	67.7	50
A-217.6-G	T-61 Alumina	13.7	25
	80Ni-20Cr	86.3	75
A-218.2-G	T-61 Alumina	7.8	15
	80Ni-20Cr	92.2	85
A-218.7-G	T-61 Alumina	2.4	5
	80Ni-20Cr	97.6	95
219.0-G	80Ni-20Cr	100	100
A-225.7-G	T-61 Alumina	32.5	50
	65Co-30Cr-5Mo	67.5	50
A-227.6-G	T-61 Alumina	13.9	25
	65Co-30Cr-5Mo	86.1	75
A-228.2-G	T-61 Alumina	7.8	15
	65Co-30Cr-5Mo	92.2	85
A-228.7-G	T-61 Alumina	2.5	5
	65Co-30Cr-5Mo	97.5	95

Table 1 (continued)

Designation	Component	Weight %	Volume %
229.0-G	65Co-30Cr-5Mo	100	100
A-235.7-G	T-61 Alumina	32.7	50
	66Ni-18Cr-16Fe	67.3	50
A-237.6-G	T-61 Alumina	14.0	25
	66Ni-18Cr-16Fe	86	75
A-238.2-G	T-61 Alumina	7.9	15
	66Ni-18Cr-16Fe	92.1	85
A-238.7-G	T-61 Alumina	2.5	5
	66Ni-18Cr-16Fe	97.5	95
239.0-G	66Ni-18Cr-16Fe	100	100
A-355.6-G	T-61 Alumina	34.4	50
	80Cr-20Mo Alloy	65.6	50

In addition to the above compositions a number of modifications were made on the composition containing 50% Al_2O_3 and 50% 80Ni-20Cr alloy by volume. These modifications were in the nature of attempts to improve the wetting characteristics of the Al_2O_3 by the metal. The 80Ni-20Cr alloy was chosen for this study because it was available as alloy powder. It is believed that a means of improving the wetting between this alloy and Al_2O_3 would also be applicable to other Ni-base and Co-base alloys.

The modifications and their designations are given in Table 2.

Table 2

Modifications to the A-215.7-G Composition

Designation	Modification
B-1-X	A batch addition of 5% TiC and 5% Mo.
B-2-X	A batch addition of 3% Cr_2O_3 .

Table 2 (continued)

Designation	Modification
B-3-X	A 95% Al_2O_3 + 3% Bentonite + 1% MgO + 1% CaO mixture substituted for the Al_2O_3 .
B-4-X	A mixture containing 10% $\text{NiO} \cdot \text{Al}_2\text{O}_3$ + 90% Al_2O_3 (mole basis) substituted for the Al_2O_3 .
B-5-X	A prereacted mixture of 10% TiC + 90% Al_2O_3 substituted for the Al_2O_3 .
B-6-X	A mixture of 16% TiO_2 + 84% Al_2O_3 substituted for the Al_2O_3 .
B-7-X	A batch addition of 5% Si .
B-8-X	A batch addition of 1% TiC .
B-9-X	A batch addition of 5% TiC .
B-10	76Cr - 24Ti alloy substituted for the 20% Cr in the 80Ni-20Cr component.

IV. BATCH PREPARATION

All compositions were prepared in 500-gram batches by wet milling in one-quart capacity steel mills using approximately 12 pounds of cobalt-bonded tungsten carbide slugs as the grinding medium. Methyl alcohol was used as the vehicle. After milling, the batches were air dried and screened through a 42-mesh screen.

The use of a binder-lubricant was found to be necessary with all the compositions in order to provide sufficient handling strength and eliminate pressure cracks in the bar specimens formed in a steel die. For this purpose two types of binders were employed. The first was Carbowax "4000" which was melted in a mortar and hot mixed into the body. The waxed batch was then granulated through a 42-mesh screen. The other binder used was camphor dissolved in ether, the liquid being mixed into the batch material until the ether evaporated. The carbowax "4000" was used at the beginning of the investigation, but was later discontinued in favor of camphor. The advantages in using the camphor are the ease of mixing it into

the body and the faster and safer removal from the pressed specimens. There was no apparent difference in the properties of the compositions caused by the binder used and no attempt is made to differentiate between the two in regard to the results given in this report.

The $\text{NiO} \cdot \text{Al}_2\text{O}_3$ - Al_2O_3 mixture used in the composition B-4-x was prepared by mixing Ni and Al_2O_3 together and firing the mixture to 2600°F in an oxidizing atmosphere. The batch was compounded so that the fired mixture would contain 10 $\text{NiO} \cdot \text{Al}_2\text{O}_3$ + 90 Al_2O_3 calculated on a molecular basis. Microscopic examination of the fired mixture revealed no traces of unoxidized metal and the formation of the $\text{NiO} \cdot \text{Al}_2\text{O}_3$ spinel was apparently complete.

The 10% TiC + 90% Al_2O_3 mixture employed in composition B-5-x was prereacted by firing to 3000°F in vacuo in a graphite-susceptor induction furnace.

In an attempt to effect prealloying of the metal phase in the compositions made from elemental metal powders the compositions A-225.7-G and A-235.7-G were prepared in the usual manner and pressed into compacts $1\frac{1}{2}$ inches in diameter and approximately one-half inch thick. The compacts were then fired to 2450°F in hydrogen and held at this temperature for 12 hours. At this temperature the compacts remained porous but did not sweat out. The compacts were then broken down to approximately 4-mesh pieces in a steel mortar. The material was then further reduced to minus 200 mesh in a type SH Mikro-Pulverizer. The pulverized material was used in preparing specimens.

V. FORMING

BAR SPECIMENS

Bar specimens were formed by pressing the 42-mesh material containing a binder in a strip-case steel die having an opening 4.5 inches long by 0.5 inch wide. The die was charged and struck off, the thickness of the bar being controlled by the volume of the charge. After an initial low-pressure application of approximately 1000 psi, die supports were removed, allowing the case to float and both punches to close on the charge from above and below. A pressure of 22,000 psi was normally employed to provide handling strength without pressure cracking.

After this initial forming the bars were placed in rubber envelopes, evacuated to approximately 1 mm of mercury, and repressed hydrostatically at 35,000 psi.

Difficulty was encountered in the forming of specimens from the compositions containing stainless steel. When Carbowax "4000" was used as the binder, the specimens were broken up by the hydrostatic pressing operation and when the hydrostatic pressing was eliminated the bars did not possess sufficient handling strength after the binder had been removed. These difficulties were believed to be caused by an unfavorable grain-size distribution and/or the spherical shape of the stainless steel particles. Bars possessing adequate green strength were obtained by milling the batches for 100 hours instead of the usual 25 or 50 hours, using 6% camphor as the binder, and die pressing the bars at 44,000 psi followed by hydrostatic pressing at 35,000 psi.

There appeared to be a reaction between the methanol and the stainless steel, and the pressure built up in the mill had to be relieved periodically.

The specimens containing Carbowax "4000" were dewaxed at 140°C on a 48-hour schedule allowing 12 hours at maximum temperature. The removal of camphor from the specimens was accomplished by placing them in an oven at 100°C overnight.

TENSILE SPECIMENS

Rod specimens were formed by direct hydrostatic pressing of granulated batch material. A thin-walled rubber tube held within an outer, perforated, brass tube was filled with powder while the assembly was being vibrated. One end of the tube was closed with a movable, cylindrical rubber stopper before filling; the other end was closed with a shouldered rubber stopper after filling. The filled tube was then evacuated through a hypodermic needle inserted through the shouldered stopper. After evacuation the needle was withdrawn, the hole covered with grease, and the assembly subjected to 35,000 psi hydrostatic pressure. This method of forming requires no temporary binder to be added to the batch material.

The pressed rods were fired to maturity and machined down to rods 8 inches long by 0.375-inch diameter, gradually necked to a minimum diameter of 0.250 inch in the central 1.5 inches of the length. In order to keep within safe loading pressures on the tensile test apparatus the tensile rods tested at room temperature were necked to a minimum diameter of 0.150 inch.

VI. FIRING

FURNACES

The 2-inch and 6-inch I.D. molybdenum wire furnaces described in O.S.U. Reports^{4,5} were employed in the bulk of the

firings. The furnaces consist of a molybdenum-wound alumina tube appropriately insulated and contained within a gastight steel case. Several exploratory firings were made in a graphite-susceptor induction furnace described in O.S.U. Report No. 23.

ATMOSPHERE

Firings were made in atmospheres of purified hydrogen, wet hydrogen, wet helium, and vacuum. The hydrogen was purified by passing over copper turnings at 1000°F, through a tower of activated alumina, and over calcium chips at 600°F. Titanium hydride was also used as a getter within the firing chamber during firings in which purified hydrogen was employed. The dew point of the hydrogen purified in this manner is less than -90°F. The wet hydrogen was obtained by passing the H₂ through water that was maintained at 32°F, before entering the furnace. The wet helium was passed through water at room temperature. The measured dew points of the gases passed through water is the temperature of the water, indicating saturation of the gas and providing a convenient method for controlling the degree of oxidation desired. In the early stages of the firing cycle, the furnace was flushed three times with the gas being used for that firing.

