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RESEARCH AND DEVELOPMENT ON INORGANIC HIGH TEMPERATURE ADHESIVES FOR METALS AND COMPOSITE CONSTRUCTIONS

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FOREWORD

This report was prepared by the Engineering Department, Aeronca Manufacturing Corporation, Middletown, Ohio under USAF Contract Number AF 33(616)-5538. This contract was initiated under Project Number 7340 "Non-Metallic and Composite Materials," Task No. 73401, "Organic and Inorganic Adhesives." This work was administered under the direction of the Materials Laboratory, Directorate of Laboratories, Wright Air Development Center with Floyd H. Bair acting as project engineer.

This report covers work done between March 1, 1958 and February 28, 1959.

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ABSTRACT

An inorganic adhesive for 17-7 PH stainless steel metal bond was evaluated for resistance to various media and to various stresses. Factors were explored which affect the reproducibility of tests made on inorganic adhesive lap shear specimens.

An inorganic adhesive was developed for 17-7 PH stainless steel honeycomb sandwiches.

An inorganic adhesive bonding process was developed for fabricating 17-7 PH stainless steel honeycomb sandwiches.

Honeycomb sandwiches of 17-7 PH steel bonded with inorganic adhesives were evaluated in flexure and edgewise compression.

PUBLICATION REVIEW

This report has been reviewed and is approved.

R. T. Schwartz

R. T. SCHWARTZ
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INTRODUCTION

This technical report* reviews the experimental work on practical inorganic adhesives performed by the Aeronca Manufacturing Corporation under the terms of Contract AF 33(616)-5538 during the period from 1 March 1958 to 28 February 1959.

The object of this investigation is to develop and evaluate practical inorganic adhesives for bonding heat treatable stainless steel honeycomb sandwiches, and to develop tooling and fabricating methods for producing such sandwiches.

The University of Illinois, under Contract AF 33(616)-2556 and reported in WADC TR 55-491, Parts I, II and III, developed inorganic adhesives which attained tensile shear strengths of approximately 1500 psi at temperatures up to 1000°F in lap shear specimens of 17-7 PH stainless steel. These adhesives were used as the basis for developing practical bonding agents for stainless steel honeycomb sandwiches.

The program reported is divided into five major steps:

1. The evaluation of the UI 117-63 adhesive reported in WADC 55-491, Part III.

2. The reproducibility studies necessitated by the lack of consistent tensile shear strength results for UI 117-63 adhesive.

3. The development of processes and tooling for manufacturing inorganic adhesive honeycomb sandwiches.

4. The adhesive modification to develop an inorganic adhesive for honeycomb sandwiches.

5. The fabrication and test evaluation of inorganic adhesive bonded honeycomb sandwiches.

A Recommended Procedure for Fabricating Sandwiches follows this Introduction. The body of the report consists of three sections - Results, Testing Procedures, and Conclusions. In each section, the steps in the program are covered in the previously outlined sequence. Recommended future work is then outlined. An Appendix contains detailed test results and references.

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*Manuscript released by the authors on 1 March 1959 for publication as a WADC technical report.

RECOMMENDED PROCEDURE FOR FABRICATING SANDWICHES

Sandwiches consist of .032" 17-7 PH facing sheets, .500" 17-7 PH, .002" foil, 1/4" cell core; bonded with UI 1067-1A adhesive.

I. Preparing the Adhesive

Adhesive Used: UI 1067-1A

A. Raw Materials, Frit UI 1067

	%	Grams/ 2000 g batch
400 Mesh Silica, Chicago Vitreous	24.8	496
Sodium Nitrate, Coarse Granular, Tech.	9.0	180
Boric Acid, Chicago Vitreous	66.2	1324

B. Calculated UI 1067 Frit Oxide Composition

SiO_2	38.0%
Na_2O	5.0%
B_2O_3	57.0%

C. Method of Manufacturing Frit

Basic laboratory size batch - 2000 grams of raw materials.

1. Heat the smelting crucible to approximately 2400°F.
2. Add approximately 1000 grams of the well-mixed raw materials.
3. Heat at 2400°F for approximately 20 minutes until most of the frothing has stopped.
4. Add the rest of the raw materials.
5. Heat at 2400-2500°F from 20 to 60 minutes until all frothing has stopped and a pulled thread of the glass contains no bubbles.
6. Pour the molten smelt into cold water. To keep the frit chips as small as possible, the volume of water should be as large as possible and the frit should be poured very slowly.

7. Remove the excess water by blotting. Dry the frit in an oven at 250°F.

D. Milling of the Frits and Preparation of the Slip

1. Fill the dry ball mill to 40% of its capacity with 50% 3/4" porcelain balls and 50% 1/2" porcelain balls.

2. Add the frit to the 3/4 level of the mill.

3. Mill the frit for 3 hours at 68 rpm in a Paul Abbey "Assay", 3-quart capacity ball mill or equivalent.

4. Remove all material passing the 48 mesh screen. With this much milling, approximately half of the frit will pass the 48 mesh screen.

5. Mill the remaining frit at the same speed for approximately 5 more hours or until less than 1/2 % of the frit is contained on the 48 mesh screen. Discard the material retained on the 48 mesh screen.

6. Weigh all of the ground frit and return to the ball mill.

7. Add 2 parts of Fe_2O_3 powder and 20 parts -325 mesh 304 PC stainless steel powder to the 100 parts of ground UI 1067 frit already in the mill.

8. Add 160 parts of water to the mill and mill the mixture for 1 hour. Additions up to 240 parts of water may have to be made to make a flowable slip.

9. Continue wet milling until all but 1/2% of the material passes the 200 mesh screen. This takes approximately 3 hours.

10. The slip should be discarded after 3 days or at any time there is evidence of crystals forming in the slip.

II. <u>Surface Preparation of Facing Sheets</u>

NOTE: Handle all parts with clean white cotton gloves.

A. Cut sheets to size.

B. Vapor degrease facing sheets using best practice.

C. Stand skins in a rack, each skin separated from the other. Any support for the skin should touch the nonbonding side of the skin.

D. Place in furnace at 1000°F for 30 minutes to heat scale the sheet.

E. Remove from furnace and air cool.

F. Place vertically in 20% HNO_3, 4% HF by volume acid etch bath for 5 minutes at 150-160°F. All heat scale should be loosened. If not, continue until scale is loosened. However, remove the sheet from the acid etch as soon as possible after 5 minutes and do not leave in the etch for more than 7 minutes.

G. Cold water rinse for 10 minutes. Wipe off smut after 5 minutes; rinse and return to complete rinse.

H. Place the skin in 0.16 oz./gal. of borax and 0.48 oz./gal. soda ash solution at 125-130°F for 30 minutes.

I. Cold rinse for 10 minutes.

J. Air dry vertically.

III. Surface Preparation of Core

A. Cut core to size.

B. Vapor degrease with core in horizontal position. Use best practice.

C. Core shall be held in either the vertical or horizontal position through the heat scaling.

D. Place in furnace at 1000°F for 30 minutes.

E. Remove from furnace and air cool.

F. Place the core horizontally in the 20% HNO_3, 4% HF by volume acid etch bath at 150-160°F to remove the heat scale. The etching should continue for at least 3 minutes; but if at that time the heat scale is not loosened, the etching may continue up to 5 minutes. The core should be removed from the bath as soon as the heat scale is loosened.

G. Rinse in cold water for 10 minutes, agitating the core to remove the heat scale.

H. Place in the 0.16 oz./gal. borax, 0.48 oz./gal. soda ash solution at 125-130°F for 30 minutes.

I. Cold water rinse for ten minutes.

J. Air dry in the horizontal position.

IV. Applying Adhesive to the Facing Sheets

A. Shake the UI 1067-1A slip thoroughly. Do not use slips over 3 days old or slips containing crystals.

B. The spray gun and accessories suggested for this use from this study are:

1. Binks, Model 18, stainless steel spray gun

2. Binks, 64PA, stainless steel cap

3. Binks, 64 VT, stainless steel nozzle

4. Binks, Model 80, pressure cup

C. Fill pressure cup with UI 1067-1A adhesive.

D. The technique for spraying material can be learned only by experience. The following suggestions may assist in developing the technique:

1. The slip should be sprayed neither too wet nor too dry. Slips applied too wet are glossy. Slips applied too dry are grainy.

2. Correctly applied slips will result in bisques which do not crack or powder.

3. Experience has shown that best results are obtained with air pressures of between 40-60 psi and pot pressures of between 2 and 5 psi.

4. The rate of spraying of material, the rate of flow of atomizing air and the distance and angle of the gun are adjusted to control the applied slips. The viscosity of the slip itself will affect all of these adjustments.

E. All spraying is done with the gun normal to the surface.

F. Apply a coat of adhesive by cross coating to result in a 0.004" to 0.005" dry bisque.

G. Blow the surface with the spray gun.

H. Place under heat lamps for 10 minutes at a temperature of approximately 200°F. The temperature should not reach 212°F at any time.

I. Heat in oven at 250°F for 15 minutes.

J. Spray a second coat of the same thickness and dry in the same manner.

K. Spray a third coat of the same thickness. The final bisque thickness should be approximately 0.013" ± 0.002". The thickness may be checked by weight. The total dried bisque should weigh 0.25 ± 0.05 gram/sq. in.

L. Place in the furnace at 1750°F in a horizontal position for 8 minutes.

V. Applying Adhesive to the Core

A. The same general notes on the spraying techniques which apply to spraying the facing sheet apply to the spraying of the core.

B. A sample piece of core should be weighed. This core should be sprayed at the same time and in the same manner as the core being prepared for use. In some cases, the core itself can be weighed for checking purposes.

C. Spray one coat with the core held in such a position that all of cell walls facing the gun are coated. Rotate the core 90° and spray one coat. Rotate the core another 90° and spray one coat; finish by rotating the core another 90° and spraying one coat.

D. Dry the adhesive at approximately 200°F under heat lamps for 10 minutes.

E. Dry in the oven at 250°F for 10 minutes.

F. Spray the opposite side of the core in the same manner per step C and repeat the drying procedures described in steps D and E.

G. Weigh sample piece of core and determine the pickup in grams per square inch of core. If the pickup is insufficient, repeat steps C through G until the desired pickup is achieved. To add small amounts of adhesive, the travel of the gun may be sped up and/or the material flow to the gun may be reduced.

H. The desired pickup of bisque for the 0.500", 1/4" cell, 0.002" foil honeycomb core used in this program is 2.5 to 3.0 grams per square inch of core.

I. Place in the furnace at 1750°F in a vertical position for 4 minutes.

VI. Assembling the Sandwich

A cross section of a typical inorganic adhesive bonding vacuum tool is shown in Figure 1. The tool is essentially a flat stainless steel trough in which a sandwich to be bonded is enclosed and held under vacuum which applies a differential atmospheric pressure to the tool. The "Glasrock" base separated from the panel by an insulating "Fiberfrax" sheet and a stainless steel sheet supports the panel. Heat balance is achieved by placing a slip sheet, a "Fiberfrax" sheet and a duplicate sized "Glasrock" block on top of the panel. A 0.012" stainless steel lid is welded airtight to flange of the tool.

The assembly procedure is:

A. Place the "Glasrock" base at the bottom of the trough.

B. Place one thickness of 0.080" thick "Fiberfrax" sheet on the "Glasrock", followed by a 0.010" thick slip sheet.

C. Assemble the sandwich.

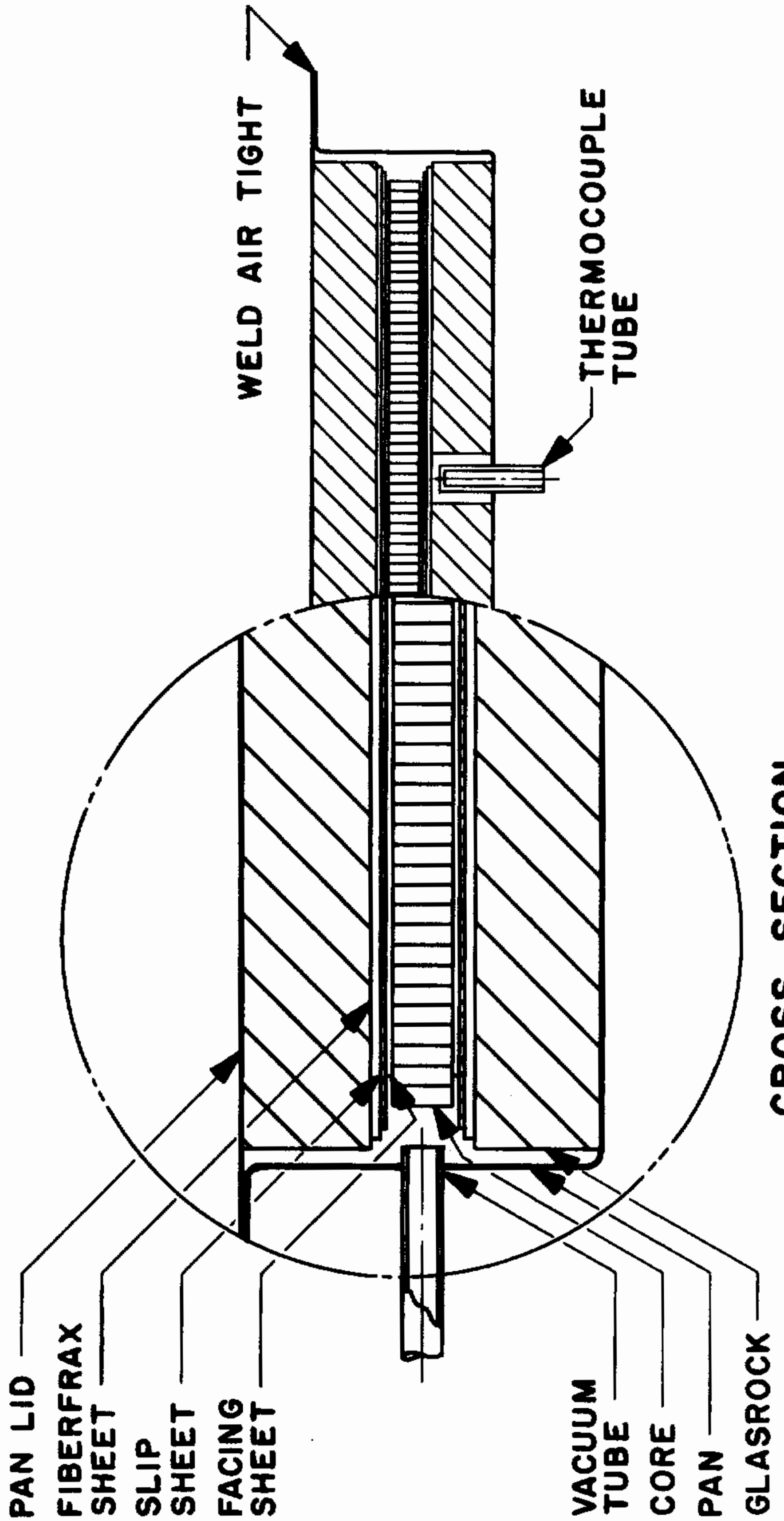
D. Place another slip sheet on the sandwich, followed by a second "Fiberfrax" sheet.

E. Place the second "Glasrock" slab on the "Fiberfrax" sheet to complete the layup.

F. Locate the lid properly and hold in place with weights. Weld the lid airtight onto the pan flange.

G. Pull a 5 psi vacuum on the envelope and allow no air to flow through the envelope.

H. Place the tool in the furnace and heat the panel to the proper temperature (1700-1750°F) as rapidly as possible and hold in this temperature range for 10 minutes.



CROSS SECTION
TYPICAL CERAMIC ADHESIVE BONDING
VACUUM PRESSURE TOOL

FIG. 1

I. Remove the tool from the furnace and air cool rapidly under a vacuum of 7 pounds until the panel temperature is 200°F.

J. Remove the panel from the tool, cool to -100°F and hold at this temperature for at least 8 hours.

K. Age the panel at 950°F for 60 minutes.

RESULTS

I. Evaluation of UI 117-63 Adhesive

A. Familiarization Tests

Tensile shear tests were made of lap shear specimens of heat treatable stainless steel bonded with UI 117-63 adhesive. This was done to compare tensile shear results obtained at Aeronca to those reported by the University of Illinois.

The tensile shear test results of specimens made in the second firing are summarized below. The test results of specimens made in Firing #1 and Firing #2 are reported fully in Table **I and II.**

Average Tensile Shear Strength - psi

Test Temp.	Aeronca Results 17-7 PH	Aeronca Results PH 15-7 MO	U.I. Results 17-7 PH
Room	2000	2140	1505
600°F	1830	1400	1480
800°F	1520	1520	1415
1000°F	1500	1540	1425

NOTE: Each joint contained an .008" 28 mesh x 28 mesh 304 stainless steel screen.

B. Selection of Stainless Steel for the Test Program

During the familiarization tests it was noted that the PH 15-7 MO stainless steel test specimens warped during and after the heat treatment, whereas the 17-7 PH specimens did not under the same conditions. It was therefore decided to use 17-7 PH steel for the balance of the program.

C. Bend Tests

One-half square inch lap shear specimens of 17-7 PH steel bonded with UI 117-63 adhesive were tested in bend by single point loading on a 1-1/2" span at room temperature, 600°F, 800°F and 1000°F. The results are summarized below and are detailed in Table **III.**

For comparison, it should be noted that MIL-A-8431 requires 130 pounds minimum bend load for high temperature resistant organic adhesives tested at room temperatures.

Bend Strength of Adhesive UI 117-63
Lap Shear Specimens

Test Temperature	Average Bend Strength - lb.
Room	89
600°F	77
800°F	80
1000°F	87

D. Tensile Shear Strengths of Bond Joint After Various Exposures

Standard lap shear specimens made with 17-7 PH steel and bonded with UI 117-63 adhesive were immersed in various fluids or exposed to simulated environment for the total time indicated. After exposure, tensile shear strengths were determined at the temperatures specified. The results are summarized below, shown in bar charts in Figure 2, and detailed in Tables IV and V.

Average Tensile Shear Reported as Percent Of Standard* Tensile Shear Results at Room Temperature

		Test Temperature		
Exposure	Room	600°F	800°F	1000°F
A	105	110	94	128
B	72	88	82	121
C	123	102	74	116
D	94	107	103	124
E	110	96	104	115
F	105	86	79	115

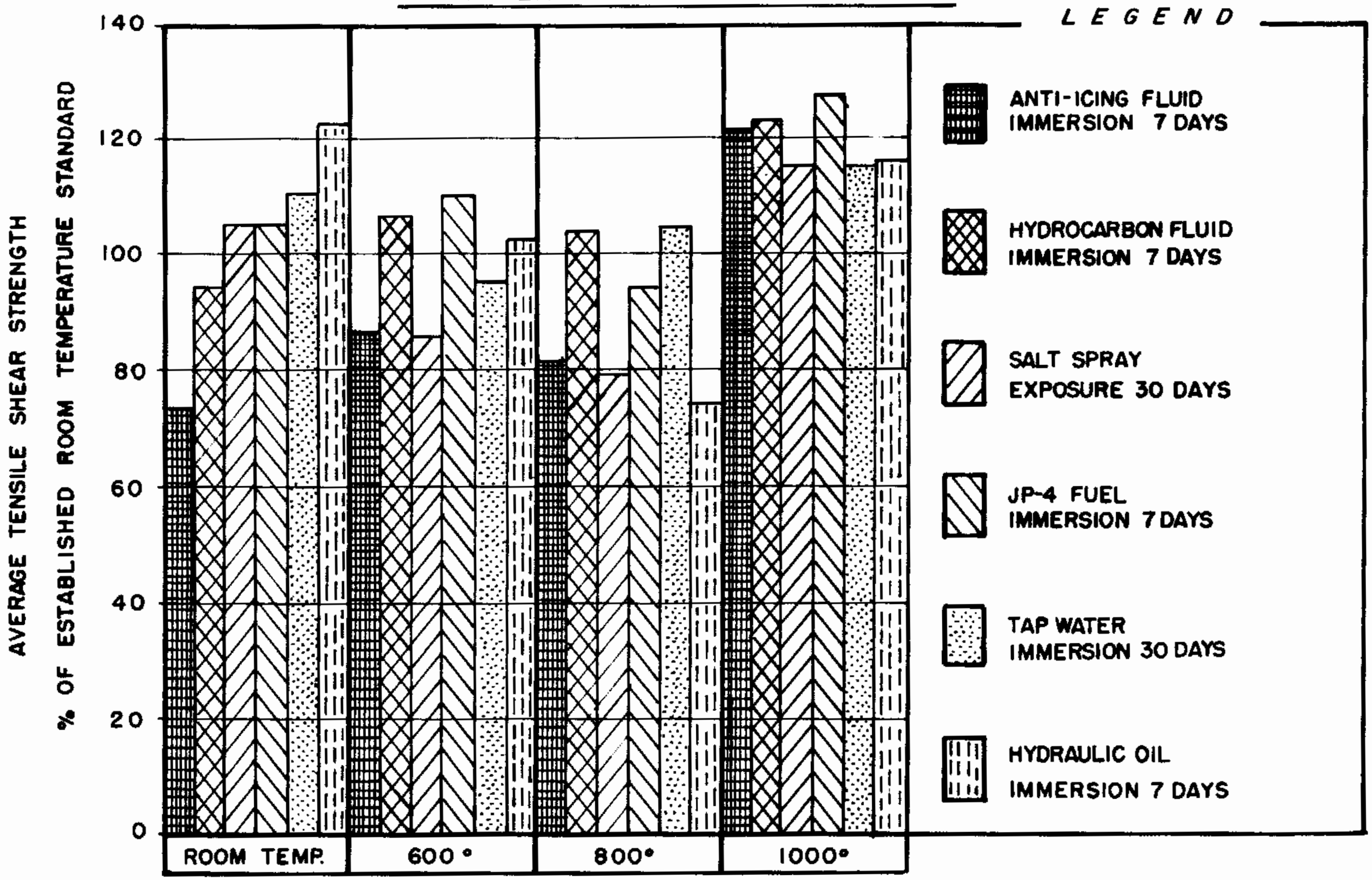
Exposures Defined:

A - 7 days immersion in JP-4 fuel, MIL-F-5624
B - 7 days immersion in anti-icing fluid, MIL-F-5566
C - 7 days immersion in hydraulic oil, MIL-O-5606
D - 7 days immersion in hydrocarbon fluid, MIL-H-3136, Type III

*See "Testing Procedures" for determination of standard tensile shear used in these comparisons.

TENSILE SHEAR STRENGTH

CERAMIC BONDED LAP SHEAR SPECIMENS AT VARIOUS TEST TEMPERATURES AFTER EXPOSURE TO VARIOUS MEDIA



TEST TEMPERATURE —°F

FIGURE 2

E - 30 days immersion in tap water
F - 30 days exposure to salt spray per QQ-M-151

No definite conclusion can be drawn on the effect of any immersion medium because test results show a large scatter. This scatter was evident on all data whether from tests made with unimmersed or immersed specimens. The extent of this scatter is shown in Figure 3. In three firings, the tensile shear strength reported for unimmersed specimens varied from 880 to 1670 psi. The scattering for a single firing varied less; for example, ranging from 1040 to 1240 psi in Firing #6, and from 1250 to 1670 psi in Firing #8. For this reason, tensile shear results for immersed samples are reported on a basis of the percentage of the tensile shear strengths of unimmersed samples fired at the same time and tested at room temperature.

However, within the significance of the test results, none of the exposures appear to affect the tensile shear strength of the bond joint.

E. Boron Embrittlement

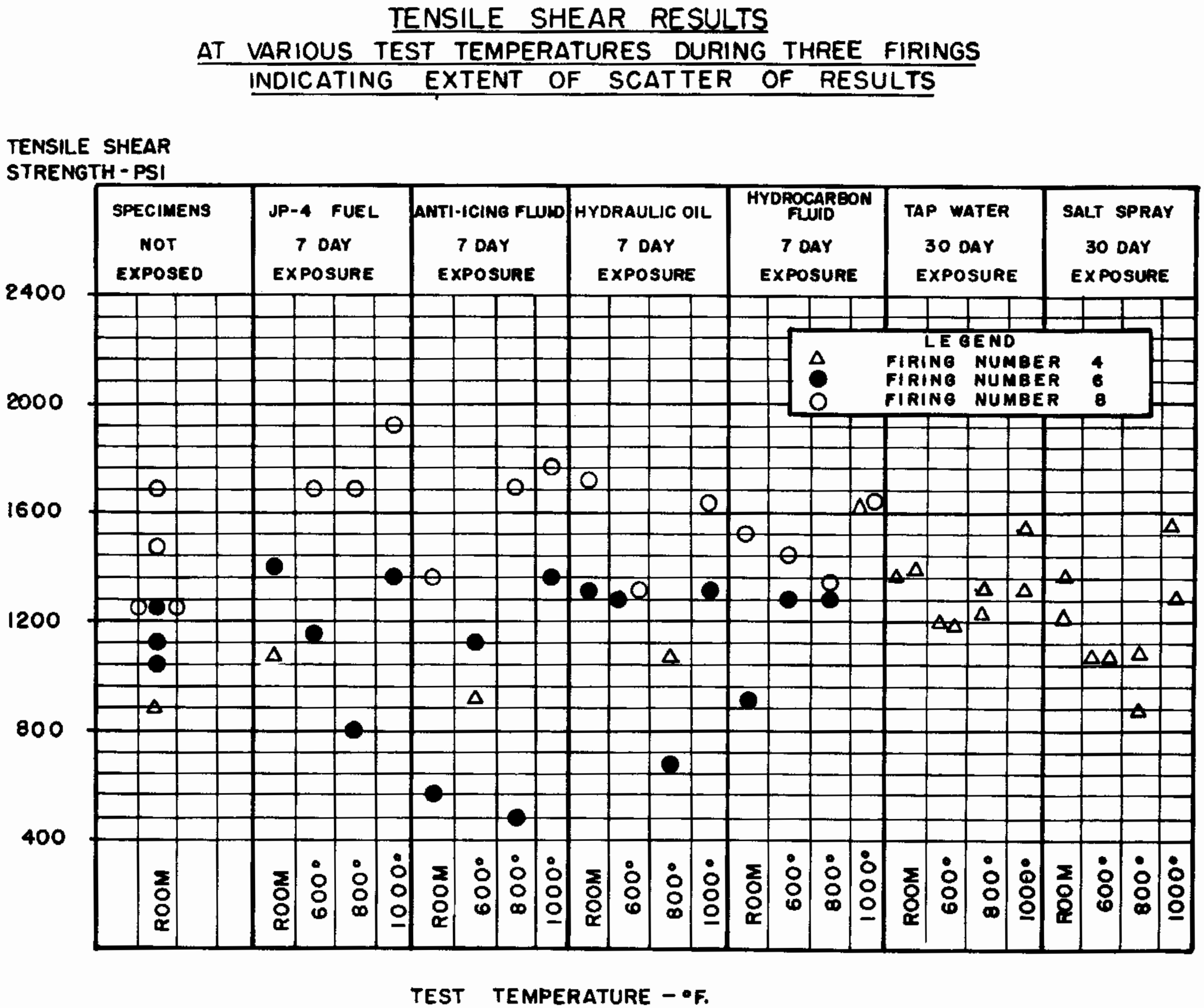
The large percentage of boron oxide in the UI 1067-1 adhesive suggested that there might be a possibility of boron embrittlement of the steel caused by this adhesive and others containing high percentages of boron oxide. Microscopic studies were made of sections of 17-7 PH core and sheet from honeycomb sandwiches fired with high boron oxide content adhesive UI 1067-1. These studies indicated no evidence of boron embrittlement.

Photomicrographs of the adhesive bond on 17-7 PH steel are shown in Figures 4 and 5.

F. Effect of Ceramic Coatings on the Tensile Strength of 17-7 PH Foil

Half-inch wide tensile coupons of .005" 17-7 PH stainless steel foil, condition A, were ceramic coated, fired and aged in accordance with Armco Steel Corporation's recommended practice for converting 17-7 PH steel to the RH 950 condition, except that air rather than inert atmosphere was used. Uncoated samples were prepared and tested in the same manner.

The specimens were tested to failure in tension and the ultimate strength calculated on the basis of the original base metal cross section. The purpose of these tests was to determine if the coatings affected the apparent tensile strength of the base metal. It was assumed that when firing in an air atmosphere an improvement in apparent tensile strength of a coated steel over an uncoated steel


TENSILE SHEAR RESULTS
AT VARIOUS TEST TEMPERATURES DURING THREE FIRINGS
INDICATING EXTENT OF SCATTER OF RESULTS
TENSILE SHEAR STRENGTH - PSI
2400
2000
1600
1200
800
400
SPECIMENS NOT EXPOSED
JP-4 FUEL 7 DAY EXPOSURE
ANTI-ICING FLUID 7 DAY EXPOSURE
HYDRAULIC OIL 7 DAY EXPOSURE
HYDROCARBON FLUID 7 DAY EXPOSURE
TAP WATER 30 DAY EXPOSURE
SALT SPRAY 30 DAY EXPOSURE
LEGEND
FIRING NUMBER 4
FIRING NUMBER 6
FIRING NUMBER 8
ROOM
600°
800°
1000°
TEST TEMPERATURE — °F.
FIGURE 3

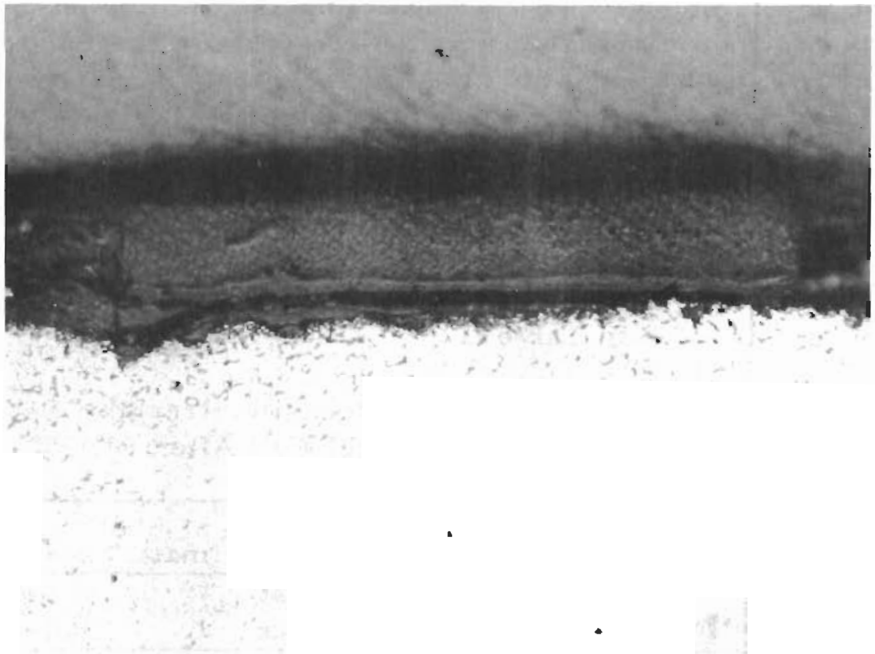


FIGURE 4

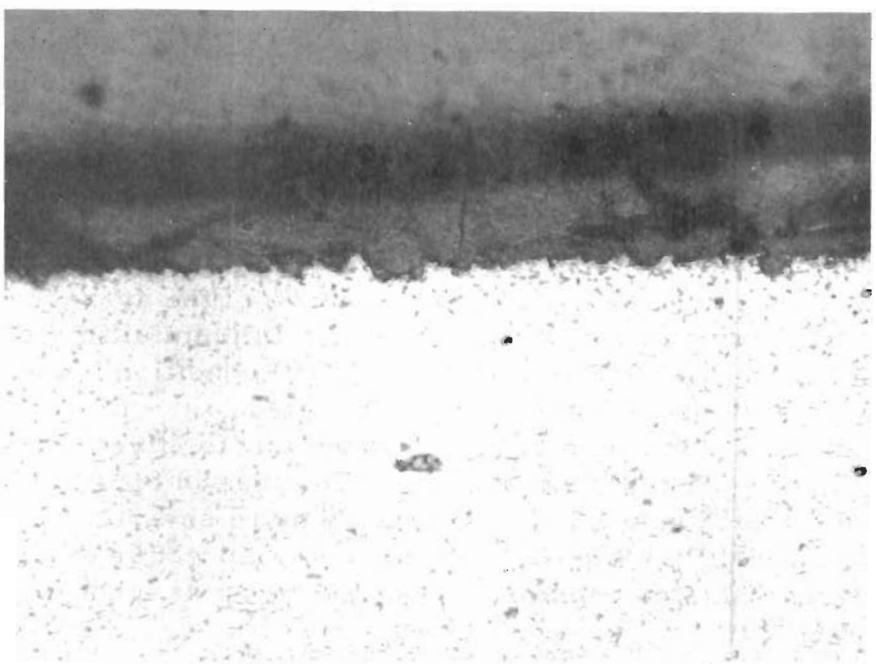


FIGURE 5

PHOTOMICROGRAPHS OF
CERAMIC ADHESIVE BOND

MAG. 500X, ETCHED 10% OXALIC ACID

could be attributed to the coating acting as a corrosion protection, and that a loss in apparent tensile strength of a coated steel over an uncoated steel could be attributed to a lack of corrosion protection and the promotion of corrosion of the steel.

The two coatings applied were UI 1067-1, a high boron oxide content ceramic, and UI 117-63, a moderate boron oxide content ceramic containing carbonyl iron and silicon powders. The average tensile strength of the uncoated and coated specimen is reported below and detailed in Table VI.

Apparent Ultimate Tensile Strength Of Coated 17-7 PH Steel Foil After RH 950 Treatment

Coating Applied	Tests	Average	Apparent Ultimate Tensile Strength: Highest Individual psi	Highest Individual % from Avg.	Lowest Individual psi	Lowest Individual % from Avg.
None	5	177,000	211,000	19.2	159,000	10.2
UI 117-63	6	149,000	185,000	24.1	100,000	33.0
UI 1067-1	6	209,000	213,000	2.0	206,000	1.5

The foil coated with UI 1067-1 attained approximately the expected minimum tensile strength (210,000 psi) for 17-7 PH steel in the RH 950 condition. The results for the six tests were very consistent, no one result varying more than 2% of the average. The uncoated foil did not attain the expected minimum ultimate tensile strength for 17-7 PH steel in the RH 950 condition averaging 15.3% below the results for the UI 1067-1 coated foil. The spread in reported tensile strength of the uncoated foil for five tests varied as much as 19.2% from the average. The loss in tensile strength of the foil coated with UI 117-63 is even more severe, averaging 28.7% less than the UI 1067-1 coated foil. The spread in test results for six tests was also greater, one result being as much as 33% from the average.

Under the conditions of these tests, the UI 1067-1 afforded good corrosion protection to the 17-7 PH steel when heat treated in air atmosphere. The UI 117-63 promoted corrosion under the same conditions.

II. Reproducibility Studies

A comprehensive study was undertaken to evaluate the factors that affect the tensile shear properties of the ceramic bonded joint and to determine how to control them in order to reduce the scatter of results.

A. Slip Control

See Table VII for test data accumlated during this study and Table VIII for slip compositions used.

1. Dip Weight Versus Viscosity

At 30 to 48% water content, the dip weight increases consistently with the viscosity of the UI 117-63 slip without metal additions independent of the water content. See Figure 6.

2. The Effect of Aging on the Viscosity of the Slip

The viscosity of a slip of UI 117-63 containing no metal additions increases with age. As the slip ages, the viscosity apparently approaches a fixed value asymptotically. The rate of increase in viscosity and the viscosity which is approached after aging are dependent upon the water content of the slip. This is shown in Figure 7. For some slips, it was noted that at approximately three days, the slip gel broke down and crystals and agglomerates (which may contain crystals) formed. Examples of these are shown in Figure 8.

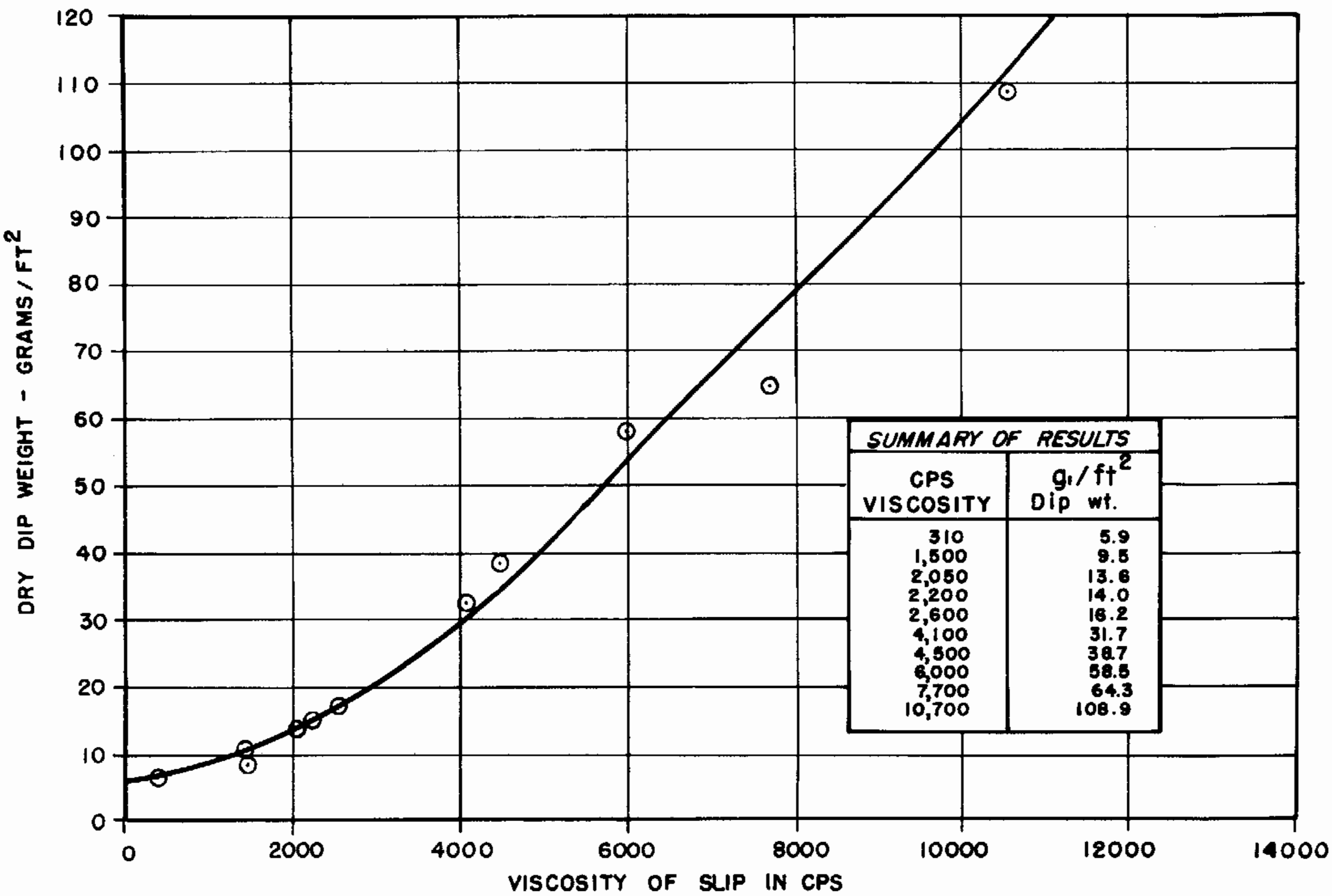
3. Relationship Between pH of the Slip and the Na_2O and B_2O_3 Dissolved in the Mill Liquor

Previous investigators have suggested using pH and the dissolved Na_2O and B_2O_3 as an index of physical properties of the slip. Tests reported herein indicate that the pH of the slip varies with the dissolved Na_2O/B_2O_3 in the mill liquor. See Figure 9. The pH of the slip increases during aging and reaches a fixed value in approximately 4 days. The resultant pH after aging varies from 7.5 to 7.85. The viscosity of the slip does not vary with the pH, though both increase as the slip ages. After 4 days, the viscosity of most slips (in particular those with low water content) continues to increase whereas the pH does not increase.