The firings in the graphite-susceptor induction furnace were in vacuum with an absolute pressure of less than 100 microns maintained throughout the firing cycle.

TEMPERATURE

The temperature in each case was raised to 150° to 200°F below the final temperature in twelve hours and held at this temperature for 12 hours, then increased to the top temperature and soaked for a period of 1.5 or 6 hours. Temperature measurements were made with an optical pyrometer.

The optimum firing temperature for the majority of the compositions has not been determined accurately at the present time. On the basis of density and modulus of rupture results the optimum sintering temperature for the 209.0-G and A-20817-G compositions was found to be 2500°F and for A-208.1-G 2550°F. These data are shown as a function of firing temperature in Figure 1.

Compositions containing 25 and 50% Al₂O₃ by volume invariably exuded metal when fired to 2600°F, but at this temperature very little consolidation of the body occurred. When fired to a temperature just below the temperature at which metal was lost, the specimens were weak and porous.

VII. TESTING AND RESULTS

FIRING SHRINKAGE

Linear firing shrinkage was determined as the ratio of the change in length in firing, to the unfired length. Many of the firings resulted in specimens in which a portion of the metal had sweated out or which were overfired to such an extent that the bars were deformed by fluid flow of the metal. Shrinkage measurements were made only where there was no loss of metal and no deformation.

MODULUS OF RUPTURE

The strength of the sintered bars in cross-bending was determined wherever possible. The bars were broken or bent on a 3-inch span using ball supports on both ends to minimize torsion effects. The span-to-depth ratio was approximately 25 to 1.

In the compositions containing 15, 5, and 0 percent Al_2O_3 by volume, fracture was preceded by considerable plastic deformation and in many cases fracture did not occur. Since the flexure formula is applicable only to brittle materials where rupture occurs at the end of the elastic range of deformation, the values given in the subsequent tables are not a true indication of strength or load carrying ability. Where it is noted in the tables that the specimens did not break, the values given are those calculated, using the flexure formula, for a stress that produced a 0.25-inch deflection in a bar supported on a 3-inch span and loaded centrally.

The data given in Tables 3, 4, and 5 relate the variation in modulus of rupture and firing shrinkage to firing temperature and time at temperature for the "21.-", "22.-" and "23.-" series of compositions. The compositions that were prefired to effect alloying of the metal phase, as explained in the section BATCH PREPARATION, have the number "2" following the designation. The A-215.7-G composition was prepared using alloyed and nonalloyed metal powder. The "x" following the designation denotes the use of the alloyed metal powder in the composition.

A more extensive firing study was made on the Al_2O_3 -stainless steel compositions. In Table 6 the variation of modulus of rupture and firing shrinkage is shown as related to maturing temperature. These data are given graphically in Figure 1.

Table 3

Firing and Strength Data on Al_2O_3 - (80Ni-20Cr)
Compositions

Compo- sition	Firing temp., °F	Time at temp., hrs.	Atmos- phere	Linear shrinkage, %	Modulus of rupture, psi
A-215.7-G	2400	1.5	Dry H_2	0.3	5,100
	2500	1.5	Dry H_2	1.4	13,100
	2550	6.0	Dry H_2	1	15,800
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
A-215.7-Gx	2500	1.5	Dry H_2	5.5	21,300
	2500	6.0	Dry H_2	6.7	25,800
	2550	6.0	Dry H_2	9.4	30,900
	2600	1.5	Dry H_2	Sweated out	
B-1-x	2500	1.5	Dry H_2	7.0	31,400
	2550	1.5	Dry H_2	Sweated out	
B-2-x	2500	1.5	Dry H_2	4.8	19,000
	2550	1.5	Dry H_2	4.4	11,500
	2600	1.5	Dry H_2	Sweated out	
B-3-x	2500	1.5	Dry H_2	1.9	11,800
	2550	1.5	Dry H_2	1.5	7,100
	2600	1.5	Dry H_2	Sweated out	
B-4-x	2500	1.5	Dry H_2	3.3	6,400
	2550	1.5	Dry H_2	3.2	6,300
	2600	1.5	Dry H_2	Sweated out	
B-5-x	2500	1.5	Dry H_2	5.5	18,200
	2550	1.5	Dry H_2	Sweated out	
B-6-x	2500	1.5	Dry H_2	3.6	14,200
	2500	6.0	Dry H_2	4.4	15,300
	2550	6.0	Dry H_2	9.1	22,700
	2600	1.5	Dry H_2	Sweated out	
B-7-x	2500	1.5	Dry H_2	Sweated out	
B-8-x	2500	1.5	Dry H_2	5.5	19,700
	2500	6.0	Dry H_2	6.5	24,800
	2550	6.0	Dry H_2	7.5	24,400
	2600	1.5	Dry H_2	Sweated out	

Table 3 (continued)

Compo- sition	Firing temp., OF	Time at temp., hrs.	Atmos- phere	Linear shrinkage, %	Modulus of rupture, psi
B-9-x	2500	1.5	Dry H ₂	5.0	21,700
	2550	6.0	Dry H ₂	6.9	24,400
	2560	6.0	Dry H ₂	Sweated out	
B-10	2500	1.5	Dry H ₂	4.5	21,800
	2500	6.0	Dry H ₂	5.6	26,900
	2550	6.0	Dry H ₂	8.0	32,300
	2600	1.5	Dry H ₂	Sweated out	
A-217.6-G	2400	1.5	Dry H ₂	1.2	22,300
	2500	1.5	Dry H ₂	1.5	29,400
	2600	1.5	Dry H ₂	Sweated out	
	2600	1.5	Wet H ₂	Sweated out	
	2600	1.5	Wet He	Sweated out	
A-218.2-G	2400	1.5	Dry H ₂	4.8	45,700
	2500	1.5	Dry H ₂	3.4	62,600
	2600	1.5	Dry H ₂	Sweated out	
	2600	1.5	Wet H ₂	Sweated out	
	2600	1.5	Wet H ₂	Sweated out	
A-218.7-G	2400	1.5	Dry H ₂	7.4	77,600*
	2500	1.5	Dry H ₂	5.7	73,300*
	2600	1.5	Dry H ₂	Melted	
	2600	1.5	Wet H ₂	Melted	
	2600	1.5	Wet He	Melted	
219.0-G	2400	1.5	Dry H ₂	7.6	49,400*
	2500	1.5	Dry H ₂	7.9	65,900*
	2600	1.5	Dry H ₂	Melted	
	2600	1.5	Wet H ₂	Melted	
	2600	1.5	Wet He	Melted	

*Bars did not break but were deformed plastically.

Table 4

Firing and Strength Data on Al_2O_3 - (65Co-30Cr-5Mo)
Compositions

Compo- sition	Firing temp., OF	Time at temp., hrs.	Atmos- phere	Linear shrinkage, %	Modulus of rupture, psi
A-225.7-G	2400	1.5	Dry H_2	0.5	2,700
	2500	1.5	Dry H_2	0.2	12,200
	2550	6.0	Dry H_2	1.5	12,300
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
A-225.7-G-2	2500	1.5	Dry H_2	5.3	25,000
	2500	6.0	Dry H_2	5.9	27,000
	2550	6.0	Dry H_2	6.6	28,000
	2600	1.5	Wet H_2	3.3	10,100
	2650	1.5	Dry H_2	Sweated out	
A-225.7-G-3*	2500	1.5	Dry H_2	9.4	42,600
	2560	6.0	Dry H_2	Sweated out	
A-227.6-G	2400	1.5	Dry H_2	6.4	32,600
	2500	1.5	Dry H_2	4.3	33,800
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
A-228.2-G	2400	1.5	Dry H_2	8.6	65,000
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
A-228.7-G	2400	1.5	Dry H_2	11.6	65,800**
	2500	1.5	Dry H_2	12.9	194,500**
	2600	1.5	Dry H_2	Melted	
	2600	1.5	Wet H_2	Melted	
	2600	1.5	Wet H_2	Melted	
229.0-G	2400	1.5	Dry H_2	13.2	135,500**
	2500	1.5	Dry H_2	13.4	137,000
	2600	1.5	Dry H_2	Melted	
	2600	1.5	Wet H_2	Melted	
	2600	1.5	Wet H_2	Melted	

* A batch addition of 5% TiC was made to the A-225.7-G composition.