4. Effect of Selected Electrolytes and Organics for Reducing "Set"

The set of a ceramic slip may be reduced by additions of specific inorganic acids and salt solutions. These tests were run

DRY DIP WEIGHT VS SLIP VISCOSITY
(UI 117-63 SLIP WITHOUT METAL POWDERS)



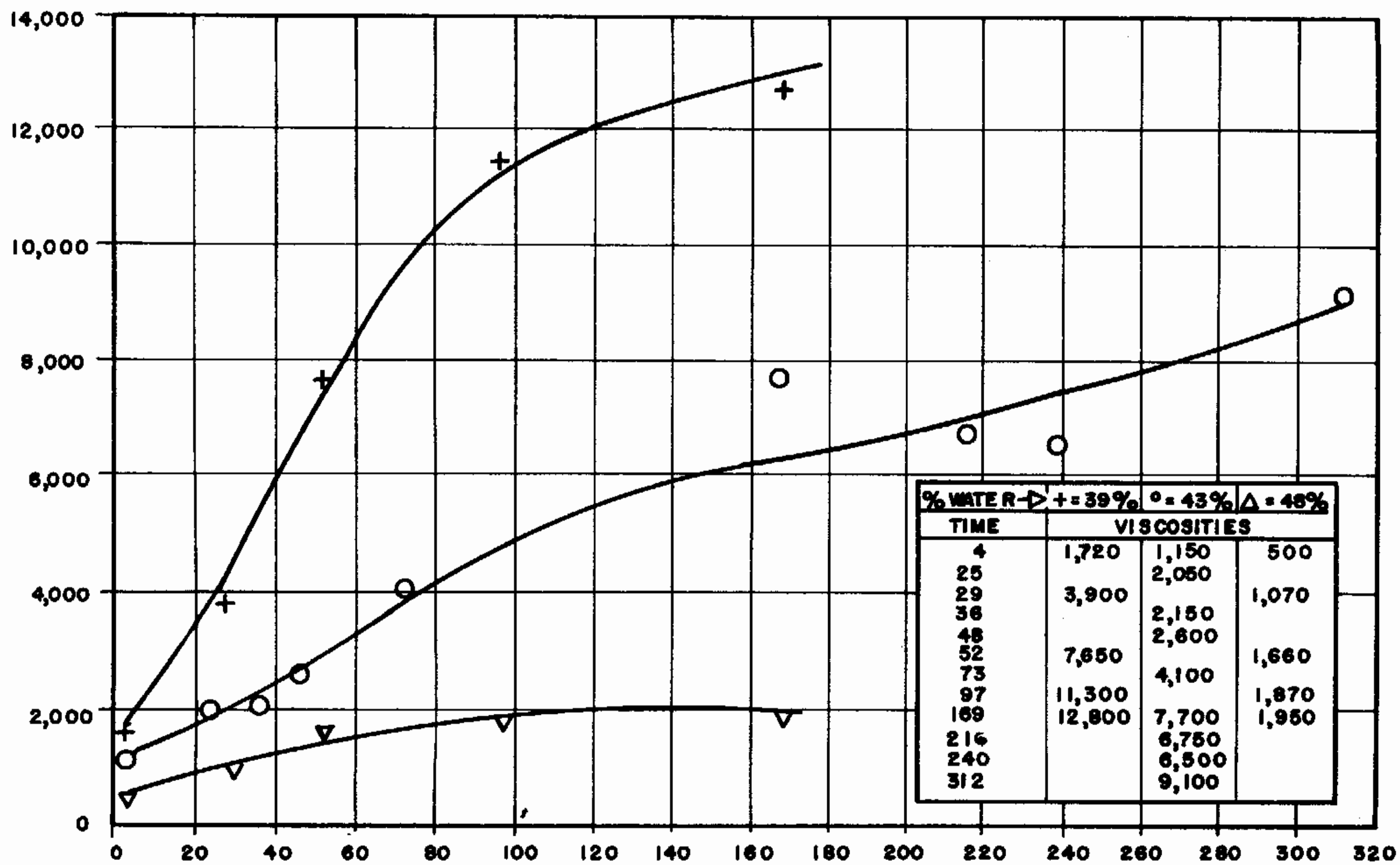
SUMMARY OF RESULTS

CPS VISCOSITY	g_1/ft^2 Dip wt.
310	5.9
1,500	9.5
2,050	13.6
2,200	14.0
2,600	16.2
4,100	31.7
4,500	38.7
6,000	58.5
7,700	64.3
10,700	108.9

FIGURE 6

AGING EFFECT ON VISCOSITY

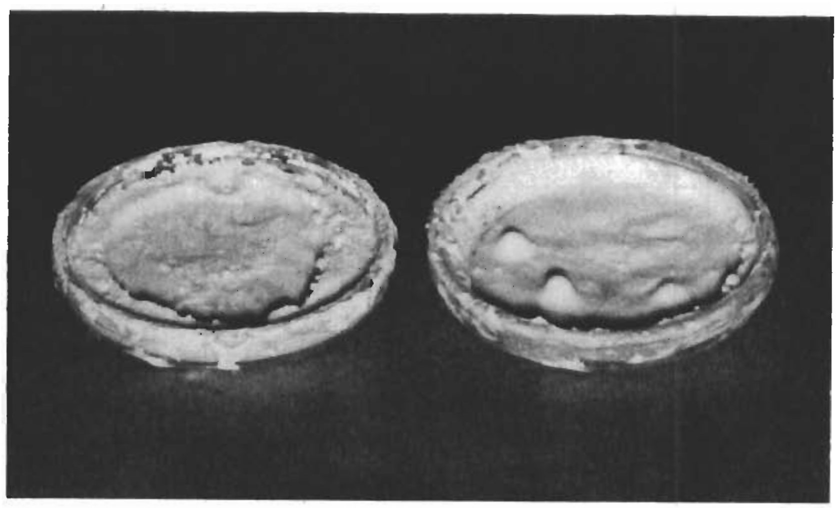
(UI 117-63 SLIPS WITH METAL ADDITIONS)



% WATER →	+ = 39%	○ = 43%	△ = 48%
TIME	VISCOSITIES		
4	1,720	1,150	500
25		2,050	
29	3,900		1,070
36		2,150	
48		2,600	
52	7,650		1,660
73		4,100	
97	11,300		1,870
169	12,800	7,700	1,950
216		6,750	
240		6,500	
312		9,100	

TIME IN HOURS (FROM START WET MILLING)

FIGURE 7



CRYSTAL DEPOSITS

AGGLOMERATION

EXAMPLES OF CRYSTAL DEPOSITS (LEFT) AND AGGLOMERATION (RIGHT) UPON THE AGING OF MIXED SLIPS

FIGURE 8

Na_2O / B_2O_3 OF MILL LIQUOR FROM UI 117-63 SLIP WITHOUT METAL FILLERS VS pH OF SLIP. (FOR TWO SLIPS OF VARYING SOLID CONTENT.)

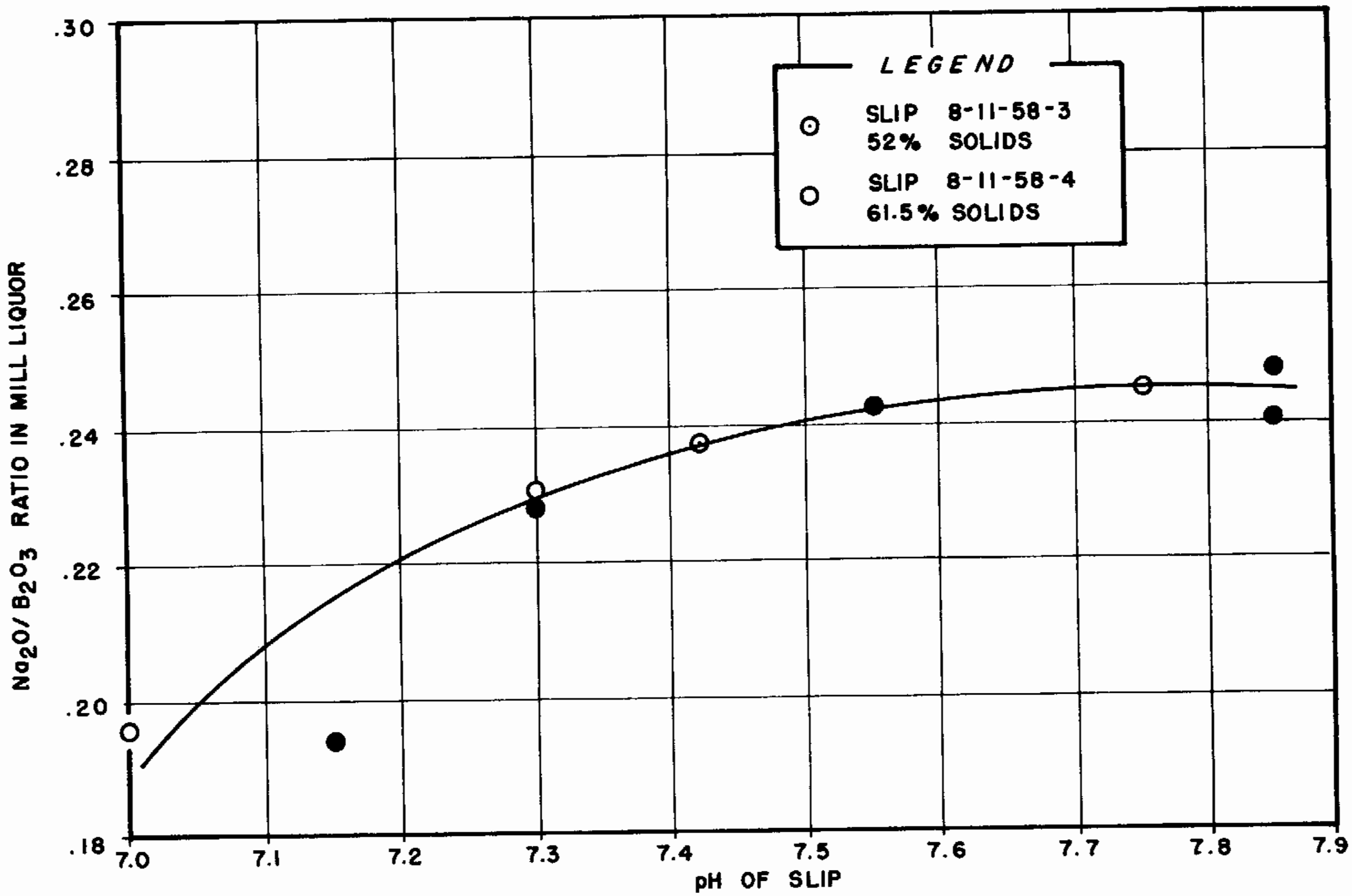


FIGURE 9

on slips made by mixing equal parts of UI 117 and UI 1067 frits. The solutions are listed in order of decreasing effectivity: Sodium pyrophosphate (saturated), six normal acetic acid, two normal hydrochloric acid, two normal nitric acid. All acids colored the solution yellow, indicating a strong chemical reaction. For this reason, acids are not recommended for this use. Two normal sodium hydroxide, ethyl alcohol, and N-butyl alcohol are not effective as set reducers.

A saturated solution of sodium pyrophosphate is recommended as an electrolyte for reducing the set of a UI 117-63 slip. The data are reported in Table IX.

B. Relation of Bond Line Thickness to Tensile Shear Strength

Three sets of lap shear specimens were fired to determine the relationship between bond line thickness and tensile shear strength. Four conclusions concerning joints made with UI 117-63 adhesive may be drawn from tests run on these specimens:

(1) The tensile shear strength increases as the bond line thickness decreases.

(2) Minimum bond line thickness may not necessarily result in optimum tensile shear strength since a thin bond line may only indicate an adhesive starved joint.

(3) The firing temperature affects the tensile shear strength of a lap shear joint. Samples fired at 1750°F had higher tensile shear strengths for joints with comparable thin bond line thicknesses than those fired at 1650°F.

(4) The tensile shear strength of a lap shear joint is dependent upon the bond line pressure only to the extent that the bond pressure affects the resultant bond line thickness.

1. Lap Shear Specimens Fired at 1650°F - First Set

Five series of 17-7 PH steel lap shear specimens were fired at 1650°F for 10 minutes. The UI 117-63 adhesive joint included an .008" 28 mesh 304 stainless steel wire screen. Each series differed from each other in the following particulars:

First 1650°F Series No.	Bond Pressure psi	Bisque Thickness On Each Coupon Inch	Firing Number
1	50	.008"	2nd
2	50	.013"	2nd
3	50	.023"	1st
4	50	.032"	1st
5	23.5	.017"	3rd

The results are summarized below and detailed in Table X.

Avg. Tensile Shear Strength vs. Bond Line Thickness
Firing Temperature - 1650°F

Bond Line Thickness Inch	Coupon Bisque Thickness Inch	Bond Pressure Used psi	Avg. Tensile Shear Strength Room Temp. psi	No. Tests In Avg.
.008	.008	50	1170	1
.0085	.008	50	1280	4
.0095	.008	50	1480	1
.010	.013	50	1310	3
.012	.013	50	1410	3
.012	.023	50	1590	1
.013	.023	50	1430	2
.014	.023	50	1330	3
.0145	.017	23.5	1220	2
.015	.032	50	800	1
.015	.017	23.5	980	2
.016	.032	50	610	2
.0165	.017	23.5	810	1
.017	.032	50	500	2
.019	.032	50	350	1

The individual results are charted in Figure 10. One curve signified by a broken line indicates the apparent relationship between bond line thickness and tensile shear strengths. However, the apparent maximum tensile shear strength at .012" is not consistent with the balance of the tests run in this program.

The bond joints made with the thin adhesive bisques of .008" and .013" may not contain sufficient adhesive and this may have lower tensile shear strengths. If the values for .008" and .013" bisque thickness are omitted, a curve representing the balance of values (indicated by the solid line) does not show a maximum at .012" bond line thickness and has the general shape of the

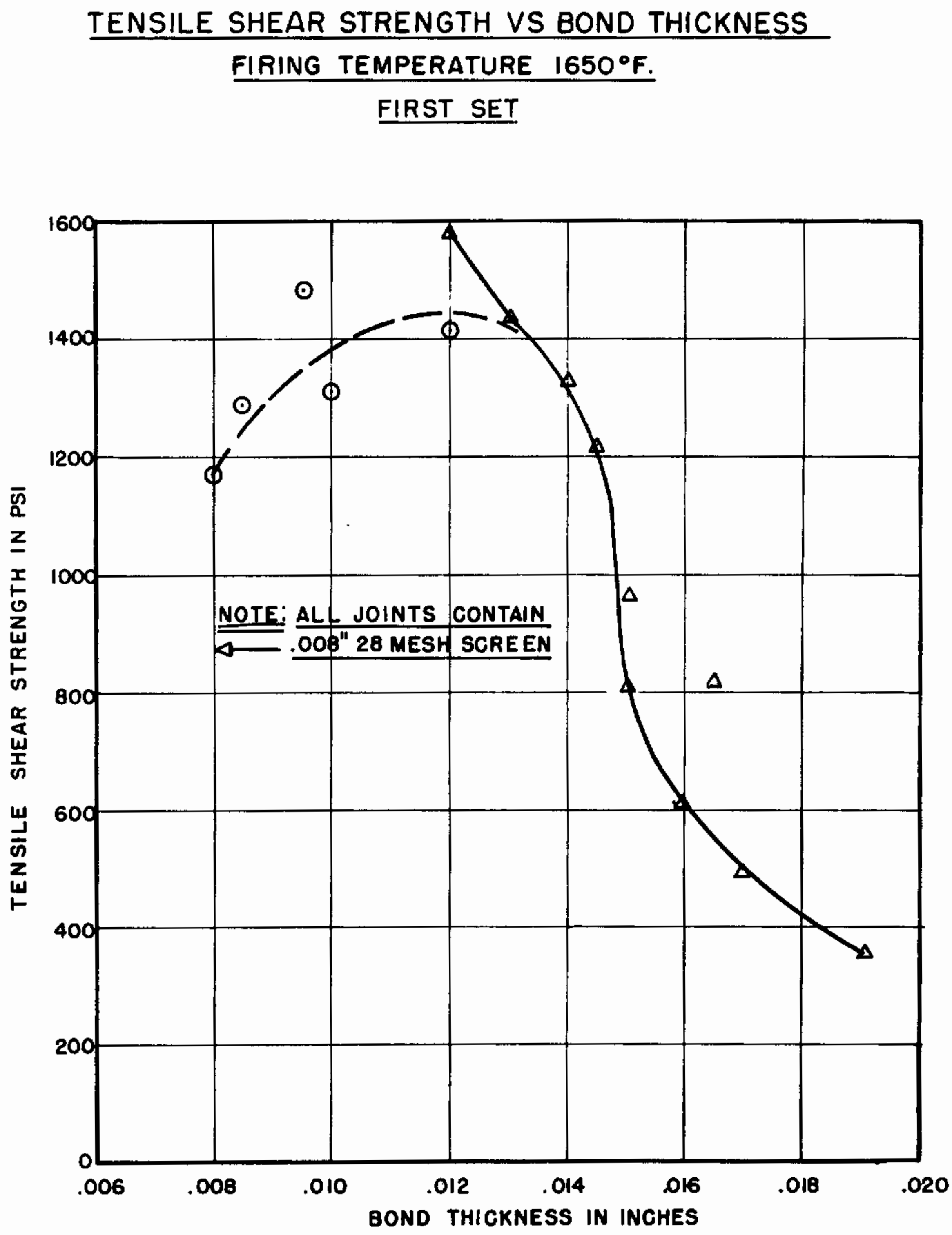

TENSILE SHEAR STRENGTH VS BOND THICKNESS
FIRING TEMPERATURE 1650°F.
FIRST SET
1600
1400
1200
1000
800
600
400
200
0
TENSILE SHEAR STRENGTH IN PSI
NOTE: ALL JOINTS CONTAIN
.008" 28 MESH SCREEN
.006
.008
.010
.012
.014
.016
.018
.020
BOND THICKNESS IN INCHES

FIGURE 10

curves based on data for firings at 1650°F and 1750°F. A second set of tests was made to check this assumption.

2. Lap Shear Specimens Fired at 1650°F - Second Set

Nine series of 17-7 PH steel lap shear samples were produced in three firings at 1650°F for 10 minutes, each firing using a different bond line pressure; i. e., 50, 30 and 10 psi. The UI 117-63 adhesive joint included an .008" 28 x 28 mesh 304 stainless steel wire screen. Each series differed from the other in the following particulars:

Second 1650°F Series No.	Bond Pressure psi	Bisque Thickness On Each Coupon Inch	Firing Number
1	10	.014	1st
2	10	.021	1st
3	10	.032	1st
4	30	.014	2nd
5	30	.021	2nd
6	30	.032	2nd
7	50	.014	3rd
8	50	.021	3rd
9	50	.034	3rd

The results are summarized below, detailed in Table XI, and illustrated in Figure 11.

Tensile Shear Strength vs. Bond Line Thickness
Firing Temperature - 1650°F

Bond Line Thickness Inch	Average Tensile Shear Strength At Room Temperature psi	No. of Tests in Average
.008	1910	1
.009	1740	2
.011	1580	1
.012	1350	2
.013	1360	5
.014	1100	1
.015	1100	2
.016	960	2
.017	870	1
.019	480	1

TENSILE SHEAR STRENGTH vs BOND LINE THICKNESS

FIRING TEMPERATURE 1650°F.