** Bars did not break but were deformed plastically.

Table 5

Firing and Strength Data on Al_2O_3 -(66Ni-18Cr-16Fe)
Compositions

Compo- sition	Firing temp., OF	Time at temp., hrs.	Atmos- phere	Linear shrinkage, %	Modulus of rupture, psi
A-235.7-G	2400	1.5	Dry H_2	0.2	4,800
	2500	1.5	Dry H_2	1.3	9,100
	2550	6.0	Dry H_2	2.3	16,000
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
A-235.7-G-2	2500	1.5	Dry H_2	3.8	18,200
	2500	6.0	Dry H_2	1.7	13,100
	2550	6.0	Dry H_2	4.6	19,600
	2600	1.5	Wet He	Sweated out	
A-237.6-G	2400	1.5	Dry H_2	2.0	27,600
	2500	1.5	Dry H_2	4.0	36,300
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet He	Sweated out	
A-238.2-G	2400	1.5	Dry H_2	3.4	37,600
	2500	1.5	Dry H_2	6.2	73,800
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet He	Sweated out	
A-238.7-G	2400	1.5	Dry H_2	9.2	61,300*
	2500	1.5	Dry H_2	8.9	83,000*
	2600	1.5	Dry H_2	Sweated out	
	2600	1.5	Wet H_2	Sweated out	
	2600	1.5	Wet He	Sweated out	
239.0-G	2400	1.5	Dry H_2	9.2	51,300*
	2500	1.5	Dry H_2	9.9	60,800*
	2600	1.5	Dry H_2	Sweated out	

* Bars did not break but deformed plastically.

Table 6
Firing and Strength Data on Al₂O₃ - Stainless Steel
Compositions

Compo- sition	Firing temp., hrs.	Time at temp., hrs.	Atmos- phere	Linear shrinkage, %	Modulus of rupture, psi
A-205.6-G	2400	1.5	Dry H ₂	3.4	3,200
	2500	1.5	Dry H ₂	9.2	22,600
	2500	6.0	Dry H ₂	9.3	21,500
	2550	6.0	Dry H ₂	10.5	23,200
	2600	1.5	Dry H ₂	Sweated out	
	2600	1.5	Wet H ₂	Sweated out	
	2600	1.5	Wet H ₂	Sweated out	
A-207.6-G	2400	1.5	Dry H ₂	6.2	14,400
	2600	1.5	Dry H ₂	Sweated out	
	2600	1.5	Wet H ₂	Sweated out	
	2600	1.5	Wet He	Sweated out	
A-208.1-G	2400	1.5	Dry H ₂	15.9	17,200
	2450	1.5	Dry H ₂	16.1	101,800
	2500	1.5	Dry H ₂	17.3	95,100
	2550	1.5	Dry H ₂	16.8	132,900
	2600	1.5	Dry H ₂	16.9	112,300*
A-208.7-G	2400	1.5	Dry H ₂	11.2	85,300*
	2450	1.5	Dry H ₂	12.8	108,000*
	2500	1.5	Dry H ₂	14.4	130,000*
	2550	1.5	Dry H ₂	15.9	111,800*
	2600	1.5	Dry H ₂	15.0	102,100*
209.0-G	2400	1.5	Dry H ₂	15.8	88,900*
	2450	1.5	Dry H ₂	14.6	89,900*
	2500	1.5	Dry H ₂	16.0	102,300*
	2550	1.5	Dry H ₂	10.2	70,700*
	2600	1.5	Dry H ₂	16.3	90,400*

* Bars did not break but deformed plastically.

DENSITY

Density and porosity measurements were made only on the three high-metal compositions in the Al_2O_3 -stainless steel cermets.

In the porosity determination of these three compositions by the ASTM water-boiling test the porosity was negligible and in many instances the saturated weight was less than the dry weight by 1 to 2 mg. The limits of error in this test probably cover the small range of near-zero porosities measured.

Density measurements and metallographic examination of specimens reveal the presence of closed pores not detectable with the water-boiling test.

The densities with respect to firing temperature are given in Table 7.

Table 7

Density of Al_2O_3 -Stainless Steel Compositions

Composition	Sintering temperature, °F	Density, gm/cc	Density, % of theoretical
A-208.1-G	2400	6.48	88.6
	2450	6.66	91.0
	2500	6.77	92.5
	2550	6.90	94.3
	2600	6.87	93.9
A-208.7-G	2400	6.92	89.8
	2450	7.14	92.7
	2500	7.17	93.1
	2550	7.00	90.9
	2600	7.14	92.7
209.0-G	2400	7.44	94.2
	2450	7.39	93.5
	2500	7.54	95.4
	2550	6.81	86.2
	2600	7.35	93.0

MODULUS OF ELASTICITY

Young's modulus of elasticity, E , was determined on compositions A-208.1-G, A-208.7-G, and 209.0-G using a sonic method. The same bar specimens that were used for the modulus of rupture test were used in the determination. The method consisted of vibrating the specimen in its fundamental frequency by means of an audio oscillator and loudspeaker. Vibrations of the specimen are transmitted through a crystal pickup, amplified, and fed to the vertical plates of an oscilloscope. Resonance of the specimen is noted when the amplitude of the wave form reaches a maximum. The resonant frequency is then measured with a frequency meter. Figure 3 is a schematic drawing of the setup.

The modulus of elasticity was calculated using Pickett's ⁶ formula:

$$E = C w n^2,$$

in which C is a factor embodying shape, size, mode of vibration, and Poisson's ratio; w is the specimen weight, and n is the fundamental frequency of the specimen.

The values of E obtained are given in Table 8, and are shown graphically as a function of sintering temperature in Figure 2.

Table 8

Modulus of Elasticity of Al_2O_3 -Stainless Steel
Compositions in 10^6 psi.

Composition	Firing temperature, °F				
	2400	2450	2500	2550	2600
A-208.1-G	24.5	27.2		27.8	28.3
	24.4	28.2		29.0	28.9
	23.8	26.0		28.9	28.2
A-208.7-G	24.1	25.7	23.8	25.6	25.4
	23.5	25.8	23.0	23.6	26.4
	23.5	24.9	23.0	23.5	25.6
209.0-G	25.8	23.3	25.5	20.8	28.2
	25.7	25.5	25.6	21.3	26.3
	24.7	25.5	24.7	21.4	26.3

TENSILE STRENGTH

A limited amount of tensile strength data was obtained for the compositions A-208.1-G, A-208.7-G, and 209.0-G. These data are given in Table 9 below and in Figure 4.

Table 9

Tensile Strength of Al_2O_3 - Stainless Steel
Compositions at Various Temperatures

Composition	Specimen number	Temperature of test, °F	Tensile strength, psi
A-208.2-G	5	75	43,510
	6	75	44,180
	10	75	44,020
	8	1600	23,840
	1	1800	8,790
	2	1800	7,290
A-208.7-G	2	75	53,590
	5	75	56,790
	1	1600	22,000
	7	1800	9,830
209.0-G	1	75	56,970
	2	75	70,160
	5	75	58,010
	4	1600	25,740
	3	1800	12,310

TENSILE STRESS RUPTURE

Tensile stress rupture data for the compositions A-208.1-G and A-208.7-G were determined at 1800°F. The results are given in Table 10 and in Figure 5.

Table 10

Tensile Stress Rupture Data at 1800°F

Composition	Specimen number	Stress, psi	Time to failure, hrs.
A-218.1-G	3	5,000	0.2
	4	4,000	0.8
	7	3,000	6.5

Table 10 (continued)

Composition	Specimen number	Stress, psi	Time to failure, hrs.
A-218.7-G	3	5,000	1.0
	4	4,000	3.2
	8	3,000	12.6

VIII. FLAMEHOLDER SEGMENTS

Since June, 1953 this laboratory has been engaged intermittently in fabricating flame holders for testing purposes as a cooperative activity with the National Bureau of Standards. The first four of these flameholders were of the composition A-16.14-G (72%Cr-28%Al₂O₃)². Although this composition was considered marginal in thermal shock resistance in tests on nozzle diaphragm blades, the flameholders withstood 30 cycles between 100°F, and 2100°F without any sign of thermal failure. Next, the composition A-355.6-G was submitted for testing and it also withstood 30 cycles without failure. The design and method of forming the flameholders is fully described in a previous report.³ The following discussion is concerned with the work done during the present contract period on the development of a method of fabricating flameholder segments having a rather intricate configuration.

Five best-effort flameholder segments of suitable shape and cross section for incorporation into a complete flameholder ring were produced for tests by the National Bureau of Standards. These segments were fabricated from cermet body A-355.6-G, consisting of equal volume proportions of an 80Cr20Mo alloy and Al₂O₃, and had the configuration shown in Figure 6.

To obtain this complex shape a two-stage forming procedure was used. First the cermet powder, prepared as described in Wright Air Development Center Technical Report 54-173, Part 1,² was placed in a flexible plastic container and hand tamped around a steel gutter form. The bag containing the cermet and gutter form was then sealed in a section of rubber tubing and evacuated, after which the complete assemblage was immersed in a water - soluble oil mixture contained in a 5½ inch I.D. hydrostatic pressing cylinder and a pressure of 25,000 psi applied. Figure 7 shows the plastic container, pressed shape, and steel gutter form.

In the second stage of preparation the rough shape formed by hydrostatic pressing was ground to the desired green dimensions on a specially constructed apparatus. (See Figures 8 and 9.) Adjustments on this apparatus provided for grinding all faces and the grooves of the segments. The steel gutter form was used as a base upon which the segments were placed for grinding. Finishing of the surfaces to the desired green dimensions was done with abrasive paper.