SECOND SET

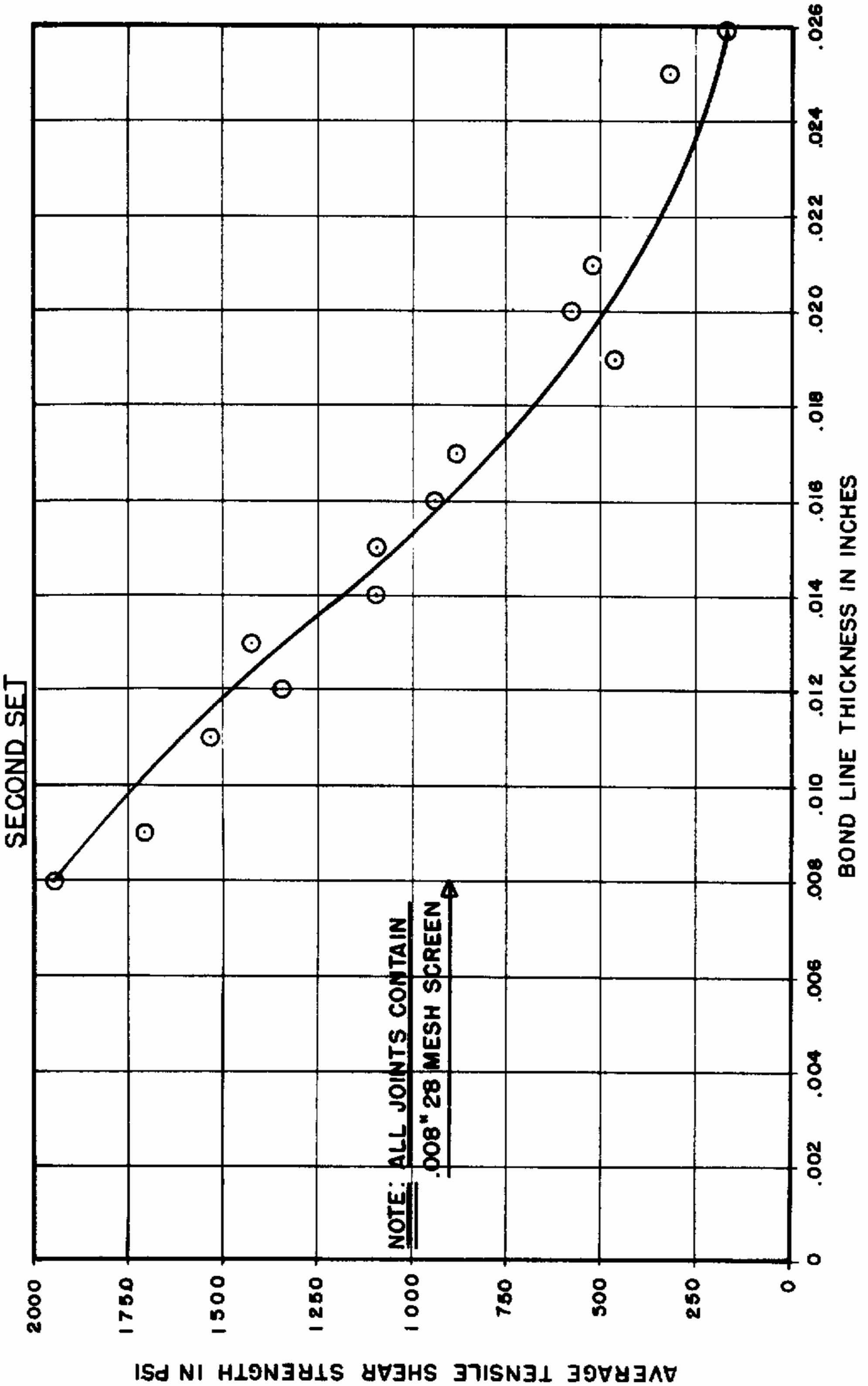


FIGURE 11

(continued)

Bond Line Thickness Inch	Average Tensile Shear Strength At Room Temperature psi	No. of Tests In Average
.020	640	2
.021	510	1
.025	310	1
.026	170	1

3. Lap Shear Specimens Fired at 1750°F

Nine series of 17-7 PH steel lap shear samples were produced in three firing at 1750°F for 10 minutes, each firing using a different bond line pressure; i.e., 50, 30 and 10 psi. The UI 117-63 adhesive joint included an .008" 28 x 28 mesh 304 stainless steel wire screen. The nine series differed from each other in the same details as the nine series for 1650°F differed.

The results are summarized below, detailed in Table XII and illustrated in Figure 12.

Tensile Shear Strength vs. Bond Line Thickness
Firing Temperature - 1750°F

Bond Line Thickness Inch	Average Tensile Shear Strength At Room Temperature psi	No. of Tests In Average
.009	2290	2
.010	1860	5
.011	1920	2
.012	1870	3
.013	1930	3
.014	1370*	3*
.017	780	1
.018	630	2
.020	430	1
.024	240	1

*If extremely low result of 560 psi is omitted from this average, the two remaining results average 1780 psi.

TENSILE SHEAR STRENGTH vs BOND LINE THICKNESS

FIRING TEMPERATURE 1750° F.

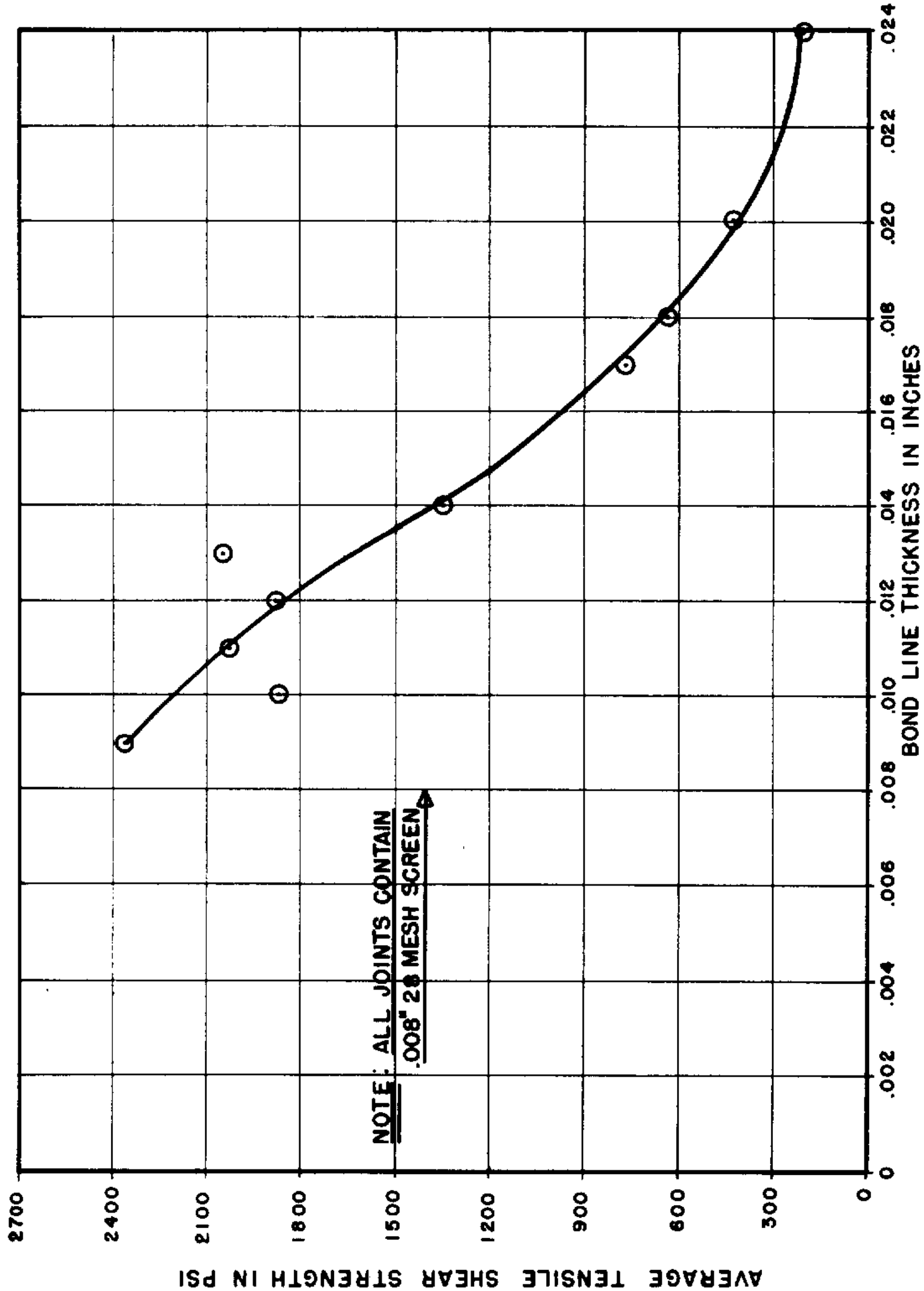


FIGURE 12

C. Effect of Bond Line Pressure on Tensile Shear Strengths of Inorganic Adhesive Lap Shear Joints

As demonstrated in the series of tests made to determine the relation between bond line thickness and the ultimate tensile shear strength of a lap shear joint, the tensile shear strength of a lap shear joint will depend upon the bond line pressure only to the extent that the bond line pressure affects the resultant bond line thickness.

III. Process Development

A. Temperature Survey of the Firing Furnace

A temperature survey was made during a firing in the "Hevi-Duty" furnace of ten points on individual lap shear specimens in various positions in the pressure fixture.

Temperatures recorded for the ten points are shown in Figure 13, and detailed in Table XIII. The maximum spread in temperature at any time between 1 to 3-1/2 hours in the oven, and in temperatures ranging from 1330°F to 1724°F was 60°F.

This survey indicates that the temperature gradients expected during firing are satisfactory.

B. Maturing Temperature Survey

A maturing temperature study disclosed:

1. Firing the UI 117-63 slip without metal powder additions indicated that the maturing temperature was approximately 1600°F for fifteen minutes exposure to temperatures. The appearance of the test specimens are illustrated in Figure 14.

2. The table below and Figure 16 summarize observations made when the same UI 117-63 slip without metal powder additions was fired at various temperatures for extended periods of time. Figure 17 illustrates the appearance of the test specimens.

Effect of Varying Time & Temperature On Quality of Glass Made of Mixed Slip of Adhesive UI 117-63

Firing Temp. °F	Maturing Time at Temp. (Minutes)	Overfiring Starts (Minutes)
1550	35	+ 125 minutes
1600	22*	112*
1650	12*	66*
1700	5	40

*Interpolated Data

TEMPERATURES
10 LOCATIONS IN
LOADED LAP SHEAR PRESSURE TOOL DURING FIRING

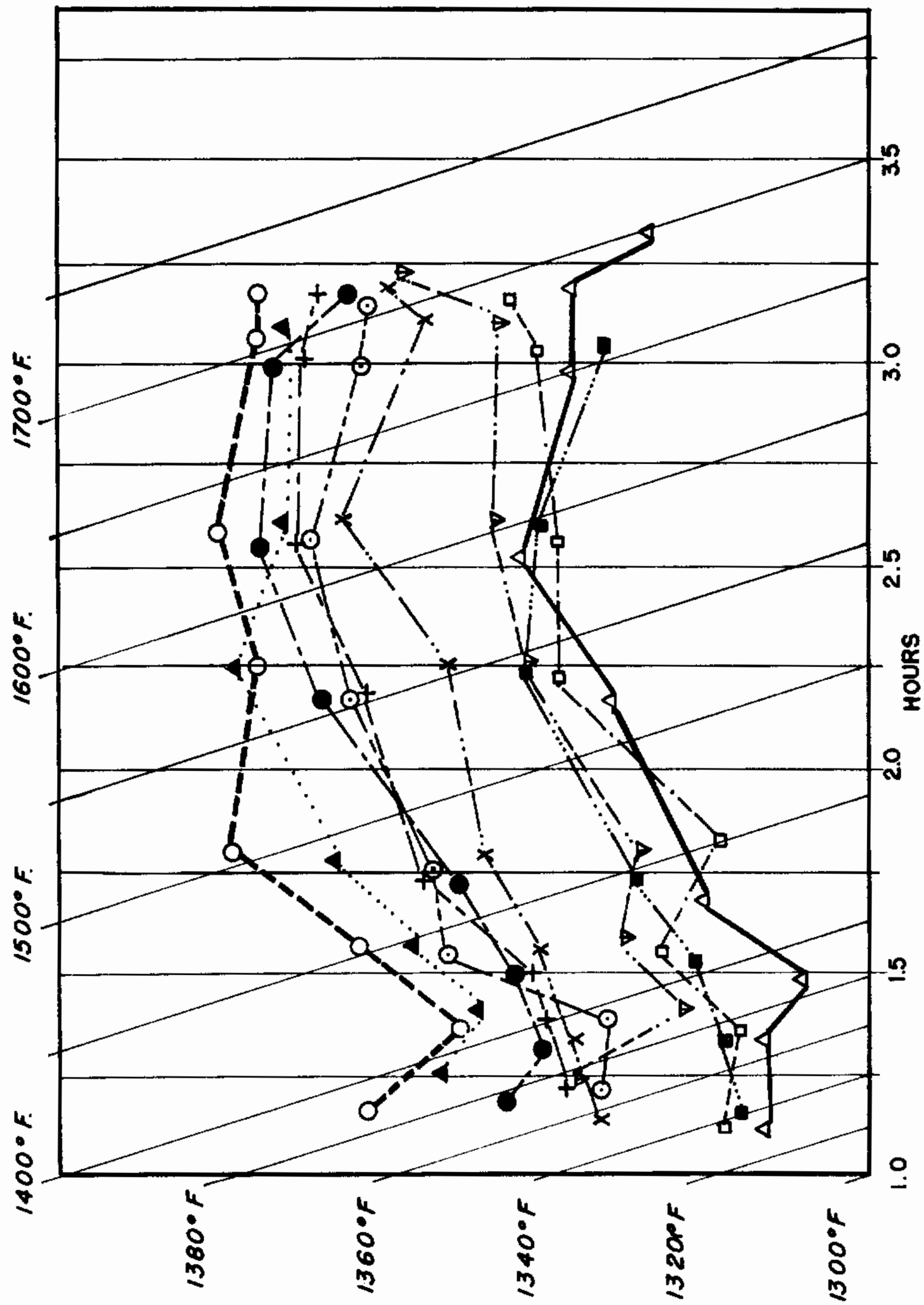


FIGURE 13

FIRED ENAMELED SPECIMENS
SHOWING MATURING TEMPERATURE

1400° 1500° 1600° 1675° 1750°
FIRING TEMPERATURE (°F.)

FIGURE 14

FIRED ENAMELED SPECIMENS
SHOWING CRUMBLING OF 117-63 ADHESIVE

10 15 20 25 45 85 125
FIRING TIME IN MINUTES

FIGURE 15

TIME AND TEMPERATURE VARIATION EFFECT
ON QUALITY OF GLASS MADE OF UI 117-63 SLIP WITHOUT METAL ADDITIONS

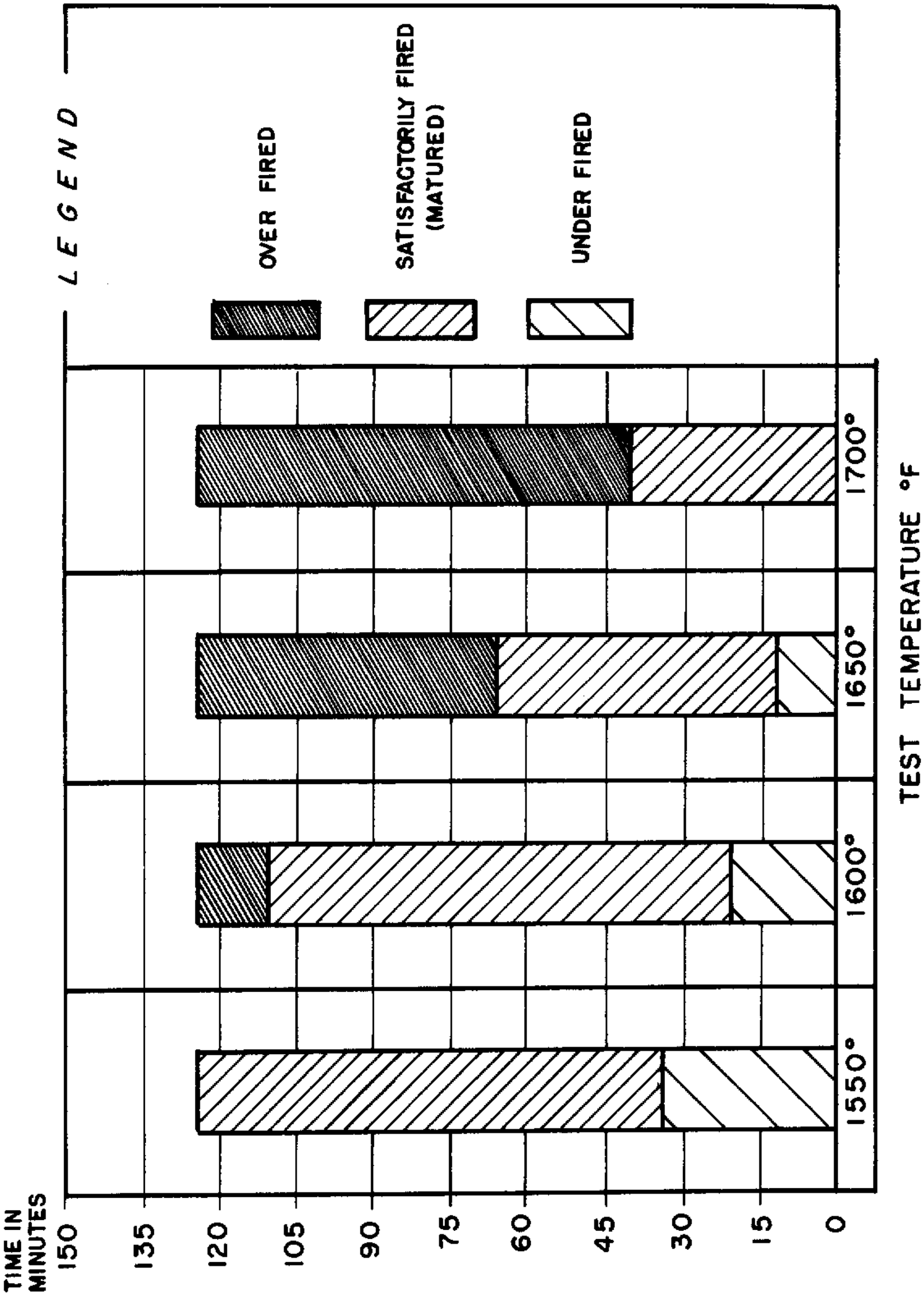
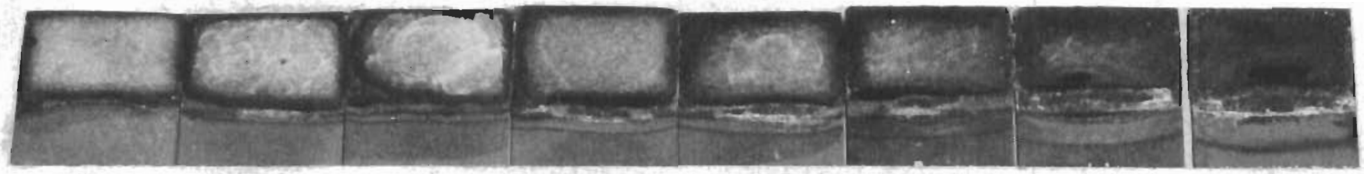


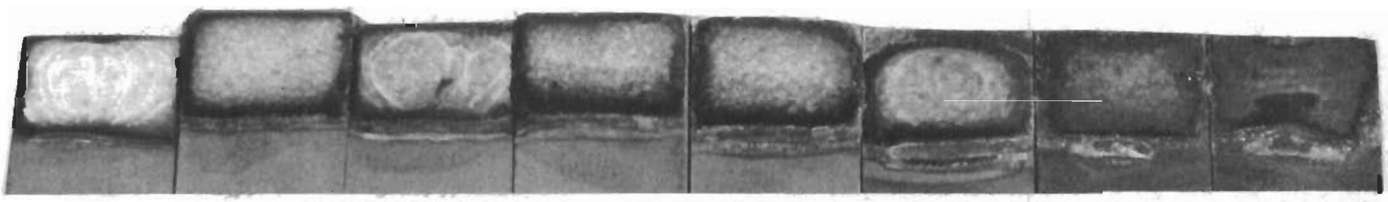
FIGURE 16

FIRED ENAMELED SPECIMENS
MATURING AT DIFFERENT TEMPERATURES AND EXPOSURES



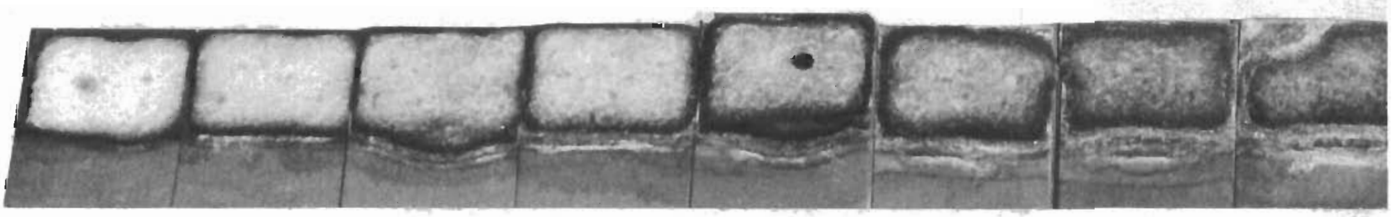
5 10 15 20 25 45 85 125

MINUTES AT 1700°F



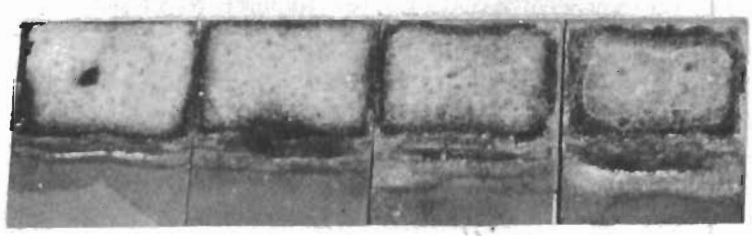
5 10 15 20 25 45 85 125

MINUTES AT 1650°F



5 10 15 20 25 45 85 125

MINUTES AT 1600°F



25 45 85 125

MINUTES AT 1550°F

FIGURE 17

3. Similar firings were made at 1700°F and 1750°F using UI 117-63 slip containing the carbonyl iron and silicon powders. A chemical reaction took place which caused a physical reaction with the adhesive. This resulted in an irregular surface which cracked and spalled upon cooling. See Figure 15. This study indicated that the additon of the metal additives in the ceramic adhesive promoted excessive oxidation where samples are fired unprotected in air atmosphere.

   It was also concluded that the firing temperature for maturing the adhesive may be any temperature between 1650°F and 1750°F. The total exposure at these temperatures will depend, among other factors, upon the firing temperature.

C. Methods of Applying Adhesives

Three studies were conducted to develop better methods for applying adhesives.

1. Use of Organic Solvents

   Tests were conducted using organic solvents instead of water as an application vehicle. UI 117-63 slips were made using butanol, methyl ethyl ketone, methyl alcohol, and "Nicro-braze" (a mixture of solvent and binder using in brazing).

   None of the solvents evaluated was found satisfactory for the following reasons:

   a. Bisques formed from slips made with all the solvents except "Nicro-Braze" crumbled and powdered.

   b. Bisques formed from slips using "Nicro-braze" had a smooth plastic-like surface. However, the center of the film tended to crumble and powder.

   c. Lap shear specimens made with bisques deposited from a "Nicro-braze" mixture fell apart when they were removed from the firing fixtures.

   d. Slips made with organic vehicles are difficult to keep in suspension.

   e. These organic solvents evaporate too rapidly to be used as vehicles for slips.

2. Development of Screeding Technique For Applying Adhesives

Screeding is a technique for applying a predetermined thickness of a viscous fluid to a base material by applying an excess of the fluid between two spacer bars resting on the base material and drawing a knife edge across the face of the bars to level the fluid.

A UI 117-63 slip having a viscosity of from 10,000-15,000 cps may be applied to lap shear coupons by screeding. The experimental screeding device used is described in "Testing Procedures" and shown in Figures 18 and 19.

Viscous slips may also be applied to large sheets using a saw-toothed trowel in the manner that lineoleum mastic is applied to a floor. The furrowed surface is smoothed out by gentle tapping of the metal sheet.

3. Spraying Techniques

A defined procedure for spraying adhesive has been developed and is reported in detail in "Recommended Procedures for Fabricating Sandwiches." Spraying of the ceramic adhesive is recommended.

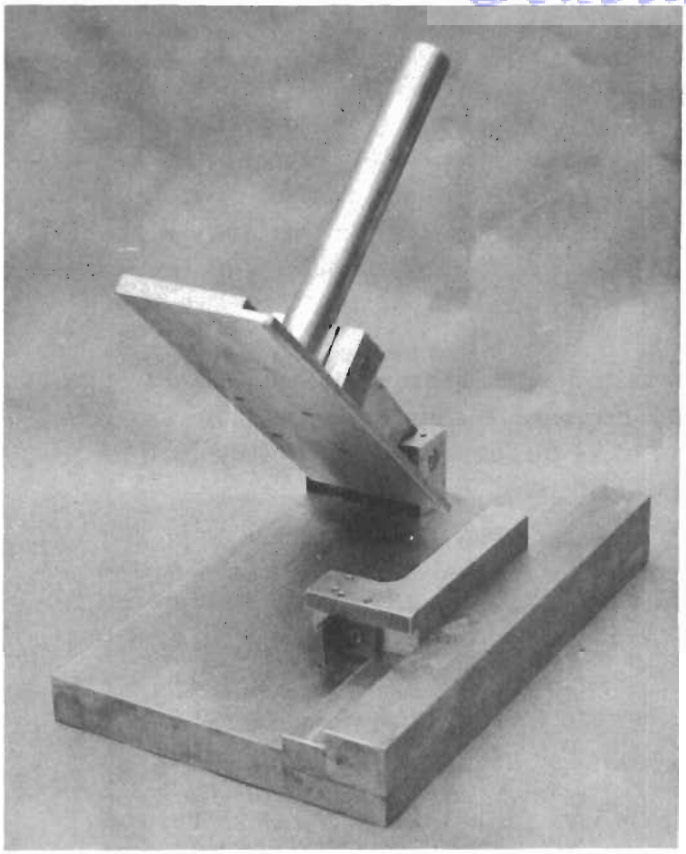
4. Drying the Slip

a. When applying the adhesive slip by dipping or screeding, the slip must be air dried, Any rapid drying of thick slip deposits will result in the cracking and spalling of the bisque.

b. When spraying the adhesive slip, the slip may be "forced" dried in an oven or under heat lamps provided that the slip is sprayed in thin coats. The water content of the applied slip is kept low and each coat is dried separately. The recommended practice for drying is contained in the "Recommended Procedures for Fabricating Sandwiches."

D. Surface Preparation of Facing Sheets

1. Four methods of solution cleaning stainless steel now in common use were compared to sandblasting. The comparison is based upon the tensile shear strengths at room temperature of lap shear specimens bonded with UI 117-63 adhesive and cleaned by the following methods:

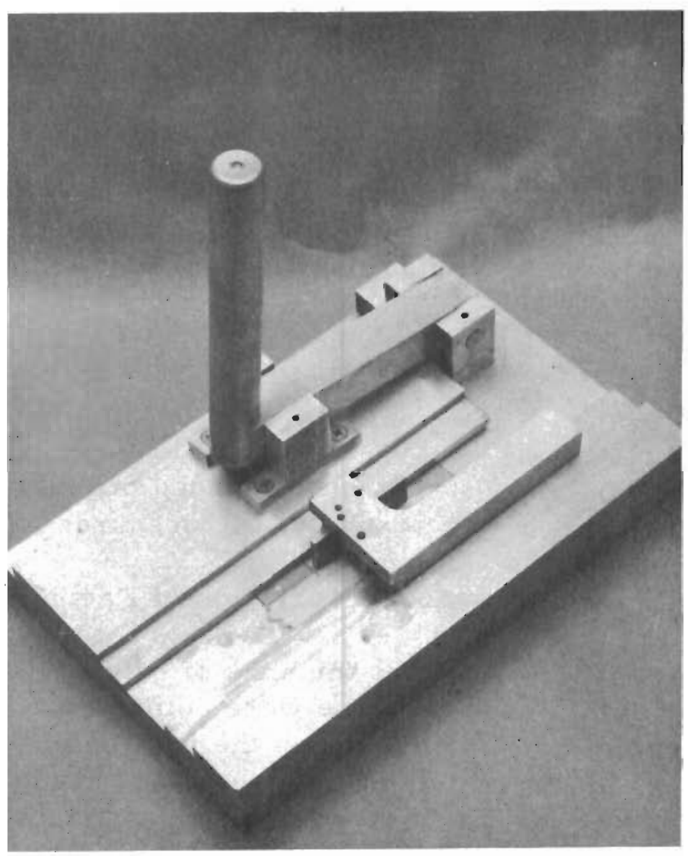


SCREEDING DEVICE

FIGURE 18

ADHESIVE APPLICATION ON LAP SHEAR SPECIMEN

FIGURE 19



Series 1. Vapor degreasing and light sandblasting.

Series 2. Vapor degreasing, electrolytic alkaline cleaning and light sandblasting.

Series 3. Vapor degreasing, electrolytic alkaline cleaning and etching in cold hydrochloric acid.

Series 4. Vapor degreasing, electrolytic alkaline cleaning and etching per Aeronca's method for cleaning 17-7 PH steel.

Series 5. Vapor degreasing, electrolytic alkaline cleaning and WS-22 etching as reported by Franklin Institute in WADC TR 55-87, Appendix I.

Series 6. Vapor degreasing, electrolytic alkaline cleaning and WS-4 etching as reported by Franklin Institute in WADC TR 55-87, Appendix I.

The results are reported below and detailed in Table XIV in the Appendix.

Tensile Shear Strength vs. Surface Preparation
(UI 117-63 Adhesive)

Method of Cleaning (Described Above)	Avg. Tensile Shear Strength At Room Temperature - psi
Series 2	1650
Series 1	1460
Series 4	1460
Series 5	1350
Series 3	1190
Series 6	1040

None of the solution cleaning methods tested were as effective as sandblasting.

2. Three methods of solution cleaning of 17-7 PH steel preparatory to applying ceramic adhesives were compared to each other. The comparison is based on the tensile shear strength at room temperature of lap shear specimens bonded with UI 1067-1 adhesive and cleaned by the following methods:

Series 1. Vapor degreasing, electrolytic alkaline cleaning and etching as reported by the Franklin Institute in WADC TR 55-87, Appendix I as the WS-22 method.

Series 2. Vapor degreasing, electrolytic alkaline cleaning and etching by Shell "Method B" followed by Shell "Method C" as reported by Shell Development Co. in WADC TR 53-126, Part IV, Pages 38 to 40.

Series 3. Vapor degreasing, electrolytic alkaline cleaning and etching in hot 20% HNO_3, 4% HF solution.

The results are reported below and detailed in Table XV of the Appendix.

Tensile Shear Strength vs. Surface Treatment
(UI 1067-1 Adhesive)

Method of Cleaning (Described Above)	Avg. Tensile Shear Strength At Room Temperature (psi)
Series 1	1420
Series 2	1230
Series 3	1390

3. Twenty surface treatments for preparing facing sheets of 17-7 PH for ceramic adhesive were evaluated by applying an adhesive coat of UI 1067-1 adhesive to sheets cleaned by these methods. The adhesion of a coating was determined by bending the coated sheet until the coating spalled. These empirical tests indicated that one preparation is best of all those tested. This method of surface preparation appeared to be equal to or even superior to sandblasting. Details of the method are as follows:

   a. Vapor degrease.

   b. Immerse in Matawan 48W*, 6 to 8 oz./gal. for 10 minutes, at 160°F under electric current.

   c. Rinse in cold water for 10 minutes.

   d. Heat scale at 1700°F for 30 minutes.

   e. Immerse in 20% HNO_3, 4% HF for 5 minutes at 150°F.

   f. Rinse in cold water for 10 minutes.

   g. Immerse in NaCN, 6 oz./gal. at room temperature for 5 minutes.

---

*See Appendix "Sources of Materials Mentioned in Report"

h. Rinse in cold water for 10 minutes.

i. Dry and coat.

See Table XVI for complete description of this and other treatments tested.

Summing up, the above studies indicated that strong etching of the surface by heat scaling and by hot acid attack will prepare the surface as well as by sandblasting.

4. The value of using an alkaline cleaning step in the above cleaning cycle was tested by preparing lap shear specimens by the above cleaning cycle, half of which were cleaned using the Matawan electrolytic alkaline cleaner and half of which were cleaned omitting the Matawan electrolytic alkaline cleaning step. The specimens were tested in tensile shear and no significant difference was noted between the specimens cleaned using the alkaline cleaning step. The test results are reported in Table XVII.

   Based upon this test, the alkaline cleaning step was omitted in all cleaning operations.

5. Difficulty in removing the 1700°F heat scale with the acid etch prompted a study to determine the optimum temperature and time at temperature for heat scaling the cleaning cycles.

   Core and facing sheets were heat scaled at 900°F, 1000°F, 1100°F, 1200°F, 1300°F, 1400°F and 1500°F for from 1/2-hour to 24-hours. From visual inspection and from noting the ease with which the scale was removed by acid etch, heat scaling at from 900°F to 1100°F for 30 minutes was considered optimum.

   The recommended practice is to heat scale for 30 minutes at 1000°F.

6. The cleaning cycle recommended for preparing 17-7 PH stainless steel for ceramic adhesive consists of:

   a. Vapor degrease.

   b. Heat scale at 1000°F for 30 minutes.

   c. Immerse in 20% HNO_3, 4% HF (by volume) solution for 5 minutes for facing sheets and 3 minutes for core at 150°-160°F.

d. Rinse in cold water for 10 minutes.

e. Immerse in 0.16 oz./gal. borax and 0.48 oz./gal. of soda ash at 125°-130°F for 30 minutes.

f. Rinse in cold water for 10 minutes.

g. Air dry.

E. Honeycomb Core Coating

The 17-7 PH steel core must be protected from corrosion during the firing of the honeycomb sandwich. Of the methods tested for protecting the core, the following affords the best protection:

1. The core is vapor degreased, heat scaled at 1000°F for 30 minutes, etched in 20% HNO_3, 4% HF at 150°F for 3 minutes, water rinsed, neutralized in borax-soda ash solution and water rinsed.

2. The adhesive is sprayed on the core to approximately 0.010" bisque thickness.

3. The core is dried under heat lamps.

4. The core is placed in a vertical position in the furnace and fired for 3-1/2 minutes at 1750°F.

In developing this procedure, the following tests were run:

1. Honeycomb core was prepared, coated and fired by various methods as detailed in Table 18. As a result of these tests, the surface preparation which gave the best results consisted of:

   a. Vapor degreasing.

   b. Electrolytic alkaline cleaning.

   c. Etching in 20% HNO_3, 4% HF for 150°F for 5 minutes.

   d. Water rinsed.

   e. Neutralizing in a 6 oz./gal. NaCN solution.

   f. Water rinsed.

2. As each improvement in the surface preparation of the facing sheets was determined, the altered preparation method was tried and evaluated on the honeycomb core. It was decided that the core could be prepared similarly to the facing sheet, except that the core should only be etched in the acid for three minutes as compared to five minutes for the facing sheet.

F. Assembling and Bonding the Honeycomb Sandwich

In the "Recommended Procedures for Fabricating Sandwiches," a satisfactory method for producing inorganic adhesive bonded 17-7 PH honeycomb sandwich is detailed. The steps in this method are:

1. Prepare the surface of the core and the facing sheets for application of the adhesive.

2. Apply the adhesive to the core and the bonding side of the facing sheets by spraying.

3. Prefire the core and the facing sheets to coat these parts.

4. Assemble the precoated core and facing sheets into a sandwich in the assembly tool.

5. Bond the sandwich by firing under vacuum at 1750°F for 10 minutes.

6. Cool and age the sandwich to temper the steel to the RH 950 condition.

G. Tooling

1. The Tool

A satisfactory tool for bonding the sandwich is detailed in "Recommended Procedures for Fabricating Sandwiches" and shown in Figure 1. Essentially the tool is a thin wall stainless steel envelope which contains the sandwich. A cast silica base known as "Glasrock" is placed on the flat bottom of the envelope. This base remains essentially flat throughout the firing and provides support for the sandwich. A sheet of "Fiberfrax" and a steel slip sheet separate the base from the sandwich. On top of the sandwich is placed a steel slip sheet, a layer of "Fiberfrax" and another cast silica block of "Glasrock." The envelope is sealed by welding a pan lid of thin gaged stainless steel to the envelope flanges. A vacuum is drawn in the tool during firing.

2. Materials for the Base of the Tool

A material to be used in this type of tooling must not oxidize at firing temperature. Three materials were considered:

a. "Glasrock", a cast ceramic has been found to be satisfactory material since it has the following desirable properties:

(1) Low coefficient of thermal expansion
(2) Low specific heat

However, "Glasrock" has poor heat conductivity and low resistance to flexural stresses. The flat bottom design of the above described tool is meant to avoid any large flexural stress on the base.

b. A coated graphite has been shown to resist oxidation. A sample of the material was placed in the furnace at 1750°F for 64 hours in air atmosphere. Under these conditions, the graphite showed a gain on only .024% in weight. Unfortunately, the coated graphite is not available commercially at the present time.

c. "Fahrite" and other metal bases were considered but were not used because of their high heat absorption during firing.

IV. Adhesive Modification

The UI 117-63 adhesive used successfully for lap shear bonding was modified to formulate an adhesive for bonding 17-7 PH honeycomb sandwiches.

A. One modification was necessary because of the following:

1. As reported in the tests on the effect of ceramic coatings on 17-7 PH foil, it was found that the UI 117-63 promoted the corrosion of 17-7 PH foil when the coated foil was fired in air atmosphere.

2. In firing inorganic adhesive sandwiches, it was observed that the 17-7 PH facing sheets and foil coated with UI 117-63 were badly corroded in the area close to the sandwich perimeter.

3. During the maturing temperature survey, the UI 117-63 coated specimens were badly corroded and the coating was cracked and spalled upon cooling.

It was concluded that the carbonyl iron in the UI 117-63 promoted the corrosion of the steel.

B. UI 1067-1 proved to be a better protective coating for the steel than either UI 117-50 or the combined UI 117-63 without metal powder additions. This was noted in the empirical tests made during the surface preparation studies.

C. Further attempts were made to improve the tensile shear strength of the adhesives. The purpose of these tests was to modify the adhesives by additions so as to achieve at least comparable tensile strengths to those reported for UI 117-63. An acceptable modified adhesive could not contain the carbonyl iron used in the UI 117-63 adhesive.

1. 50% UI 117 and 50% UI 1067 mixed adhesive combined with the following additions resulted in these average tensile shear strengths. See Table 19 for details.

a.	With no additions	1860 psi
b.	With 10% 304 stainless steel powder (-100 mesh)	1270 psi
c.	With 1% Fe_2O_3 and 10% 304 stainless steel powder (-100 mesh)	970 psi

2. The UI 117-50 adhesive bond joint with the following additions resulted in these average tensile shear strengths. See Table 20 for details.

a.	With no additions	1510 psi
b.	With 1% Fe_2O_3	1290 psi
c.	With 1% Fe_2O_3 and 10% 304 stainless steel powder (-100 mesh)	1300 psi
d.	With 15% stainless steel powder (-100 mesh)	1790 psi

3. The UI 1067-1 adhesive with the following additions resulted in these average tensile strengths. See table 21 for details.

a.	With no additions	1260 psi
b.	With 10% 304 stainless steel powder (-100 mesh)	1530 psi
c.	With 1% Fe_2O_3	1800 psi
d.	With 2.6% Fe_2O_3 and 26% 304 PC stainless steel powder (-100 mesh)	1840 psi
e.	With 13% Fe_2O_3 and 26% 304 PC stainless steel powder	1480 psi

f. With 2.6% Co_2O_3 and 26% 304 PC stainless steel powder (-100 mesh) — 1850 psi

g. With 13% Co_2O_3 and 26% 304 PC stainless steel powder (-100 mesh) — 1070 psi

h. With 1% Fe_2O_3 and 10% carbonyl iron — 2300 psi

4. UI 1067-1 adhesive modified by varying percentages of -325 mesh 304 PC stainless steel powder and Fe_2O_3 powder were tested in tensile shear. The results are detailed in Table and summarized below as average tensile strengths tested at room temperature.

   a. UI 1067-1, 5.8% Fe_2O_3 and 34% -325 mesh 304 PC steel powder — 2090 psi

   b. UI 1067-1, 1.5% Fe_2O_3 and 34% -325 mesh 304 PC steel powder — 2150 psi

   c. UI 1067-1, 2.5% Fe_2O_3 and 20% -325 mesh 304 PC steel powder — 2320 psi

   As a result of these studies, it was decided to make the first evaluation tests on 17-7 PH steel sandwiches with an adhesive based on the UI 1067-1 formulations with the additons of Fe_2O_3 and -325 mesh 304 PC stainless steel powder.

   No dispersing agent was used in the recommended adhesive. It was found that the UI 1067 could be dispersed without any additions.

5. The UI 1067-1 adhesive modified with 2.0% Fe_2O_3 and 20% -325 mesh 304 PC stainless steel powder was tested in tensile shear at room temperature, 600°F, 800°F and 1000°F. The results are detailed in Table XXIII and summarized below.

Tensile Shear Strength
UI 1067-1A

Test Temperature	Avg. Tensile Shear Strength (psi)
Room	1740
600°F	1690
800°F	1970
1000°F	2460

6. Tensile shear is not the only stress to be considered in evaluating an adhesive for sandwich construction. UI 1067-1 modified adhesives were applied to 17-7 PH steel flat sheets and the

adhesives evaluated by bending the sheet to compare the flexibility of the adhesives. The adhesives evaluated consisted of:

UI 1067-1, 1.5% Fe_2O_3, 20% -325 mesh 304 PC stainless steel powder

UI 1067-1, 2.0% Fe_2O_3, 20% -325 mesh 304 PC stainless steel powder

UI 1067-1, 2.5% Fe_2O_3, 20% -325 mesh 304 PC stainless steel powder

The UI 1067-1 adhesive modified with 2.0% Fe_2O_3, 20% -325 mesh 304 PC stainless steel powder (known hereafter as UI 1067-1A) proved to have the best flexibility. This adhesive was used in the honeycomb sandwich evaluation program.