After the segments were shaped to the desired green contour they were fired in a hydrogen atmosphere in the same manner as that previously described for body A-355.6-G. The segments were fired two at a time and were placed in the furnace on an alumina plaque. All of the segments were fired in the nose-down position.

After firing, the ends of the segments were cut to shape with a resin-bonded 100-grit silicon carbide cutting-off wheel.

A tolerance of ± 0.030 inches was suggested by N.B.S. as the standard toward which efforts should be directed in fabricating the flameholder segments. The dimensions of the segments were set so that the cermet segments could be tested as part of the inner flameholder ring of a J-47 engine, alternated with metal segments. Best-effort segments were to be supplied for test assuming that fabrication problems could be overcome.

The dimensions of the segments produced did not all fall within the suggested tolerance. (See Table 11 for the fired dimensions of the best five segments produced.) The ends of the segments were not all cut on a true radius so as to give a perfect fit into a flameholder ring. In addition at least one of the specimens developed a crack in firing which would severely limit its expected performance in an engine test. One segment was broken in grinding to the desired finished green shape.

These defects are illustrative of the difficulties encountered in the fabrication of a segment of this type. It was felt that producing segments more nearly within the desired tolerances and free of cracks and breaks constituted a development problem of respectable magnitude. It is believed, however, that should the time and expense be felt justified the experience gained in the development of these specimens can be utilized to advantage in producing specimens within a ± 0.030 inch tolerance or better.

This statement is made because the source of the defects in the best-effort specimens is known and they can be eliminated by redesign of some of the equipment and accessories used and by modification of part of the procedure used to produce the

shapes. For example, the variation from the desired dimensions was caused by shrinkage differences related to thickness and to uneven packing of the material in the plastic container prior to hydrostatic pressing. A redesign of the gutter form and the plastic container can provide the necessary extra allowances for shrinkage in the thinner portions of the shape. The remedy was known before the firing of the segments was completed but was not applied because the additional time and expense was not considered to be justified.

The irregular shrinkage caused by uneven packing prior to pressing can be eliminated partially by the redesign of the gutter form and a new plastic container and partially by better technique and control of packing. The greatest shrinkage occurs in those areas where the material was packed least tightly and these pockets of loosely packed material can be eliminated by improved technique.

Shapes formed by hydrostatic pressing of the A-355.6-G composition in previous investigations did not require the use of a binder-lubricant to impart adequate handling strength. However, because the size of these present flameholders necessitates the use of a larger hydrostatic cylinder, the pressing pressures are lower than those employed previously. This lower pressing pressure combined with the need for good green strength made the use of a binder necessary.

Better packing technique coupled with the use of a stronger binder such as Carbowax should eliminate cracks and breaks in the segments. The camphor used in the first segments did not give sufficient green strength to the shapes to permit fairly rough handling; as a result cracks were formed during the grinding operation. Some of these were not discovered prior to firing. Use of a binder such as Carbowax would be expected to give enough green strength to the shapes to allow them to withstand the shocks and stresses encountered in handling and grinding. Previous experience with this material as a binder has shown that it does produce high green strength, although the dewaxing procedure is a more involved and difficult one than that required for camphor.

Finally, it is possible, though not definite, that once the shrinkage characteristics of the shape have been established for body A-355.6-G the ends of the segments can be cut to the proper length and the difficulties encountered in cutting this extremely hard material can be eliminated with a resulting reduction in finishing time and improvement in finish on the specimen ends.

It is concluded from the results obtained in these first attempts to produce complex shapes like the one described above that the problems of fabrication are not insurmountable, and should such shapes be considered worthy of further study it is believed that they can be produced within tolerances of ± 0.030 inch or better provided sufficient time is allowed for working out the problems discussed.

Table 11
Fired Dimensions of Flameholder Segments

Station*	A	B	C	D	E	F
Desired Dimension						
Segment 1.**	.687	.767	.750	.750	.125	.125
a.	.811	.758	.779	.792		.129
b.	.810	.743	.750	.800	.118	.135
c.	.820	.749	--	.779	--	.140
Segment 2.						
a.	.798	.743	.749	.743	.113	.132
b.	.788	.740	.729	.760	.107	.1385
c.	.803	.740	.778	.792	.111	.140
Segment 3.						
a.	.815	.765	.771	.792	.121	.149
b.	.815	.757	.743	.780	.119	.147
c.	.828	.765	.778	.800	.120	.149
Segment 4.						
a.	.813	.750	.770	.804	.120	.138
b.	.812	.743	.784	.809	.118	.145
c.	.824	.750	.810	.815	.123	.145
Segment 5.						
a.	.775	.795	.760	.779	.134	.137
b.	.764	.782	.755	.750	.106	.139
c.	.775	.790	.780	.770	.106	.148

* Note: See Figure 10 for location of stations shown above.

** Inner leg of Segment 1 was broken in grinding.

IX. DISCUSSION

Originally the present program included the investigation of the cermet compositions containing Al_2O_3 and the alloy 76Cr-24Ti, but further work on these compositions was discontinued after a preliminary firing study. The results obtained on these bodies showed a high degree of reaction between the titanium or its oxidation products and the alumina and/or chromium. The sintered bars were cored and extremely brittle. The fact that excessive oxidation was taking place (as shown by a 3 to 5% weight gain in firing) even when the atmosphere was dry purified hydrogen (as introduced into the refractory-lined furnace) seemed to preclude the possibility of producing a satisfactory cermet with these materials.

The following discussion applies only to the compositions containing the alloys: 80Ni-20Cr, 65Co-30Cr-5Mo, 66Ni-18Cr-16Fe, and an 18-8 stainless steel.

The unsolved problem of devising a means of promoting wetting between Al_2O_3 and the metals Fe, Ni, and Co continues to be a major obstacle to the development and evaluation of cermets containing these metals.

All of the attempts to produce bonding between the oxide and metal phases by various methods of sintering and composition modifications were unsuccessful. Sintering in atmospheres other than dry purified hydrogen resulted in poorer consolidation and strength in every instance. A slight improvement in strength was noted in some compositions when given a 6-hour soak period instead of the usual 1.5 hours, but the magnitude of the improvement did not warrant going further in this direction. The addition of agents calculated to produce wetting did not give the desired results and in most cases served only to lower the temperature at which the body would begin to exude metal.

The desirability of using prealloyed metal powders in cermet compositions was demonstrated by the marked increase in both firing shrinkage and transverse strength in the A-215.7-G composition when prepared with prealloyed metal. A similar improvement was obtained with the A-225.7-G and A-235.7-G compositions that were given the prealloying treatment explained in the section, BATCH PREPARATION.

The composition A-215.7-G was used as the base composition to which batch additions were made in an attempt to produce wetting. This composition was employed because the metal phase was available as an alloy powder and because the alloy is generally representative of the alloys being investigated. It was assumed that a method of promoting bonding with this alloy would also be applicable with other nickel, iron, and cobalt base alloys.

Since efforts towards effecting a bond between Al_2O_3 and these alloys were not supplying the answer to the problem, it was decided to proceed with the evaluation of those high-metal compositions that could be sintered to a relatively dense condition. Time permitted only three of these compositions to be evaluated, namely, the stainless steel- Al_2O_3 bodies containing 0, 5, and 15 volume percent Al_2O_3 . The remainder of this section will be devoted to a discussion of these three compositions.

FORMING

Considerable difficulty was encountered in forming bar specimens. The phenomenon of the die-pressed bars being disintegrated by hydrostatic pressing has never occurred with other materials processed in these laboratories and the explanation for it is not definitely known. Only by doubling the amount of binder and the pressing pressure normally used, could satisfactory specimens be formed. The batch material did not possess good packing characteristics, as was shown by the excessive amount of shrinkage that took place when hydrostatically pressing rod specimens from material that had been packed by vibration during charging.

FIRING

The firing behavior of these three compositions is somewhat erratic as shown by shrinkage, modulus of rupture, and modulus of elasticity measurements made on the specimens fired at various temperatures. If it were not for the fact that the test specimens of all three compositions were fired together at each temperature it would be logical to assume an error had been made in temperature measurement such that one composition could manifest erratic properties while the others did not. This type of behavior would not be expected in bodies with such similar compositions being processed and fired identically.

PHYSICAL PROPERTIES

The modulus of rupture values obtained for these compositions are somewhat misleading, indicating a higher level of strength than they actually possess, because of the plastic yielding of the specimens under load. The results do show that the Al_2O_3 has a stiffening effect on the metal.

The results of this study have shown that the transverse strength test is not suitable for these high-metal cermets and that a tensile test in which large numbers of determinations can be made quickly and economically should be devised.

The modulus of elasticity was determined for all three compositions fired at each temperature. Although the results are not uniform for each composition, the modulus of elasticity increased with the higher firing temperature and was proportional to the density of the fired bars. That there is a bonding between the metal and the Al_2O_3 is shown by the increasing modulus of elasticity with increasing amounts of Al_2O_3 . If there were no bonding, the Al_2O_3 would give the effect of a lesser density or higher porosity, thereby lowering the modulus of elasticity.