V. Physical Properties of Inorganic Adhesive Bonded 17-7 PH Honeycomb Sandwich

Summarized below are physical properties of inorganic adhesive bonded 17-7 PH honeycomb sandwiches processed and bonded as described under the section entitled "Recommended Procedures for Fabricating Sandwiches." All tests were made on sandwiches composed of:

a. .032" 17-7 PH stainless steel facing sheets

b. .500" ± .003", 1/4" cell size, .002" foil thickness, square cell, spot welded 17-7 PH honeycomb core

c. UI 1067-1A adhesive compounded of 100 parts dry UI 1067-1 frit (200 mesh) 2.0% Fe_2O_3 powder, and 20% 304 PC stainless steel powder (-325 mesh). The adhesive was prepared as described under "Recommended Procedures for Fabricating Sandwiches."

A. Flexure Tests

The results below are reported for 3" x 8" sandwiches, single point loaded on a 6" span.

Average Core Shear Stress At Failure

1. For short time exposure at test temperature. See Table XXIV for details.

Test Temp.	Core Direction - 8" psi	Core Direction - 3" psi
Room	310	315
600°F	220	430
800°F	390	330
1000°F	275	245

2. After 192 hours exposure at test temperature. See Table XXIV for details.

Test Temp.	Core Direction - 8" psi	Core Direction - 3" psi
Room	310	325
600°F	190	225
800°F	345	275
1000°F	410	340

B. Edgewise Compression Tests

The results reported below are for 3" x 4" sandwiches, loaded in the 4" direction.

Average Edgewise Compression Stress at Failure

1. For short time exposure at test temperature. See Table XXV for details.

Test Temp.	Core Direction - 3" psi	Core Direction - 4" psi
Room	85,300	76,000
600°F	109,600	128,600
800°F	117,500	130,200
1000°F	70,800	33,800

2. After 192 hours exposure at test temperature. See Table XXV.

Test Temp.	Core Direction - 3" psi	Core Direction - 4" psi
Room	85,300	76,000
600°F	100,300	Broke
800°F	133,800	122,200
1000°F	Broke	71,200

C. Flatwise Tensile Tests

In an attempt to perform flatwise tensile tests in sandwich materials, 1" x 1" 17-7 PH, 1/4" cell, 0.002" foil honeycomb core was bonded between two blocks of 17-7 PH stainless steel to form a simulated sandwich. The blocks were then fitted into a testing fixture and the simulated sandwich pulled in tension. Because the simulated sandwiches did not bond satisfactorily, the tests were considered unsuccessful.

TESTING PROCEDURES

I. Evaluation of UI 117-63

A. Familiarization Tests

1. Preparation and Application of the Adhesive

The two basic frits, UI 1067 and UI 117, were prepared at the University of Illinois. The formulations are shown in Table XVI.

Slips UI 1067-1 and UI 117-50 (Table XXVIII were made at both the University of Illinois and Aeronca. The adhesive UI 117-63 (Table 28) was made at Aeronca by stirring carbonyl iron and silicon powder into a combination of UI 1067-1 and UI 117-50.

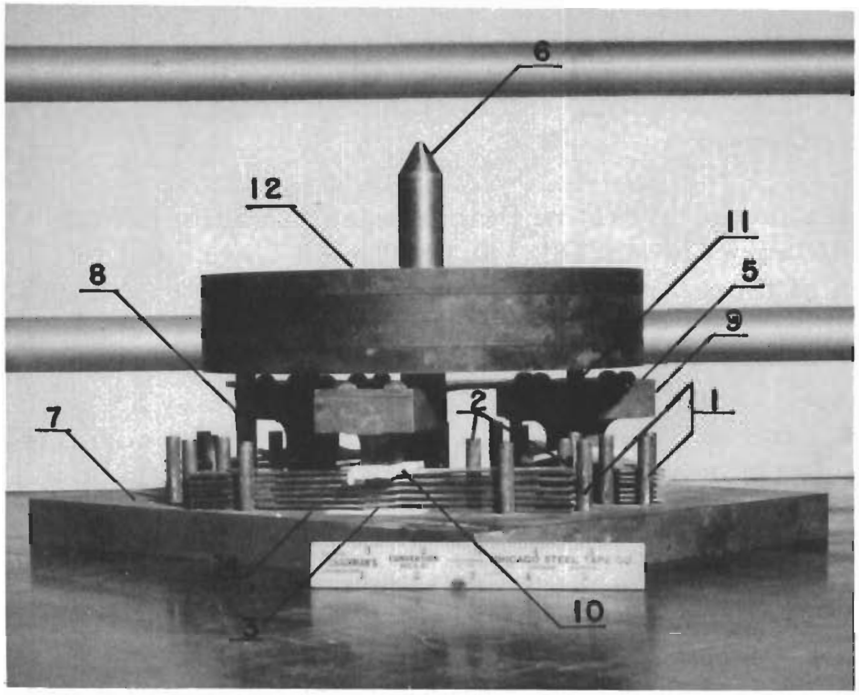
The adhesive was set (thickened) for coating application by small additions of two normal $NaNO_2$ or two normal KCl.

The thickness of the dried adhesive (bisque) was checked by dipping a standard plate in the slip and allowing the excess slip to drip from the plate. The plate was dried to constant weight at 200°F. From the weight of the adhesive on the plate, the thickness of the bisque on the plate was calculated by reference to previously determined calibrated data. If necessary, the slip was readjusted. (This test is fully described in "Ground Coat Dipping Weight Control," pages 347-351, "The Manual of Porcelain Enameling," published in 1937 by the Ferro Enamel Corporation.) The core and sheets were then dipped in the adjusted slip, allowed to drip, and dried to constant weight in the manner described.

2. Lap Shear Determination

Stainless steel coupons in the annealed condition were coated with the adhesive on a bonding area measuring 1" x 1/2" at one end of each coupon. After inserting a piece of 28 x 28 mesh .008" stainless steel open mesh screen in the bond joint, the lap shear specimens were placed in the pressure firing fixtures to form a 1/2" x 1" lap joint. (Figures 20 and 21).

Dimensional tolerances of the 0.050" thick coupons were maintained by machining the coupon to 1.000" ± .005" and 4.218" ± 0.010". Care was exercised in the removal of all burrs to minimize the rounding of edges.



1. EDGE PINS (LOCATING)
2. SIDE PINS (LOCATING)
3. LAP
4. LAP SHEAR SPECIMEN
5. SPIDER PLATE
6. CENTERING ROD
7. BASE PLATE
8. POSITIONING ROD
9. BEARING PLATES
10. FIBERFRAX
11. BEARING PINS
12. PRESSURE WEIGHTS

FIGURE 20

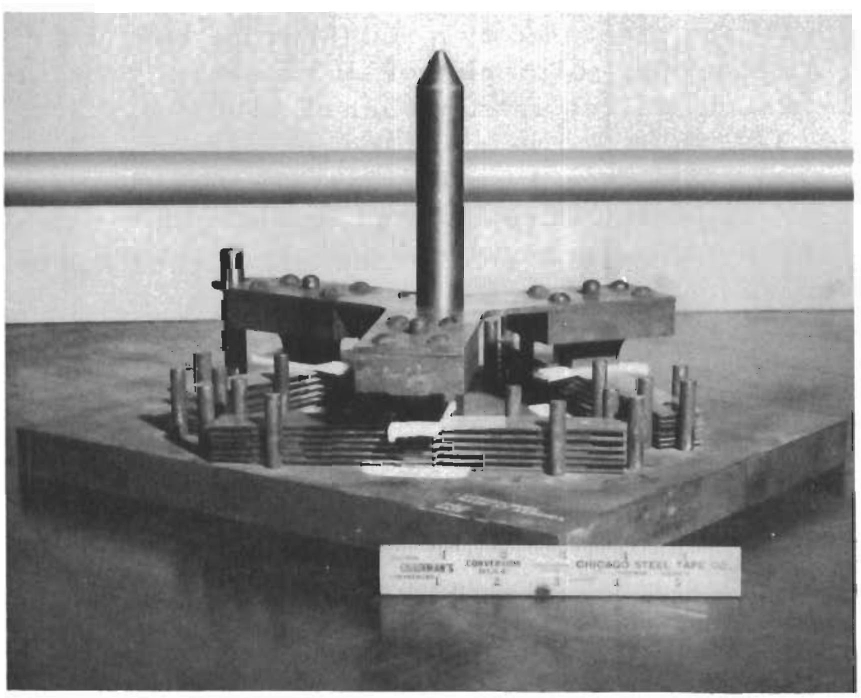


FIGURE 21

PRESSURE FIRING FIXTURE

In the evaluation program, faying surfaces were prepared for coating by light sandblasting and for other programs by other described methods.

Two edge pins on the pressure fixture were used to maintain 1/2" width of lap. The 4.218" ± 0.010" pieces were butted against each of these locating pins, maintaining the half-inch lap well within the 0.500" ± 0.030" desired.

Even pressure across the lap joint was maintained by a specially designed firing fixture (Figure 20). The fixture consists of a base plate (7) on which lap shear specimens are stacked along the legs of an equilateral triangle and held in place with three sets of positioning pins (1 and 2). To assure a uniform distribution of pressure a piece of "Fiberfrax" (10), a high temperature resistant mat which compresses evenly under pressure load, is placed on the lap joint specimens. Three plates (9) bearing down on the 1/2" lap joints are attached to the spider plate (5) by means of the top pointed bearing pins (11) at their centers, which are passed through the spider plate to become the points on which the imposed pressure load is applied to the lap joints. The thin stainless steel spider plate (5) is centered on a steel rod attached at the center of the base plate (7) and positioned by a rod (8) at the side of the base plate. After proper positioning of all members, pressure is exerted by placing the required number of mild steel discs, approximately 1/2" thick and weighing ten pounds each, over the tool center rod (6) on the pressure plate. Application of pressure to the lap joints can be adjusted by adding disc weights as desired. Except where otherwise noted, the pressure used on each lap joint is 50 psi.

The loaded pressure fixture is placed in a preheated Hevi-Duty electric furnace. The original preheat temperature of 1400°F was raised to 1800°F after maturing temperature study had been made. The bond joint temperature is elevated to 1750°F and maintained for ten minutes. During the evaluation program a ten minute soaking period at 1400°F was used. The soaking period was omitted on other studies. All temperatures are indicated by thermocouples at typical bond joints.

After firing, the loaded pressure fixture is removed from the furnace and allowed to air cool. When the tool has cooled to near room temperature the lap shear specimens are removed and soaked at -100°F for at least eight hours, after which they are aged at 950°F for 60 minutes.

The tensile shear strength of the bonded joints was measured by a 60,000-pound Tinius Olsen hydraulic tensile tester. The joint was heated to the desired temperature for fifteen minutes. The test load was applied between 1200 and 1400 pounds per minute. The temperature was controlled by a thermocouple placed close to the bond line.

B. Selection of 17-7 PH Stainless Steel

Tests were run for specimens of 17-7 PH and PH 15-7 MO steels as described for familiarization tests.

C. Bend Tests

The bend tests were made on lap shear inorganic adhesive bonded specimens fabricated as described above. The tests were performed per MIL-A-8431, paragraph 4.3.4.9, except that the three bearing rods were 3/8" in diameter instead of the 1/4" maximum diameter described. The test was performed in a Hevi-Duty test oven, placed in a Tinius Olsen hydraulic testing machine. A 1-7/8" rod was mounted on a circular plate which in turn rested on the testing machine bed. The rod passed through a 2-inch hole in the floor of the oven. A calibrated test plate was screwed to the top of the rod within the oven. Adjustable angles which acted as span supporters for the test piece were attached to the plate. A rod was suspended from a fixed platform over the oven and passed through a hole in the top of the oven. A fixture containing the 3/8" bearing rod was screwed to the bottom of this rod. The specimen was aligned carefully so that the top bearing load was centered on the 1/2" lap joint and was applied parallel to the 1-inch joint sides. The load was applied at 120 to 140 pounds per minute. The temperature was controlled by a thermocouple placed close to the bond line. Figure 22 shows the bend test setup.

D. Tensile Shear Strengths of Bond Joints After Various Exposures

1. Lap shear specimens were prepared and exposed to salt spray for 30 days per QQ-M-151, to tap water for 30 days, to JP-4 Jet Fuel, MIL-F-5624, for 7 days, to Anti-Icing Fluid, MIL-F-5566, for 7 days, to Hydraulic Oil, MIL-O-5606, for 7 days, and to Hydrocarbon Fluid, MIL-H-3136, Type III, for 7 days. The tap water was changed twice weekly during exposure to assure a supply of dissolved oxygen in the water. Before testing for tensile shear, each specimen was carefully dried. Tensile shear tests were made at room temperature, 600°F, 800°F, and 1000°F on specimens after exposure.

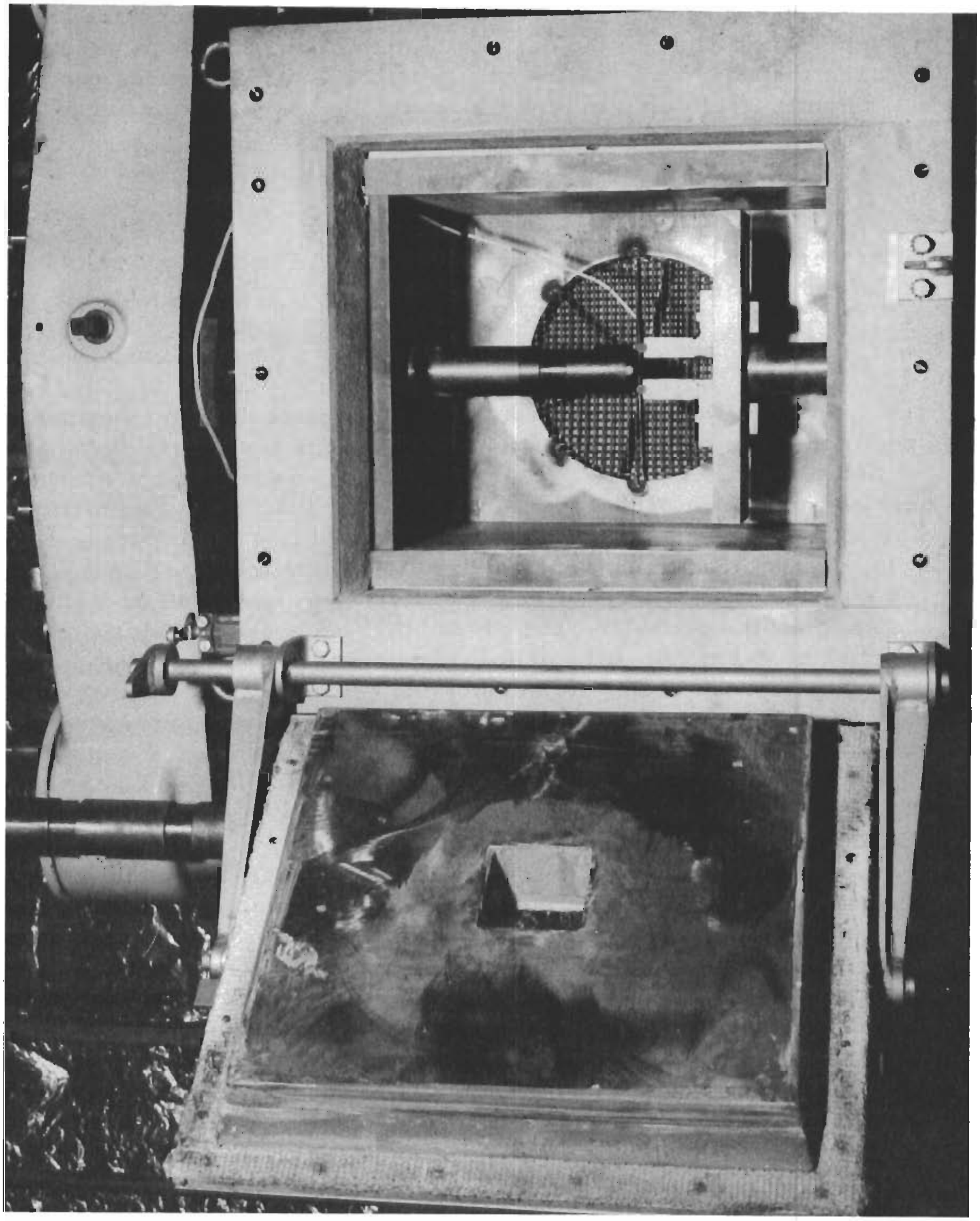


FIGURE 22
HIGH TEMPERATURE BEND TEST SET-UP

The results of these tests were erratic; however, for any individual firing, the scatter of results was small. It was, therefore, decided to compare each result to tensile shear results obtained from unimmersed samples bonded in the same firing. The results could then be compared on a percentage basis. This proved feasible except in the case of Firing #4 for which only one result was available and that result was much lower than expected. Since the other strengths reported for this firing were average for the whole lot, these results were compared to an average of all tensile shear strengths of unexposed samples made in this series.

2. Establishment of Standard Tensile Shear Strength for Comparison in Firing #4

One test run on an unexposed sample at room temperature was available. The tensile shear strength at room temperature was 880 psi.

If compared to this single result, all other results are excessively high. It was decided to compare the individual results to an average of all data on unexposed samples fired in Firing #4, #6, and #8 and tested at room temperature.

Sample	Firing No.	Tensile Shear At Room Temperature (psi)
17-7-57	4	880
17-7-62	6	1040
17-7-67	6	1240
17-7-72	6	1140
17-7-100	8	1470
17-7-105	8	1670
17-7-110	8	1250
17-7-111	8	1250
	Average	1240 psi

3. Establishment of Standard Tensile Shear Strength for Firing #6

Sample of Unexposed Specimen	Tensile Shear At Room Temperature (psi)
17-7-62	1040
17-7-67	1240
17-7-72	1140
Average	1140 psi

4. Establishment of Standard Tensile Shear Strength for Comparison in Firing #8

Sample of Unexposed Specimen	Tensile Shear At Room Temperature (psi)
17-7-100	1470
17-7-105	1670
17-7-110	1250
17-7-111	1250
Average	1410 psi

E. Boron Embrittlement

A honeycomb sandwich was made of 17-7 PH stainless steel facing sheets and 1/2" 17-7 PH stainless steel honeycomb core, 1/4" cell, 0.002" foil. Application of UI 117-63 adhesive was made by dipping the core and the faying surfaces of the facing sheets. A sandwich was made of the core and sheets coated with the UI 117-63 bisque and fired at 1750°F for ten minutes with steel plates on top of the sandwich supplying the pressure during firing.

Sections were cut from this sandwich and prepared for microscopic study. The section selected included the core, the bond joint and the facing sheet. The highly polished, 10% oxalic acid etched cross section was examined at 500 magnification for any indication of boron embrittlement.

Photo-micrographs of the bond joint indicate that no boron penetration had taken place. There is evidence of penetration of the adhesive into the base metal, but this is considered normal for a ceramic bond as has been experienced in the procelain enameling of steel.

F. Effect of Ceramic Coatings on Tensile Strength of 17-7 PH Foil

Half-inch wide coupons were filed out of 0.005" 17-7 PH stainless steel, Condition A, foil. The specimens were made with care to avoid nicking the surface and assure smooth radii where the samples necked down. The samples to be coated were dipped in prepared slip, allowed to drip and air dried. All specimens were placed in a firing fixture which held them horizontally and on edge. They were then fired and aged, as recommended by Armco Steel, to convert the steel to the RH 950 condition.

The tensile strength of the coated and uncoated samples were determined by the normal laboratory method. The apparent tensile strength was calculated by dividing the total breaking load by the original cross-sectional area of the steel.

II. Reproducibility Studies

The individual frits (UI 1067 and UI 117) were dry milled to pass a 48 mesh screen. They were then weighed separately (63.3 parts of UI 117 to 36.7 parts of UI 1067) and placed in a ball mill. Syloid 244 and water were then added. The mixture was milled for three hours. At this time, the slip was separated from the balls, and a 50 cc weighed sample of the slip screened through a 200 mesh screen. Any remaining frit was weighed after drying thoroughly and its proportion to the weighed sample determined in percent. If this was more than 1/2%, the slip was replaced and milled until the material retained on the 200 mesh screen was less than 1/2%. Dip weight was then determined and the slip tested as indicated in this report.

A. Slip Control

1. Dip Weight Versus Viscosity

The dip weight test was conducted by taring a lightly sandblasted 4" x 4" sheet of 0.022" 17-7 PH stainless steel. The panel was dipped in the slip to be tested, and suspended in a vertical position until no more slip dripped from the bottom of the panel. The panel was then dried in a circulatory air oven at 220°F for 15-20 minutes. After drying, the panel was cooled and weighed to the nearest 0.1 gram. The difference between the total dry weight and the tare weight multiplied by the factor 4.5 is equal to the dry weight of the slip in grams per sq. ft.

The slip viscosity was measured with a Brookfield viscosimeter using the No. 3 spindle at 10 rpm. If the slip viscosity was too great for the range of the given spindle at this speed, the viscosity was determined with a spindle which would operate at the same speed and effect a reading in the middle of the scale.

2. Effect of Aging on the Slip Viscosity

The slip viscosity was determined at 4, 29, 52, 96 and 169 hours after the start of wet milling.

3. Relationship Between the Na_2O and B_2O_3 Dissolved in the Mill Liquor and the pH of the Slip

The test for the B_2O_3 content of the mill liquor was conducted using a 10 cc sample of mill liquor titrated with 0.2 normal hydrochloric acid to the methyl red end point.

The volume of 0.2 normal hydrochloric acid times the factor 0.0062 is equal to the number of grams of B_2O_3 contained in a 10 cc sample of the mill liquor.

The test for the Na_2O content was conducted on a 10 cc sample of liquor. The sample was then cooled and an equal volume of neutral gylcerol added. The sample was titrated to the phenophthalein end point with 1.0 normal KOH. The volume of 1.0 normal KOH required times the factor 0.035 is equal to the grams of Na_2O contained in the 10 cc sample of mill liquor.

All pH values were obtained from a Beckman pH meter.

On the test performed for B_2O_3 and Na_2O content prior to Aug. 7, 1958, the amount of distilled water used to dilute the sample was not considered important to the test. However, it was noticed that the pH and the volume of the distilled water affected the pH of the slip. It was decided that a designated volume of water would be used in order to obtain comparable slip pH results.

4. Effect of Electrolytes and Organics for Reducing Set

A slip consisting of equal parts of UI 1067 and UI 117 frits milled with approximately 50% water was allowed to set for a period of two weeks. The slip was then very thixotropic and yielded 0.023" of dried bisque in the dip test.

A 100 cc sample of each slip was used for each electrolyte or solvent. The chosen electrolyte or solvent was then added in increments. The slip was stirred and a coating thickness obtained after each addition by the dip weight test. Solutions evaluated were saturated sodium pyrophosphate, 2N HCl, 2N HNO_3, 2N acetic acid, and 2N NaOH. Solvents evaluated were ethyl alcohol and n-butyl alcohol.

B. Relationship of Bond Line Thickness to Tensile Shear Strength

The faying surfaces of the 17-7 PH stainless steel coupons were prepared by vapor degreasing and light sandblasting. Various bisque thicknesses of UI 117-63 adhesive were applied by the use of the laboratory screeding device. The doctor blade was set so as to result in the desired dry bisque thicknesses. Drying was accomplished by air drying followed by heating under lamps so as to avoid bisque cracking. The lap shear specimens were assembled with 0.008" 28 x 28 mesh 304 stainless steel screen and fired, using the reported pressure.

The firing temperatures were chosen to determine whether variation in temperatures and exposure times could improve the tensile strengths and subsequent oxidation of the steel.

C. Effect of Bond Line Pressure on Tensile Shear Strength

The conclusions reached on the effect of bond line pressure on the tensile shear strength were based on data determined in the study of the effect of bond line thickness on tensile shear strength.

III. Process Development

A. Temperature Survey of the Firing Furnace

The temperature survey was made during a firing of 21 17-7 PH steel lap shear specimens. The firing load consisted of the pressure firing fixture and the specimens. The furnace surveyed was a Hevi-Duty Electric Furnace, Type-Hot-243618-CU, which was used throughout this work. The thermocouples were welded to the edge of the spacers. The edge of the spacer was then placed within a fraction of an inch of the edge of the bond joint.

Location of Thermocouples at Specimens as Indicated

	Thermocouple No.		
Location in Furnace	Back	Fwd. Right	Fwd. Left
Bottom specimen	33	1 - 2	34
Middle of specimen pile	39/44	35	40
Top specimen	50	41	45

Furnace temperatures were also recorded for the back and the front of the furnace.

B. Maturing Temperature Studies

All observations reported in this study were made on PH 15-7 MO or 17-7 PH test coupons coated with either UI 117-63 bisque without metal additions or UI 117-63 adhesive bisque with metal additions. It should be understood that no adhesive joint was made, but that the adhesive coatings were applied in much the same way as porcelain enamel. When firing to determine the proper melting temperature, trials indicated that it took approximately 5 minutes for the coupons to come to temperature. As all test firings were conducted for 15 minutes, it is assumed that the adhesive coating was at actual firing temperature for ten minutes. The furnace temperature was assumed to be the firing temperature.

Determining when the adhesive coating had matured was subjective and the results recorded herein should be accepted with this in mind. The same was true for reporting of underfired, satisfactorily fired and overfired conditions.

The determination of permissible soaking time at various temperatures was done in the same way with the total time reported assumed to include five minutes of heat-up time.

C. Better Methods of Applying Adhesives

1. Use of Inorganic Solvents

Slips of UI 117-63 with metal additions were dried and remilled using butanol, methyl ethyl ketone, methyl alcohol, or "Nicro-braze." The milling was kept to a minimum to avoid combustion of the solvent and to discourage any further reduction in particle size of frit. The suspended slip was then applied by dipping. As reported in the results, a cohesive film was not achieved with any but the "Nicro-braze" suspension.

Lap shear joints containing the "Nicro-braze" deposited bisque were fired in the usual manner.

2. Development of Screed Technique

Figures 18 and 19, page 35, illustrate the screeding device used to apply the adhesive in this study. The device was designed by the Franklin Institute for the Materials Laboratory, WADC. The prepared lap shear coupons are clamped into the device by lowering and locking the plate onto the back face of the coupons. The faying surface end of the coupon is thus butted against the steel member. The screed bar rides on one side on top of the steel member, and on the other side on a blade parallel to the steel edge bar. This blade defines the width of adhesive applied. The doctor blade is normal to this side blade and is adjusted to a desired height above the base of the tool to wipe a determined thickness of adhesive on the coupon. An excess of viscous slip is applied to the faying surface of the sample before drawing the doctor blade across the coupon. After applying the adhesive in this manner, the coupon is removed from the device and tapped gently to smooth out the adhesive layer.

3. Spraying Techniques

Under "Recommended Procedures for Fabricating Sandwiches," there is a detailed description of the spraying technique developed for applying the recommended adhesive.

4. Drying the Slip

Familiarity with materials led to development of the recommended drying methods.

D. Surface Preparation of Facing Sheets

Tables XIV, XV, XVI and XVII contain complete descriptions of the procedures followed.

E. Honeycomb Core Coating

Table XVIII contain complete descriptions of procedures followed.

F. Assembling and Bonding the Honeycomb Sandwich

The procedures recommended were arrived at by observing the results of a number of small firings using various process techniques. The recommended practice is detailed in "Recommended Procedures for Fabricating Sandwiches."

IV. Adhesive Modifications

Procedures used are detailed in the Results and in Tables XIX, XX, XXI, XXII AND XXIII.

V. Physical Properties of Inorganic Adhesive Bonded 17-7 PH Honeycomb Sandwich

A. Honeycomb sandwiches were prepared as described in the "Recommended Procedures for Fabricating Sandwiches." Sandwiches 18" x 33" were fired. Before the panel was subjected to -100°F treatment, the large panels were cut to test size and the edges polished with a sanding disc. The individual sandwiches were then kept at -100°F overnight and aged at 950°F for 60 minutes.

Excellent descriptions of the testing methods and fixtures for testing sandwich construction at elevated temperatures are contained in Forest Products Laboratory Report No. 2063, dated September, 1956, by Edward W. Kuenzi, Reference No. 18.

B. Test Furnace

The test furnace was placed on the lower platen of the testing machine. The furnace is an electrically heated Hevi-Duty furnace for continuous operation up to 1200°F. The heat provided by elements located in the sides of the furnace is radiated by steel baffles one inch from the surface of the elements. These baffles also serve to channel the circulated air from a fan at the back of the furnace.

Tests were conducted in the center portions of the furnace. The loads were transmitted to the test fixture through stainless steel rods extending from the tensile machine platens through holes in the floor and ceiling of the furnace.

The furnace temperatures were controlled by a chrom-alumel thermocouple on the test piece which activated a capacitrol.

C. Long Time Exposure Tests

Samples tested after 192 hours exposure were maintained at test temperatures in a Hevi-Duty circulating furnace. After 192 hours, the samples were removed and allowed to cool to room temperature. They were mounted in the test fixture within a Hevi-Duty test furnace in a Tinius Olsen 60,000-pound tensile machine. The samples were then heated to the test temperature, maintained at that temperature for ten minutes and tested.

D. Flexure Test

The specimens were supported on a span of six inches. The reaction points were 3/8" rods welded to a shim plate which prevented buckling of the skin. The load was applied at midspan with a 3/8" rod welded to the stainless steel activating rod. A shim plate was placed between the load rod and the sandwich to prevent buckling.

The load was applied at a rate of 60 pounds a minute. The results are reported in core shear stress at failure. Other ceramic systems may be compared to the UI 1067-1A system by testing sandwiches of the same configuration and material.

The core shear stress at failure is defined as:

$$f_s = \frac{P}{w(t + t_c)}$$

f_s = core shear stress, in psi

P = total load at failure in pounds

w = width of sample in inches

t = total thickness of sandwich

t_c = thickness of the core

E. Edgewise Compression

The edgewise compression test was made on 4-inch long and 3-inch wide sandwiches. The specimen ends were machined flat and square and parallel to each other. The alignment of a specimen was achieved by placing the bottom edge of a specimen on the flat bottom rigid base and holding it tightly by adjustable triangular wedges. To align the specimen, the top plate was adjusted by a ball on the top side of the top plate which swiveled in a socket on the bottom of the load bearing steel rod. The alignment was checked by attaching strain gages at similar positions on both facing skins, loading the specimens below capacity at room temperature, and measuring the stresses. Adjustments were made until the stresses in the two skins were in balance.

Edgewise compression is defined as:

$$f_F = \frac{P}{w(t - t_c)}$$

f_F = edgewise compression in psi

P = total load at failure in pounds

w = width of sandwich in inches

t = thickness of sandwich in inches

t_c = thickness of core in inches

F. Flatwise Tensile

The method of tests attempted are briefly described in Results.

CONCLUSIONS

1. The UI 117-63 adhesive system evaluated in this program was designed as a result of experiments indicating factors which increased the tensile shear strength of an inorganic adhesive bonded lap shear joint. Some factors do not prove advantageous in a sandwich adhesive.

2. UI 117-63 adhesive is composed of two frits, UI 117 and UI 1067. Current project experiments indicate that UI 1067 adhesives afford stainless steel corrosion protection obtainable with neither UI 117 adhesives nor adhesives of UI 117 and UI 1067 in combination.

3. Marked improvement in tensile strength of the adhesive joint in lap shear with addition of carbonyl iron to some ceramic adhesives led to the assumption that carbonyl iron additives should be included in a ceramic base adhesive for sandwich structure. Further research indicated that carbonyl iron promoted such corrosion of the steel that, in firing, honeycomb core crumbled and the facing sheets were severely attacked. Consideration was then given to substitution of material with the advantages of carbonyl iron without the corrosion. The factors contributed by carbonyl iron are not known. It is postulated that carbonyl iron promotes the formulation of iron oxide which is desirable when the reaction is limited as in air starved lap joint, but undesirable when the reaction is excessive as in a sandwich fully air exposed at high temperatures. It is reasoned that the iron acts for form many metal to ceramic bonding surfaces reducing the thickness of the brittle unreinforced ceramic matrix.

4. A practical inorganic adhesive for sandwich construction can be compounded by adding to a ceramic Fe_2O_3, supplying desirable iron oxide, and stainless steel powder effecting a reduction in the thickness of the brittle ceramic matrix.

5. The inclusion of a .008" 304 stainless steel 28 mesh screen in the joint appeared to increase the tensile strength of a lap joint. The same screen used in the honeycomb sandwich between the sheet and the core tends to act as a weak plane for the promotion of peel failure.

6. The many and complex problems in controlling the slip when ceramic adhesives are applied by dipping are resolved or minimized by spraying the adhesive.

7. Unlike UI 117-63 which contains a dispersing agent, the recommended adhesive for sandwiches contains none.

8. The flexibility of the ceramic adhesive remains a problem. Lap shear bend tests indicate that UI 117-63 adhesive averages approximately sixty

percent of the flexibility exhibited by high temperature (500°F) organic adhesives. Tests on honeycomb sandwiches indicated a comparative flexibility for the sandwich ceramic adhesive. The flexibility of the adhesive requires improvement.

9. Lack of reproducible properties of ceramic adhesives and components bonded with ceramic adhesives was noted throughout the program. In the early tensile shear strength tests, this lack of reproducibility made the interpretation of results difficult. Studies were made on factors which affect reproducibility of test results. Similar studies are indicated covering each phase of the work.

10. A method of preparing the 17-7 PH surface resulting in better adhesion between the ceramic and the steel has been developed. The preparation itself reduced the thickness of the steel by chemical attack. Further work is required to develop surface preparations which either do not attack the steel or keep the attack to a minimum.

11. The method of evaluating inorganic sandwich adhesives should be confined to tests on honeycomb sandwiches of standardized core and facing sheet materials and sizes.

12. At the start of the program no satisfactory process for assembling and firing an inorganic bonded honeycomb sandwich had been developed. Core and facing sheets were dipped in a slip, assembled, and fired. Many reactions occurred during firing over which very little control could be exercised. It was found that controls could be exercised over some of the undesirable reactions by dividing the sandwich fabrication into separate steps. The core and facing sheets are coated with adhesives prior to their assembly. The sandwich is then assembled and fired under heat and pressure to form integral bonds between the core and the sheets.

13. The honeycomb panels produced in this work were made in an electric fired furnace using vacuum pressure tooling. This tooling and method of heating are not ideal for the purpose since it requires excessive heat-up time, exposing the sandwich to long periods at oxidizing temperatures. A study is required to develop methods of bonding utilizing light tools and direct sources of intense heat.

14. Use of an inert atmosphere during firing is considered impractical since unless the ceramic adhesive is saturated with iron oxide, no bond is formed.

15. The flexure and edgewise compression test results for inorganic adhesive bonded sandwiches reported herein are indicative of the evaluated inorganic adhesives. It is indicated that with better process control, modified adhesive formulations, and advanced bonding methods, these strengths can be improved considerably.

RECOMMENDATIONS

1. Continue the modification of the adhesives. Explore further the effects of metallic oxide additions such as ferrous oxide, cobalt oxide, and nickel oxide. Study the effect of using powders of varying particle size in an attempt to achieve microscopically thin ceramic films in the void areas of the powder. Determine effect of cleaning metallic powders by solvents and chemical treatments to promote ceramic adhesion to the metal powders. Investigate other methods of increasing flexibility.

2. Evaluate other ceramic systems as a basis of inorganic adhesives.

3. Investigate other methods of applying inorganic adhesives such as flame spraying and electrolytic deposit.

4. Continue the steel surface preparation study in an attempt to find a method which is less damaging to the steel.

5. Continue the reproducibility study to determine effects of variations in raw materials and smelting and grinding techniques.

6. Investigate methods for preserving adhesives in storage.

7. Make a comprehensive study of firing temperature cycles in the bonding of inorganic adhesive sandwiches.

8. Evaluate other methods of firing inorganic adhesive bonded sandwiches such as radiant heating and heat blankets, in an attempt to reduce the heat-up time during firing.

9. Evaluate other materials for light bases in bonding tools.

10. Develop prefabricated inorganic adhesive tapes.

11. Investigate methods of protecting the exterior sides of the sandwich from tarnish and corrosion during firing.

12. Determine properties of inorganic adhesive bonded honeycomb sandwiches such as fatigue and long span bending. Determine tensile shear strength of inorganic adhesives up to 1800°F. Develop improved methods of determining flatwise tensile of inorganic adhesive bonded honeycomb sandwich.

13. Develop inorganic adhesives for other high temperature alloys. Evaluate inorganic adhesive bonded sandwiches of these materials.

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16. Naps, Marguerite. Elevated temperature resistant modified epoxide resin adhesives for metals. Shell Development Company, WADC TR 53-126, Part IV, ASTIA Document No. 130884.

17. Department of Defense. Sandwich construction and core materials; general test methods. MIL-STD-401A, 1956.

18. Kuenzi, Edward W. Methods of testing sandwich construction at elevated temperature. Report No. 2063. Madison, Wis., Forest Products Laboratory, U. S. Department of Agriculture, 1956.

19. Department of Air Force, Air Research and Development Command; Department of the Navy, Bureau of Aeronautics and Department of Commerce, Civil Aeronautics Administration. Sandwich construction for aircraft. ANC-23 Bulletin, Part II. 2nd ed. 1955.

SOURCES OF MATERIALS

MENTIONED IN REPORT

1. Stainless Steel Powders - Charles Hardy, Inc., 420 Lexington Ave., New York 17, N. Y.

2. Honeycomb Core - Hexcel Products, Havre de Grace, Maryland.

3. "Fiberfrax", Grade 970, .040" and .080" thicknesses, 24" wide - The Carborundum Company, Niagara Falls, New York.

4. Stainless Steel, 17-7 PH - Armco Steel Corporation, Butler, Pa.

5. Miscellaneous Ceramic Raw Materials - Chicago Vitreous Corp., 1425 South 55th Street, Cicero 50, Illinois.

6. UI 1067 and UI 117 Frits - Chicago Vitreous Corp., 1425 South 55th Street, Cicero 50, Illinois.

7. Carbonyl Iron, Grade C - General Aniline and Film Company, Special Products Sales Department, 270 Park Avenue, New York 17, N. Y.

8. Screen, Stainless Steel, .008" thick of .004" 304 stainless steel wire, 28 x 28 mesh - Reynolds Wire Division, National Standard Co., Dixon, Illinois.

9. "Glasrock" - Glasrock Corporation, P.O. Box 4334, 1101 Glidden St., Atlanta 2, Georgia.

10. Crucibles, No. J-50, Type "h" - Joseph Nixon Crucible Company, Jersey City 3, N. J.

11. Silicon Powder, No. 101 - Metals Disintegrating Company, Inc., Elizabeth, N. J.

12. "Fahrite" - Ohio Steel Foundry Company, 1075 James St., Springfield, Ohio.

13. "Matawan 48W" - Hanson, Van Winkle and Munning, Matawan, N. J.

14. "Nicrobraze" - Wall Colmonoy Corporation, 19345 John R Street, Detroit 3, Michigan.

APPENDIX

TABLE I

TENSILE SHEAR STRENGTH
LAP SHEAR SPECIMENS
FIRING NO. 1

Sample No.	Type of Steel	Test Temperature °F	Tensile Shear Strength psi
9	PH 15-7 MO	Room	232
2	PH 15-7 MO	600°	224
4	PH 15-7 MO	800°	120
7	PH 15-7 MO	1000°	492
1	17-7 PH	Room	416
2	17-7 PH	600°	164
3	17-7 PH	800°	384
8	17-7 PH	800°	440
4	17-7 PH	1000°	1224

NOTES:

1. UI 117-63 adhesive used.
2. 28 x 28 mesh .008" 304 stainless steel screen used in the lap joint.
3. All samples were heat treated and aged to harden the steel to the RH 950 condition.
4. PH 15-7 MO samples 1, 3, 5, 6 and 8 broke before testing. 17-7 PH samples 5, 6, 7 and 9 broke before testing.
5. These results are not those generally expected. They are lower than those previously reported and reflect the inexperience expected in handling new materials and techniques.
6. Pressure used - 50 psi.
7. All coupons sandblasted.