Although the E values obtained indicate that sufficient bonding is taking place to permit stresses to be transmitted from one phase to the other, it is strongly suspected that the bond material is in the nature of a glass. If this is true, it would help to explain why the Al_2O_3 , instead of increasing, actually decreased the high temperature strength and oxidation resistance properties of the material. That the bond material may be glass is borne out by the occurrence of small glass beads adhering to the surface of the sintered specimens. The explanation for the formation of this glass must be that the silicon content of the stainless steel (2.43%) oxidized either in processing or in the early stages of firing, and then combined with the Al_2O_3 and other metallic oxides to form a glass.

The sonic method used for determining modulus of elasticity provides a relatively quick and easy way for making this measurement. In checks with materials of known E values it has proved to be highly reliable. Plans for future work include the use of this method as a routine determination.

The tensile strength values obtained on these compositions were so close that the effects of small amounts of Al_2O_3 in a metal are obscured. The data do show, however, that the Al_2O_3 did not provide increased strength at elevated temperatures, but tended to weaken the metal.

The results of tensile stress-rupture tests at 1800°F on the compositions containing 5 and 15 volume percent Al_2O_3 show that both possess rather low load-sustaining ability. Here again the higher alumina content has a detrimental effect on the strength, the 5% Al_2O_3 composition having the ability to sustain a load more than twice as long as the one with 15% Al_2O_3 .

Oxidation resistance tests at 2000°F were made on all three compositions, but because provision was not made for collecting spalled oxide, an accurate quantitative measurement of this property cannot be given. The test did show qualitatively the spalling (and oxidation) to be greater with the higher Al_2O_3 content. Since the primary purpose of the

silicon in the stainless steel is to increase the resistance to scaling at high temperatures, the results of this test substantiate the theory that the silicon was used up in combining with the Al_2O_3 to form a glass.

X. CONCLUSIONS

1. There is no inherent tendency for the metals nickel, cobalt, iron, or their alloys to wet alumina at or near the melting temperature of the metal.
2. For the beneficial effects of alumina in metal to be realized the two phases must be intimately bonded and furthermore the nature of the bond should be such that it is not markedly weakened at elevated temperatures.
3. Materials containing silicon or other metals whose oxides are glass formers should not be allowed to oxidize in the sintering of oxide-base cermets because the formation of an interstitial glass is normally detrimental to the high-temperature strength.
4. The use of prealloyed metals is desirable in cermet compositions because production of a homogeneous metal phase during sintering is difficult where local eutectic melting and differences in oxidation potential of the constituent metals is encountered.

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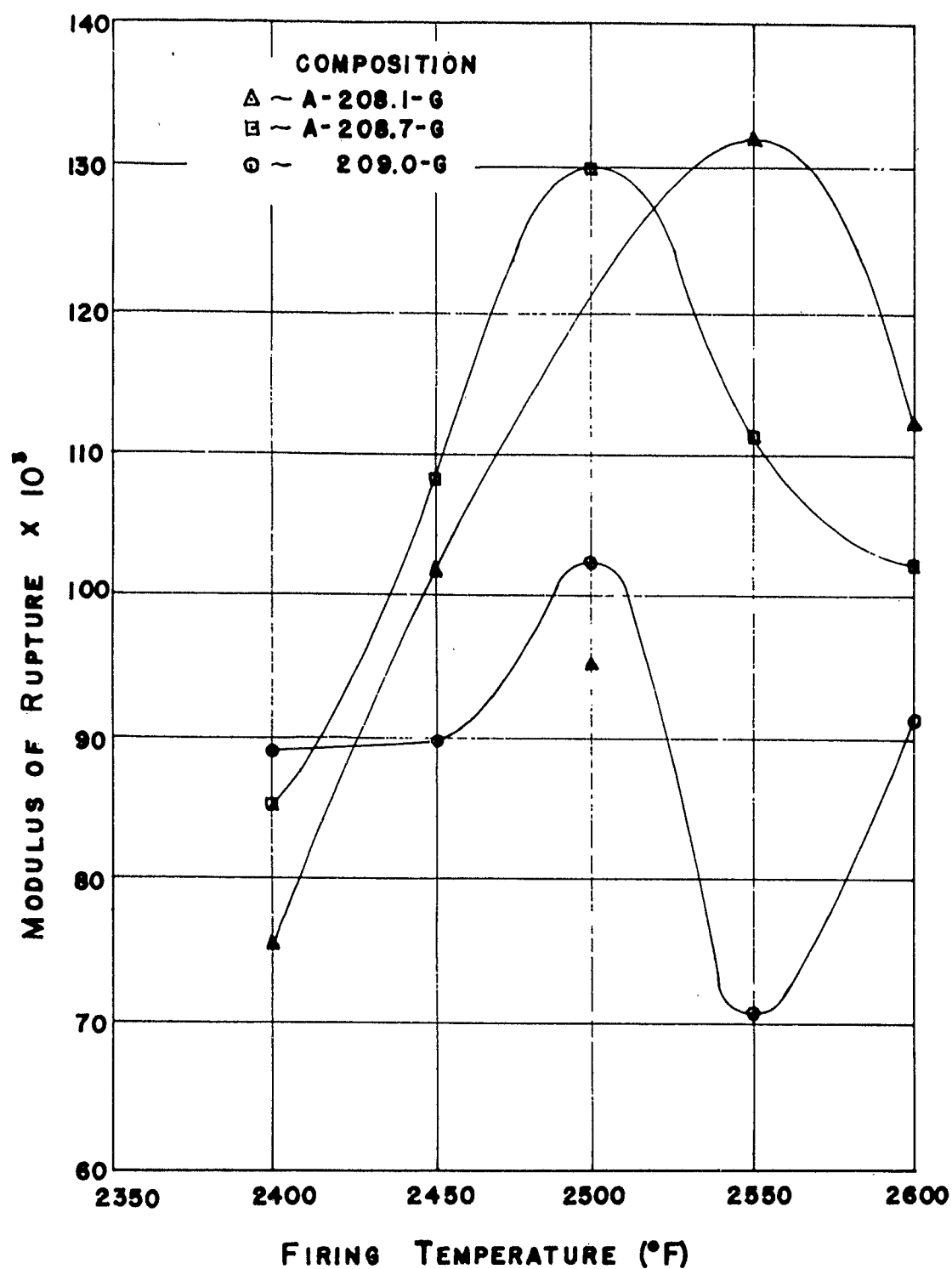


Figure 1. Modulus of Rupture vs Firing Temperature For Three Al_2O_3 Stainless Steel Compositions

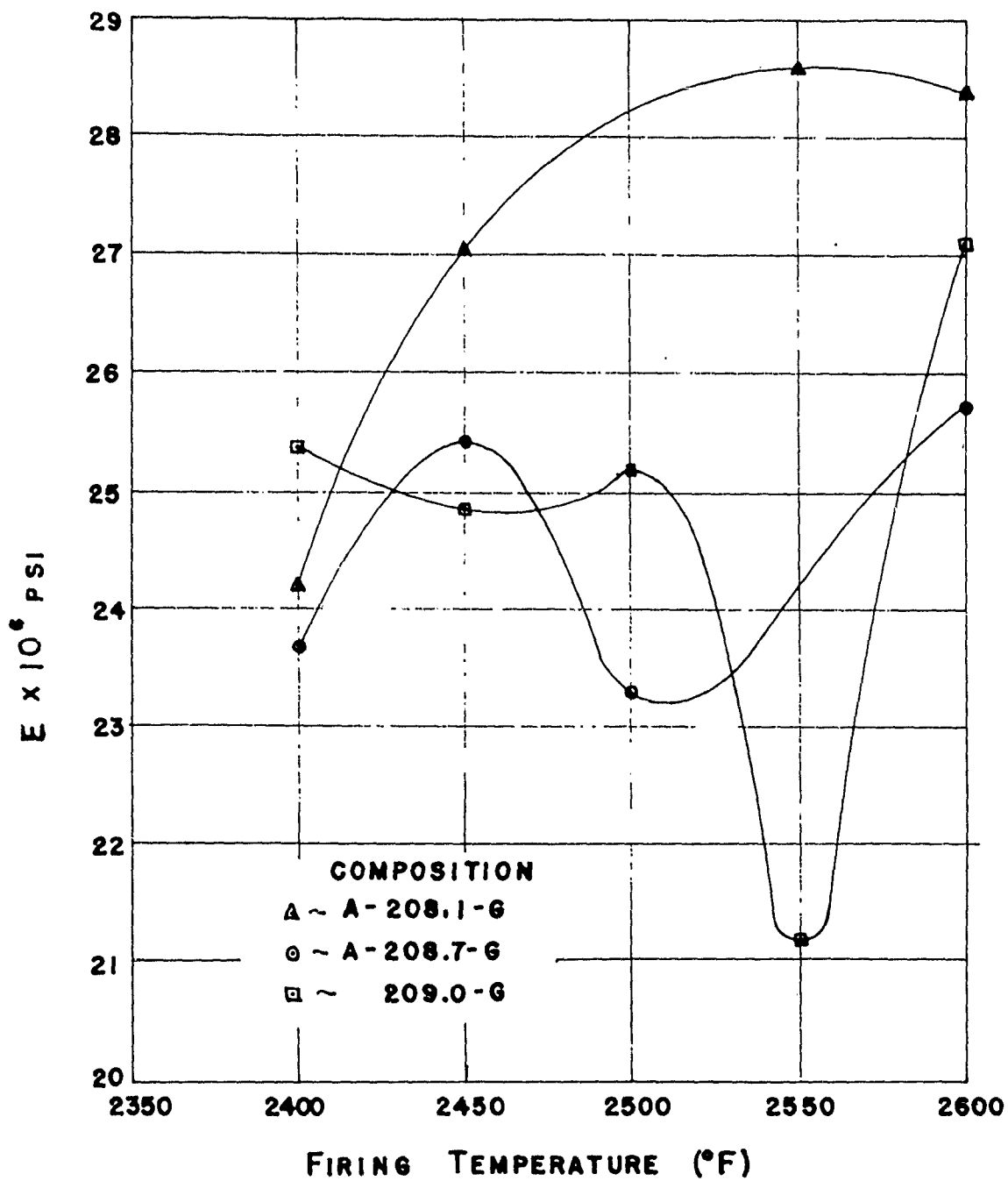


Figure 2. Modulus of Elasticity vs Firing Temperature
For Three Al_2O_3 Stainless Steel Compositions

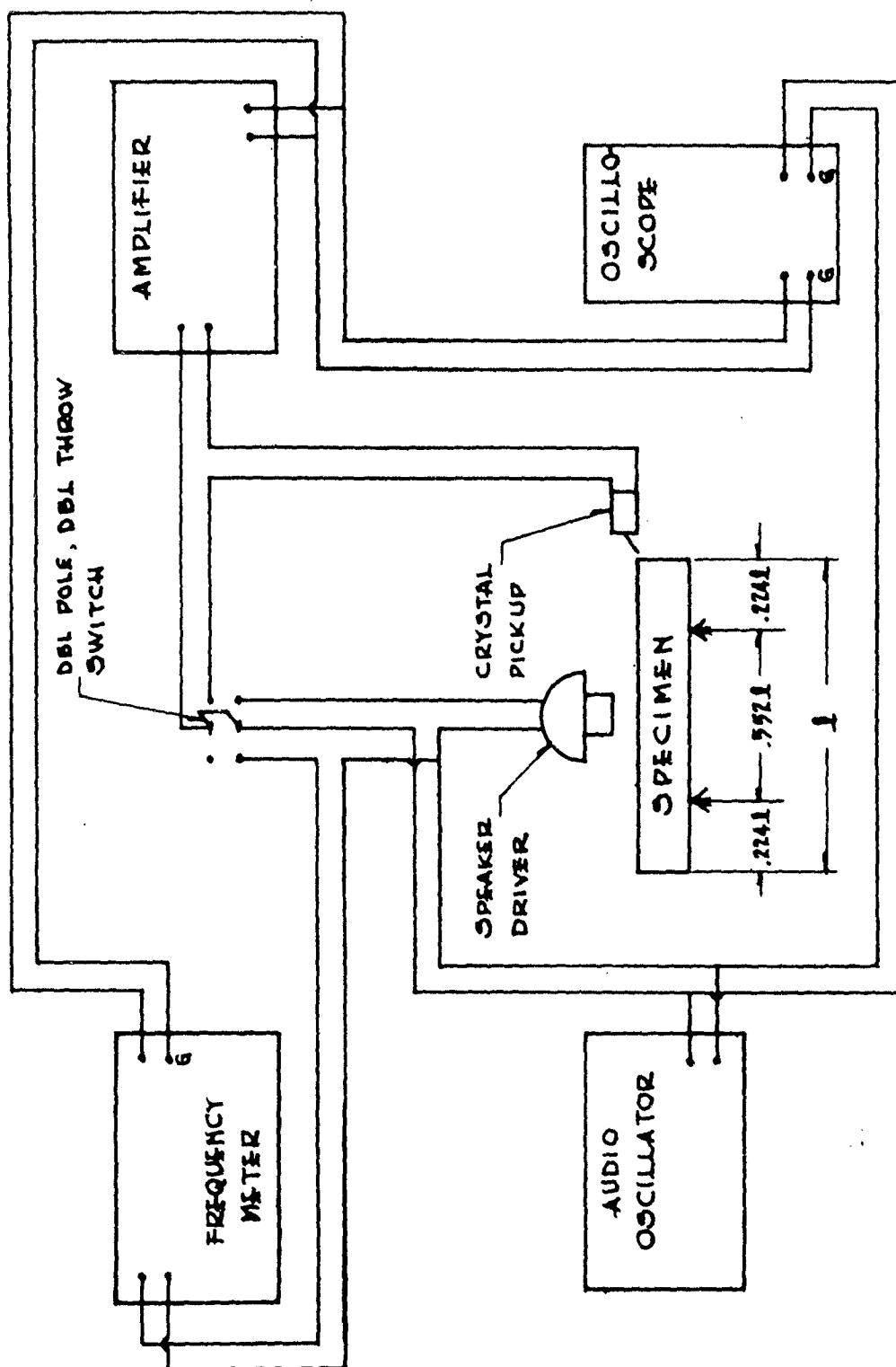


Figure 3. Sonic modulus of Elasticity Apparatus

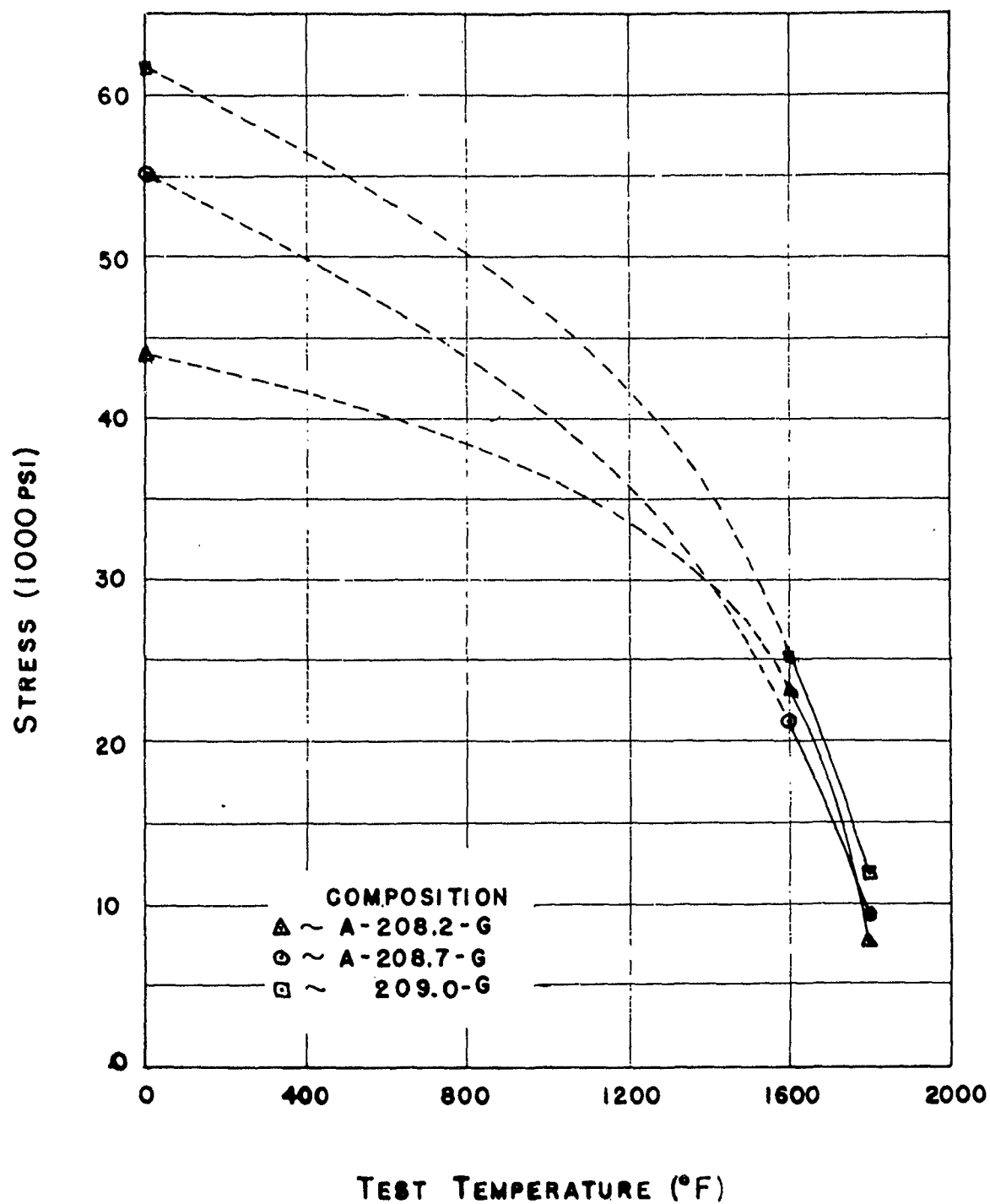


Figure 4. Tensile Strength vs Test Temperature For Three Al_2O_3 Stainless Steel Compositions

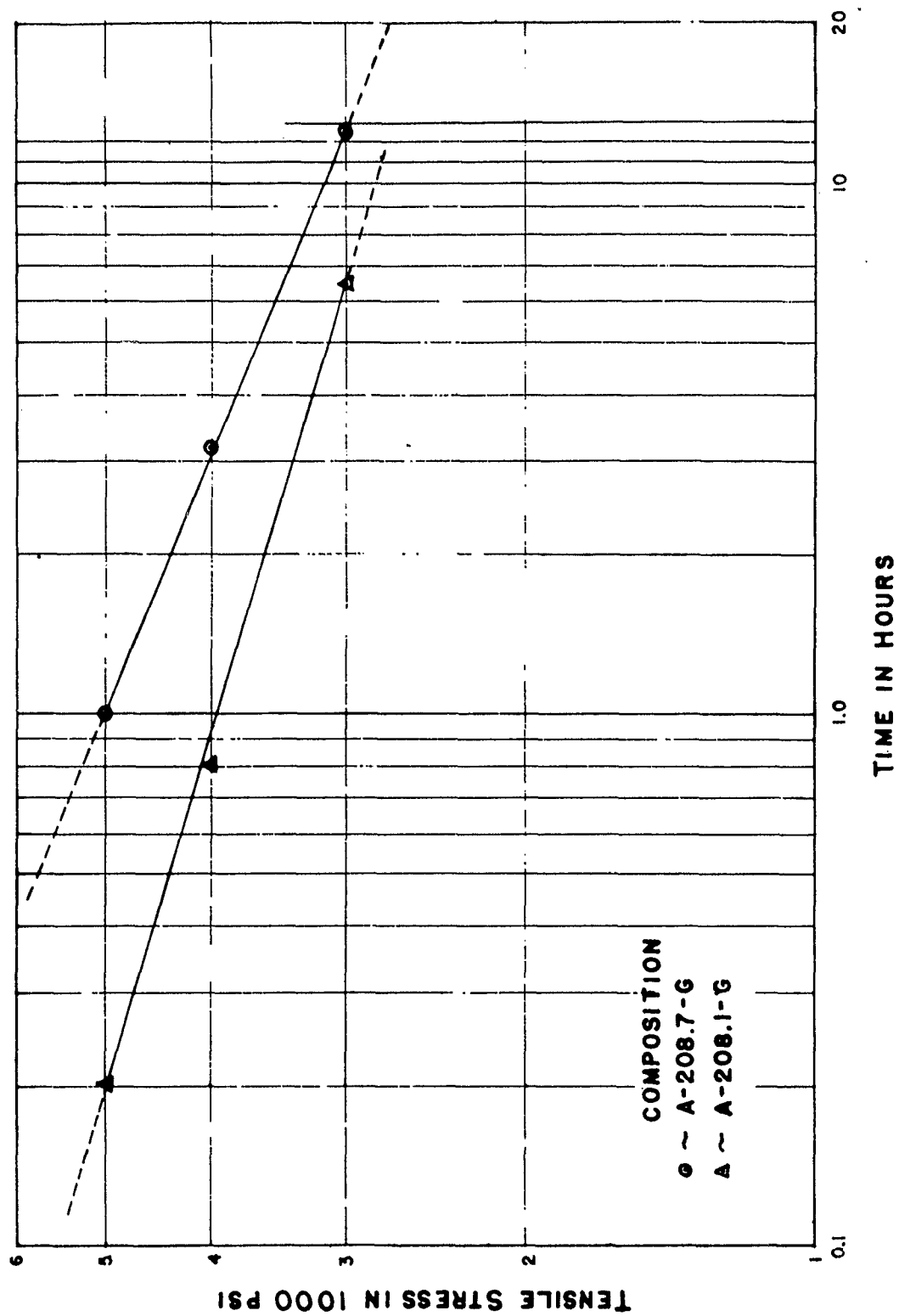


Figure 5. Stress Rupture in Tension at 1800°F

FLAMEHOLDER SEGMENT FOR NATIONAL BUREAU OF STANDARDS

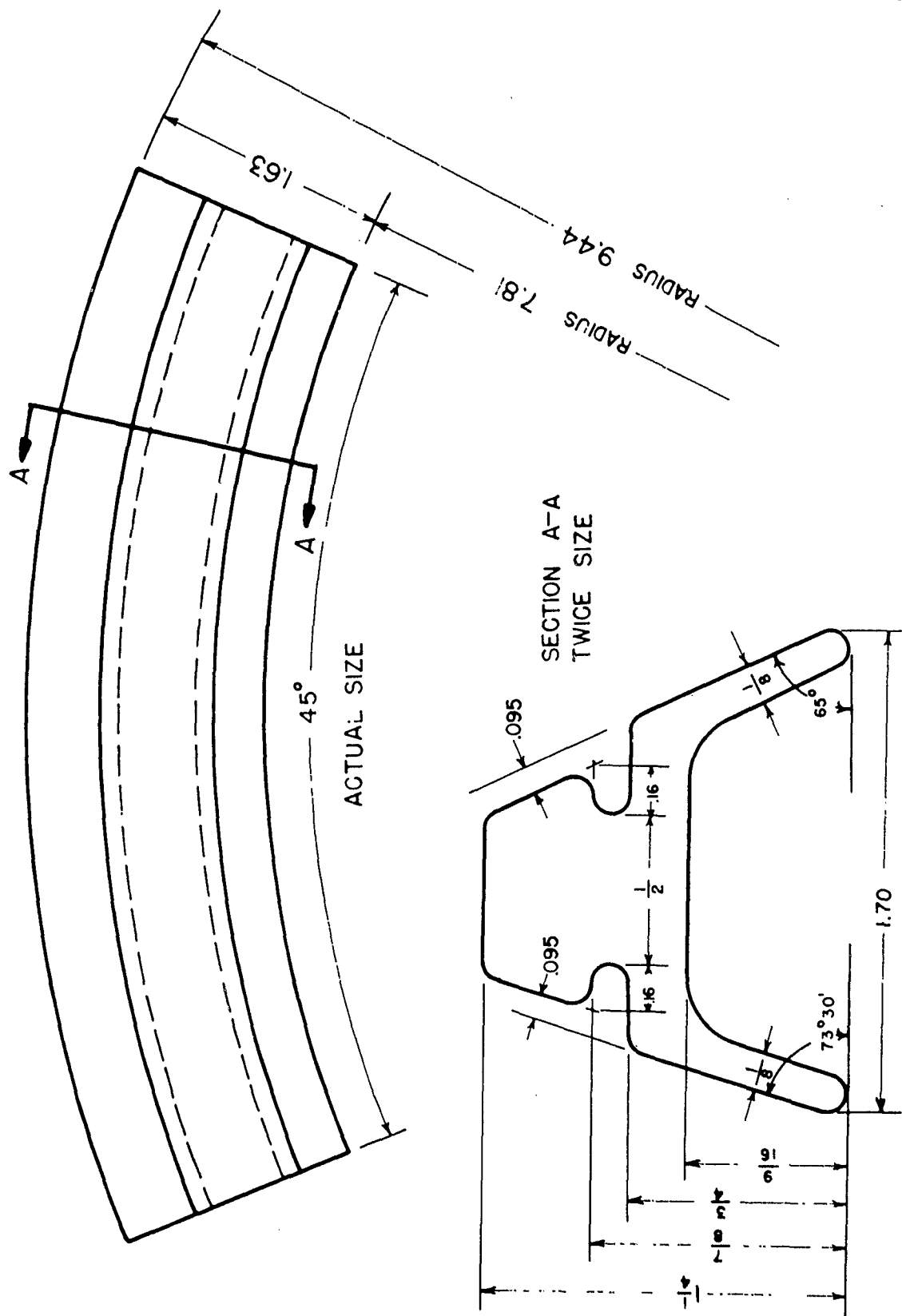


Figure 6.

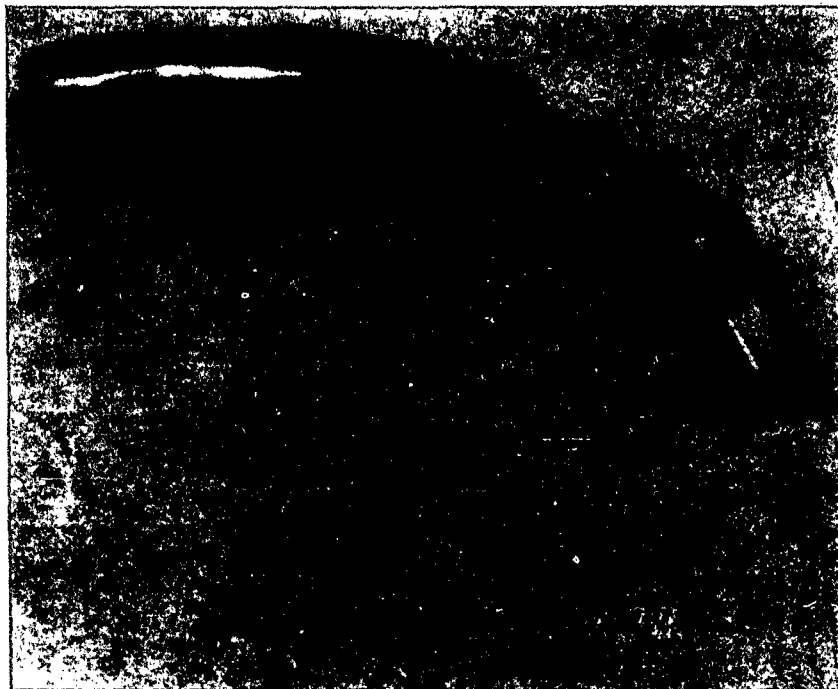


Figure 7. Flameholder Forming
Accessories

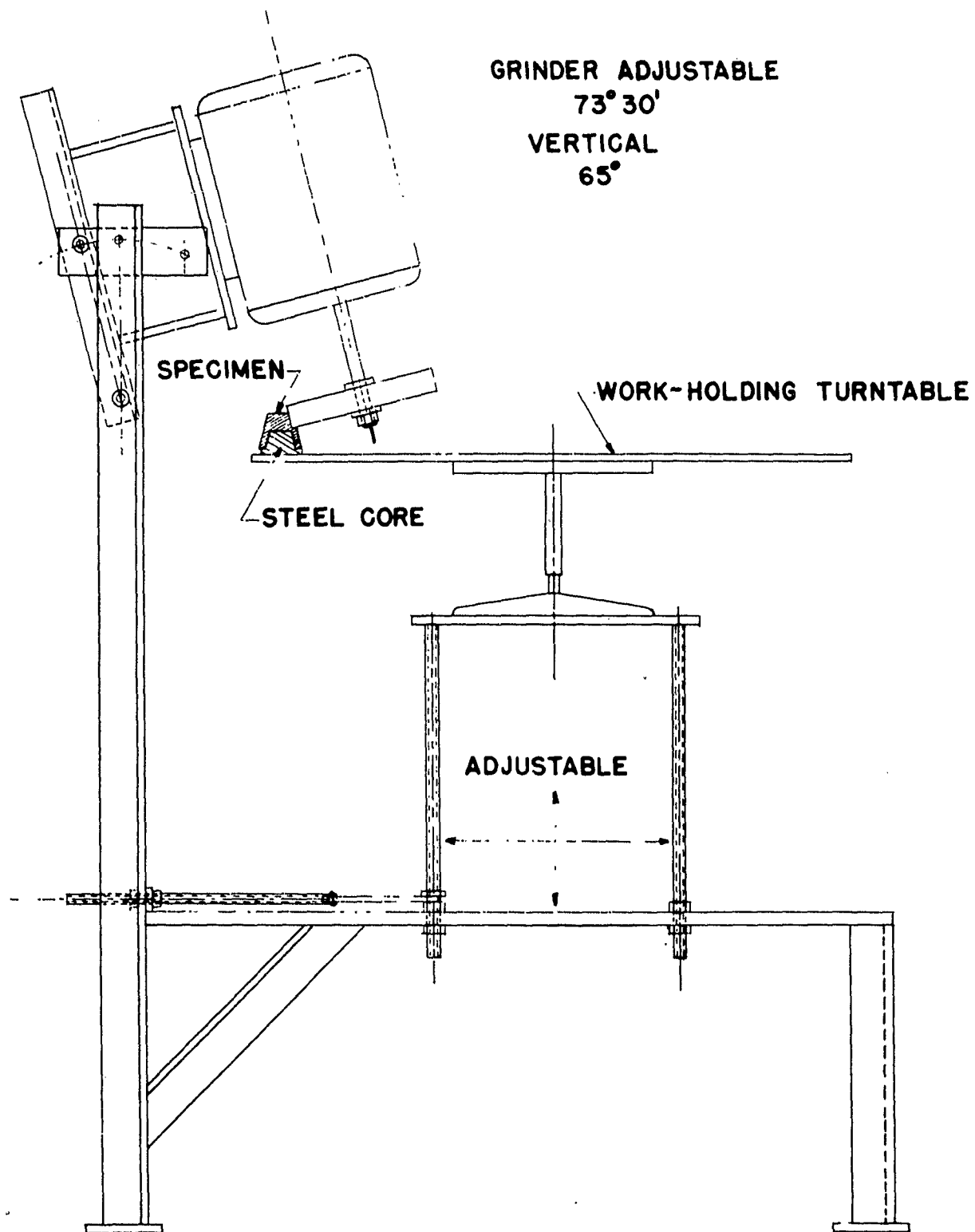


Figure 8. Grinding Device for Flameholders



Figure 9. Grinding Device for
Flameholders

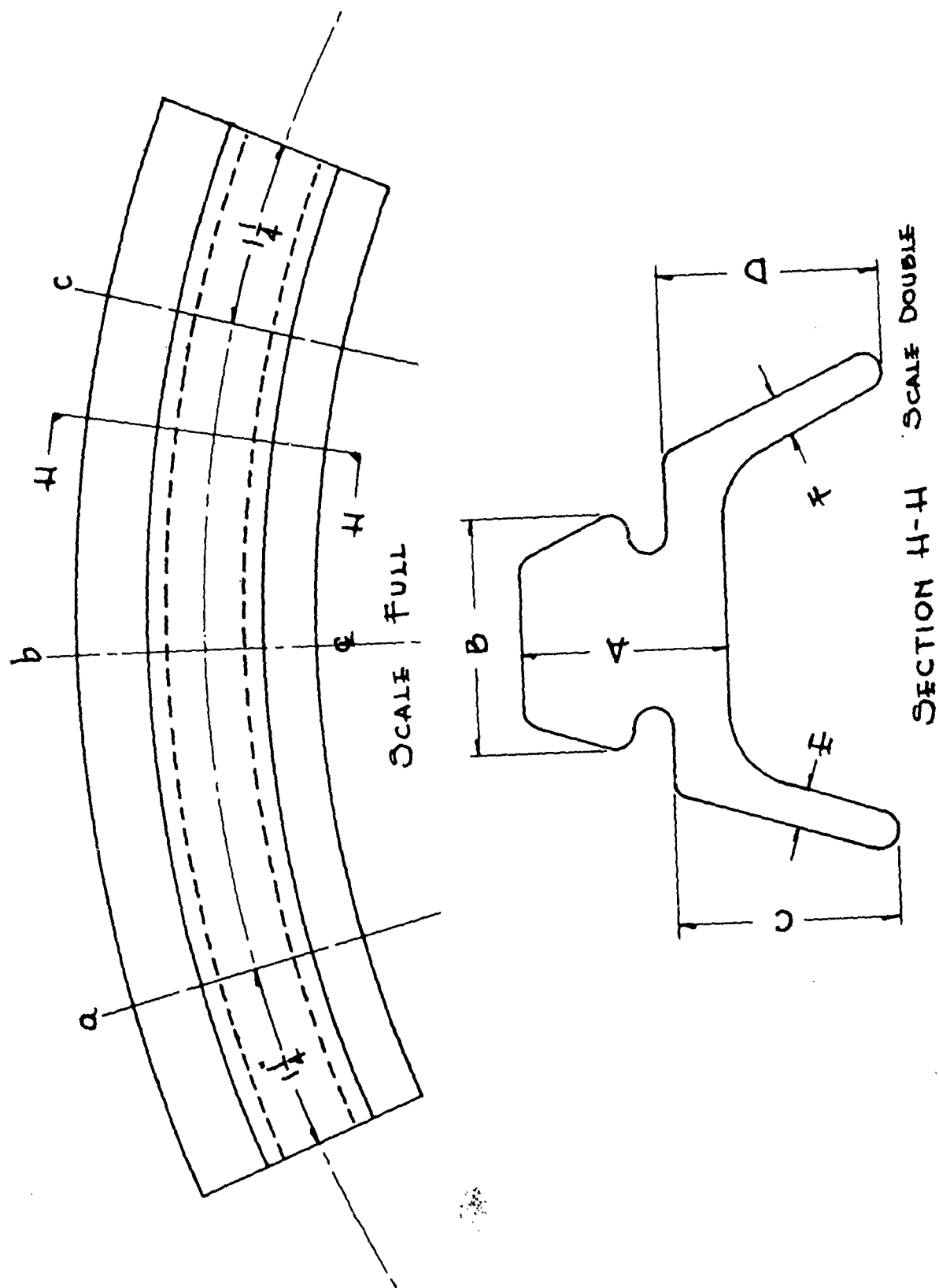


Figure 10. Location of Stations for Dimensions Shown in Table 11