TABLE II

TENSILE SHEAR STRENGTH
LAP SHEAR SPECIMENS
FIRING NO. 2

Sample No.	Type of Steel	Temperature °F	Bond Thickness inch	Tensile Shear Strength psi
12	17-7 PH	Room	.0085	1860
18	17-7 PH	Room	.010	2140
Average	17-7 PH	Room		2000
10	17-7 PH	600°	.009	1920
17	17-7 PH	600°	.0105	1740
Average	17-7 PH	600°		1830
13	17-7 PH	800°	.0085	1650
14	17-7 PH	800°	.0105	1400
Average	17-7PPH	800°		1520
15	17-7 PH	1000°	.009	1500
16	17-7 PH	1000°	.0115	1510
Average	17-7 PH	1000°		1500
10	PH 15-7 MO	Room	.0075	2160
14	PH 15-7 MO	Room	.0095	2130
Average	PH 15-7 MO	Room		2140
11	PH 15-7 MO	600°	.011	1280
17	PH 15-7 MO	600°	.009	1510
Average	PH 15-7 MO	600°		1400
12	PH 15-7 MO	800°	.0075	1560
13	PH 15-7 MO	800°	.0105	1470
Average	PH 15-7 MO	800°		1520
15	PH 15-7 MO	1000°	.0115	1320
18	PH 15-7 MO	1000°	.009	1760
Average	PH 15-7 MO	1000°		1540

NOTES:
1. UI 117-63 adhesive used.
2. 28 x 28 mesh, .008" stainless steel screen used in lap joint.
3. All samples were heat treated and aged to harden the steel to the RH 950 condition.
4. These results are equal to or in excess of test results reported by the University of Illinois for samples of the same material.
5. Pressure used - 50 psi.
6. All coupons sandblasted

TABLE III

BEND STRENGTH
LAP SHEAR SPECIMENS
FIRING NO.3

Sample No.	Test Temperature °F	Bend Strength pounds
19	Room	96
27	Room	83
34	Room	89
Average	Room	89
21	600°	92
30	600°	62
35	600°	77
Average	600°	77
23	800°	77
22	800°	93
36	800°	69
Average	800°	80
28	1000°	83
32	1000°	86
33	1000°	92
Average	1000°	87

NOTES:

1. UI 117-63 adhesive.
2. 17-7 PH stainless steel cleaned by sandblasting.
3. Pressure used - 50 psi.
4. All samples were heat treated and aged to harden the steel to the RH 950 condition.
5. 28 x 28 mesh .008" 304 stainless steel screen used in lap joint.
6. MIL-A-8431 requires a minimum 130 pounds in bend at room temperature for high temperature organic adhesive.

TABLE IV

TENSILE SHEAR RESULTS
AFTER 7-DAY EXPOSURE
IN ORGANIC FLUIDS

Sample No.	Firing No.	Immersion Medium	Test Temp. °F	Tensile Shear psi	Tensile Shear Standard psi	Tensile Shear % of Standard
17-7-53	4	JP-4 Fuel	Room	1080	1240	87
17-7-53	6	JP-4 Fuel	Room	1390	1140	122
Average		JP-4 Fuel	Room			105
17-7-58	6	JP-4 Fuel	600°	1160	1140	102
17-7-96	8	JP-4 Fuel	600°	1670	1410	118
Average		JP-4 Fuel	600°			110
17-7-63	6	JP-4 Fuel	800°	790	1140	69
17-7-101	8	JP-4 Fuel	800°	1660	1410	118
Average		JP-4 Fuel	800°			94
17-7-68	6	JP-4 Fuel	1000°	1340	1140	118
17-7-106	8	JP-4 Fuel	1000°	1930	1410	137
Average		JP-4 Fuel	1000°			128
17-7-69	6	Anti-icing Fluid	Room	560	1140	49
17-7-107	8	Anti-icing Fluid	Room	1340	1410	95
Average		Anti-icing Fluid	Room			72
17-7-54	4	Anti-icing Fluid	600°	960	1240	78
17-7-74	6	Anti-icing Fluid	600°	1130	1140	99
Average		Anti-icing Fluid	600°			88
17-7-59	6	Anti-icing Fluid	800°	480	1140	42
17-7-97	8	Anti-icing Fluid	800°	1700	1410	121
Average		Anti-icing Fluid	800°			82
17-7-64	6	Anti-icing Fluid	1000°	1360	1140	119
17-7-102	8	Anti-icing Fluid	1000°	1740	1410	123
Average		Anti-icing Fluid	1000°			121
17-7-65	6	Hydraulic Oil	Room	1330	1140	117
17-7-103	8	Hydraulic Oil	Room	1820	1410	129
Average		Hydraulic Oil	Room			123
17-7-70	6	Hydraulic Oil	600°	1280	1140	112
17-7-108	8	Hydraulic Oil	600°	1300	1410	92
Average		Hydraulic Oil	600°			102
17-7-55	4	Hydraulic Oil	800°	1080	1240	87
17-7-75	6	Hydraulic Oil	800°	700	1140	61
Average		Hydraulic Oil	800°			74

TABLE IV (Concluded)

Sample No.	Firing No.	Immersion Medium	Test Temp. °F	Tensile Shear psi	Tensile Shear Standard psi	Tensile Shear % of Standard
17-7-60	6	Hydraulic Oil	1000°	1330	1140	117
17-7-98	8	Hydraulic Oil	1000°	1630	1410	116
Average		Hydraulic Oil	1000°			116
17-7-61	6	Hydrocarbon F.	Room	900	1140	79
17-7-99	8	Hydrocarbon F.	Room	1530	1410	108
Average		Hydrocarbon F.	Room			94
17-7-66	6	Hydrocarbon F.	600°	1260	1140	111
17-7-104	8	Hydrocarbon F.	600°	1450	1410	103
Average		Hydrocarbon F.	600°			107
17-7-71	6	Hydrocarbon F.	800°	1270	1140	112
17-7-109	8	Hydrocarbon F.	800°	1320	1410	94
Average		Hydrocarbon F.	800°			103
17-7-56	4	Hydrocarbon F.	1000°	1630	1240	132
17-7-94	6	Hydrocarbon F.	1000°	1650	1410	117
Average		Hydrocarbon F.	1000°			124

NOTES:

1. .050" 17-7 PH stainless steel, tempered to RH 950 condition, UI 117-63 adhesive with 28 mesh wire screen of .008" 304 stainless steel used in the joint.
2. See "Testing Procedures" for determination of standard for comparison.
3. JP-4 Fuel, MIL-F-5624; Anti-icing Fluid, MIL-F-5566; Hydraulic Oil, MIL-O-5606; Hydrocarbon Fluid, MIL-H-3136, Type III.
4. Pressure used - 50 psi.
5. All steel cleaned by sandblasting.

TABLE V

TENSILE SHEAR STRENGTH AFTER 30-DAY EXPOSURE
TO SIMULATED WEATHERING

Specimen No.	Exposure To	Test Temp. °F	Tensile Shear Shear psi	Tensile Shear % of Standard
17-7-38	Tap Water	Room	1360	110
17-7-46	Tap Water	Room	1350	109
Average	Tap Water	Room		110
17-7-40	Tap Water	600°	1180	95
17-7-48	Tap Water	600°	1200	97
Average	Tap Water	600°		96
17-7-42	Tap Water	800°	1230	99
17-7-50	Tap Water	800°	1340	108
Average	Tap Water	800°		104
17-7-44	Tap Water	1000°	1540	124
17-7-52	Tap Water	1000°	1320	106
Average	Tap Water	1000°		115
17-7-37	Salt Spray	Room	1210	98
17-7-45	Salt Spray	Room	1370	111
Average	Salt Spray	Room		105
17-7-39	Salt Spray	600°	1060	86
17-7-47	Salt Spray	600°	1060	86
Average	Salt Spray	600°		86
17-7-41	Salt Spray	800°	1100	89
17-7-49	Salt Spray	800°	860	69
Average	Salt Spray	800°		79
17-7-43	Salt Spray	1000°	1550	125
17-7-51	Salt Spray	1000°	1290	104
Average	Salt Spray	1000°		115

NOTES:

1. .050" 17-7 PH steel, tempered to the RH 950 condition, UI 117-63 ceramic adhesive with 28 mesh 304 stainless steel wire screen.
2. Pressure used - 50 psi.
3. Salt Spray per QQ-M-151. Tap Water maintained at room temperature and changed twice per week.
4. Standard Tensile Shear Strength = 1280 psi (See "Testing Procedures" for explanation).
5. All specimens fired during Firing No.4.
6. All steel cleaned by sandblasting.

TABLE VI

EFFECT OF COATING 17-7 PH
STAINLESS STEEL FOIL ON
APPARENT TENSILE STRENGTH

Sample No.	Coating Applied	Apparent Tensile Strength psi	Average Apparent Tensile Strength psi
2	none	171,000	
3	none	168,000	
4	none	174,000	
5	none	159,000	
6	none	211,000	177,000
7	UI 1067-1	207,000	
8	UI 1067-1	208,000	
9	UI 1067-1	207,000	
10	UI 1067-1	212,000	
11	UI 1067-1	206,000	
12	UI 1067-1	213,000	209,000
13	UI 117-63	100,000	
14	UI 117-63	164,000	
15	UI 117-63	166,000	
16	UI 117-63	185,000	
17	UI 117-63	157,000	
18	UI 117-63	124,000	149,000

NOTES:
1. Tensile coupons made of .005" 17-7 PH stainless steel, condition A.
2. All tests made at room temperature.
3. Apparent tensile strength was calculated on a basis of the original steel cross section.
4. All coupons were heat treated by the recommended process for RH 950 condition in air atmosphere.
5. UI 1067-1 is a glassy silica-boron oxide-soda ceramic. UI 117-63 is a mixture of carbonyl iron and UI 117-50.
6. Steel prepared by vapor degreasing.

TABLE VII

DATA FOR SLIP CONTROL STUDIES

Slip No.	Date of Test	Viscosity cps	Solids % by Weight	Sp. Gr.	B_2O_3 Grams/ 10 cc	Na_2O Grams/ 10 cc	Ratio Na_2O/ B_2O_3	pH of Slip	Total Time Hours	Dry Dip Weight g/sq. ft.
7-21-58-1	7-21-58	310	52.9	1.458	-	-	-	6.35	-	5.9
7-23-58-1	7-25-58	-	50.4	1.430	0.434	0.092	0.214	7.15	-	38.0
7-23-58-1	7-28-58	16,750	50.3	1.456	1.722	0.434	0.252	7.45	-	-
7-31-58-1	8-1-58	560	57.0	-	-	-	-	6.95	-	-
7-31-58-1	8-4-58	1,500	55.0	1.495	1.82	0.38	0.209	7.40	-	11.1
7-31-58-1	8-5-58	-	54.0	1.48	1.61	0.38	0.236	7.5	-	-
7-31-58-1	8-6-58	-	-	-	1.46	0.40	0.274	7.7	-	-
7-31-58-1	8-7-58	1,600	-	-	0.42	-	-	-	-	-
7-31-58-2	8-1-58	5,860	67.0	-	0.99	0.42	0.424	6.80	-	-
7-31-58-2	8-4-58	12,280	64.0	1.636	2.45	0.57	0.232	7.50	-	-
7-31-58-2	8-5-58	-	63.8	1.624	2.70	0.71	0.263	7.60	-	-
8-11-8-1	8-11-58	20,800	72.9	1.797	-	-	-	6.80	4	-
8-11-8-2	8-11-58	18,600	72.9	1.79	-	-	-	6.87	4	-
8-11-8-3	8-11-58	500	52.4	1.45	1.13	0.22	0.194	7.15	4	-
8-11-8-3	8-12-58	1,070	52.0	1.46	1.27	0.29	0.229	7.30	29	-
8-11-8-3	8-13-58	1,660	52.0	1.45	1.16	0.28	0.242	7.55	52	-
8-11-8-3	8-15-58	1,870	52.0	1.47	1.24	0.30	0.243	7.85	97	-
8-11-8-3	8-18-58	1,950	52.0	1.49	1.16	0.30	0.258	7.85	169	-
8-11-8-4	8-11-58	1,720	61.5	1.57	1.53	0.30	0.196	7.0	4	-
8-11-8-4	8-12-58	3,900	61.0	1.60	1.52	0.35	0.230	7.30	29	-

TABLE VII (Concluded)

Slip No.	Date of Test	Viscosity cps	Solids % by Weight	Sp. Gr.	B_2O_3 Grams/ 10 cc	Na_2O Grams/ 10 cc	Ratio Na_2O/ B_2O_3	pH of Slip	Total Time Hours	Dry Dip Weight g/sq.ft.
8-11-8-4	8-13-58	7,650	62.1	1.60	1.35	0.32	0.237	7.42	52	-
8-11-8-4	8-15-58	11,300	62.1	1.61	1.55	9.38	9.245	7.75	97	-
8-11-8-4	8-18-58	12,800	61.2	-	1.35	0.33	0.244	7.75	169	-
8-21-58-1	8-21-58	1,500	65.8	-	-	-	-	6.3	3.5	10.5
8-21-58-1	8-22-58	6,000	-	-	-	-	-	6.9	24	-
8-21-8-2	8-22-58	15,600	-	-	-	-	-	6.85	19	-
8-26-8-1	8-26-58	1,150	56.9	-	-	-	-	6.5	4	-
8-26-8-1	8-27-58	2,050	-	-	-	-	-	6.6	25	13.6
8-26-8-1	8-27-58	2,150	-	-	-	-	-	6.8	36	-
8-26-8-1	8-28-58	2,600	-	-	-	-	-	6.9	48	16.2
8-26-8-1	8-29-58	4,100	-	-	-	-	-	7.0	73	31.7
8-26-8-1	9-2-58	7,700	-	-	-	-	-	7.5	169	64.3
8-26-8-1	9-4-58	6,750	-	-	-	-	-	-	216	53.6
8-26-8-1	9-5-58	6,500	-	-	-	-	-	-	240	-
8-26-8-1	9-8-58	9,100	-	-	-	-	-	-	312	-
8-20-58	8-20-58	25,000	69.4	-	-	-	-	-	-	-
8-20-58	8-20-58	10,700	65.1	-	-	-	-	-	-	108.9
8-20-58	8-20-58	6,000	62.7	-	-	-	-	-	-	58.5
8-20-58	8-20-58	3,200	59.0	-	-	-	-	-	-	31.5
8-20-58	8-20-58	1,900	55.0	-	-	-	-	-	-	20.3

TABLE VIII

COMPOSITION OF SLIPS MENTIONED IN TABLE VII

PARTS BY WEIGHT	Slip No.	7-21-8-1	7-31-8-1	7-31-8-2	8-11-8-1	8-11-8-2	8-11-8-3	8-11-8-4	8-20-8-1	8-21-8-1	8-21-8-2	8-26-8-1	8-26-8-2
UI 1067 Frit		51.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7
UI 117 Frit		48.3	63.3	63.3	63.3	63.3	63.3	63.3	63.3	63.3	63.3	63.3	63.3
Total Frit		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Syloid 244		2.0	--	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Water		98.0	80.0	62.0	42.0	42.0	99.0	71.0	50.0	71.0	71.0	78.0	87.0
% Water		49.5	44.5	38.3	29.0	29.0	49.5	41.8	32.9	41.0	41.6	43.4	46.0

TABLE IX

ELECTROLYTES AND SOLVENTS EVALUATION
FOR DECREASING SET

Test No.	Quantity Added	Coating Thickness 10^{-3} inches
Electrolyte: None		
1	0	23-1/2
43	0	23
45	0	25-1/2
51	0	25-1/2
Electrolyte: Saturated Sodium Pyrophosphate		
2	10 drops	27-1/2
3	20 drops	28-1/2
4	30 drops	27
5	40 drops	24-1/2
6	50 drops	23-1/2
7	60 drops	22-1/2
8	70 drops	21
9	80 drops	18-1/2
10	90 drops	22
11	100 drops	20
12	125 drops	18
13	150 drops	16-1/2
14	175 drops	15
15	200 drops	10-1/2
42	200 drops	10-1/2
Electrolyte: Two Normal Hydrochloric Acid		
16	25 drops	25-1/2
17	50 drops	25-1/2
18	75 drops	22-1/2
19	100 drops	21
20	125 drops	20
21	150 drops	16-1/2
22	175 drops	13
23	200 drops	15
24	225 drops	13-1/2
Electrolyte: Two Normal Sodium Hydroxide		
25	25 drops	25-1/2
26	50 drops	25
27	75 drops	28
28	100 drops	25
29	125 drops	27
30	150 drops	26

TABLE IX (Concluded)

Test No.	Quantity Added	Coating Thickness 10^{-3} inches
Electrolyte: Two Normal Sodium Hydroxide (continued)		
31	175 drops	29
32	200 drops	24-1/2
33	250 drops	21
34	300 drops	21
35	400 drops	18-1/2
36	500 drops	13
Electrolyte: Two Normal Nitric Acid		
37	50 drops	23-1/2
38	100 drops	17-1/2
39	150 drops	17-1/2
40	200 drops	16-1/2
41	250 drops	12-1/2
Electrolyte: Six Normal Acetic Acid		
44	200 drops	10-1/2
Electrolyte: Ethyl Alcohol		
46	2 cc	26
47	40 cc	32-45
48	60 cc	22-30
49	80 cc	25-29
50	100 cc	22-27
Electrolyte: n-Butyl Alcohol		
52	5 cc	32-36
53	10 cc	30-34

NOTES:

1. For solutions, 20 drops are equivalent to approximately 1 cc.
2. Slip used consists of equal parts of UI 1067 and UI 117 frits milled with approximately 50% water and 2% Syloid.
3. When organic solvents were added, the slip deposited unevenly on the steel during the Dip-Test.

TABLE X

RELATIONSHIP OF BOND THICKNESS TO
TENSILE SHEAR STRENGTHS
FIRED AT 1650°F
1st SET

Bond Thickness inch	Avg. Bisque Thickness Each Coupon inch	Specimen No.	Bond Pressure psi	Firing No.	Room Temperature Tensile Shear Strength psi
.008	.008	163	50	1-2	1170
.0085	.008	165	50	1-2	1270
.0085	.008	166	50	1-2	1400
.0085	.008	167	50	1-2	1260
.0085	.008	168	50	1-2	1200
.0095	.008	164	50	1-2	1480
.010	.013	157	50	1-2	1260
.010	.013	158	50	1-2	1310
.010	.013	162	50	1-2	1350
.012	.023	155	50	1-1	1590
.012	.013	159	50	1-2	1490
.012	.013	160	50	1-2	1390
.012	.013	161	50	1-2	1340
.013	.023	152	50	1-1	1450
.013	.023	153	50	1-1	1410
.014	.023	151	50	1-1	1370
.014	.023	154	50	1-1	1260
.014	.023	156	50	1-1	1370
.0145	.017	173	23.5	1-3	1190
.0145	.017	170	23.5	1-3	1260
.015	.032	150	50	1-1	800
.015	.017	171	23.5	1-3	880
.015	.017	174	23.5	1-3	1020
.016	.032	145	50	1-1	560
.016	.032	147	50	1-1	660
.016	.017	172	23.5	1-3	380
.0165	.017	169	23.5	1-3	810
.017	.032	146	50	1-1	520
.017	.032	148	50	1-1	490
.019	.032	149	50	1-1	350

TABLE X (Concluded)

NOTES:

1. Lap shear specimens made with UI 117-63 adhesive at 1650°F firing temperature for 10 minutes.
2. Two pressures used on joint during firing - 50 psi and 23.5 psi.
3. All joints contained .008" thick 304 stainless steel 28 mesh screen.
4. All tests run at room temperature.
5. After firing and air cooling, the specimens were left at -100°F for 16 hours and heated at 950°F for 30 minutes.
6. All surfaces prepared by sandblasting.

TABLE XI

RELATIONSHIP OF BOND THICKNESS TO
TENSILE SHEAR STRENGTHS
FIRED AT 1650°F
2nd SET

Bond Thickness inch	Avg. Bisque Thickness Each Coupon inch	Specimen No.	Bond Pressure psi	Firing No.	Room Temperature Tensile Shear Strength psi
.008	.014	182	50	3-3	1910
.009	.014	176	50	3-3	1800
.009	.014	188	50	3-3	1680
.011	.014	177	10	3-1	1520
.011	.014	183	10	3-5	1650
.012	.014	187	30	3-2	1470
.012	.021	218	50	3-3	1230
.013	.014	189	10	3-1	1530
.013	.014	181	30	3-2	1440
.013	.021	212	50	3-3	1250
.013	.020	224	50	3-3	1210
.014	.032	206	50	3-3	1100
.015	.014	175	30	3-2	1210
.015	.032	194	50	3-3	990
.016	.021	213	10	3-1	980
.016	.032	200	50	3-3	940
.017	.021	219	10	3-1	870
.019	.021	211	30	3-2	480
.020	.021	223	30	3-2	640
.020	.021	225	10	3-1	650
.021	.021	217	30	3-2	510
.025	.032	207	10	3-1	310
.026	.032	195	10	3-1	170
Broke*	.032	201	10	3-1	Broke*
Broke*	.032	193	30	3-2	Broke*
Broke*	.032	199	30	3-2	Broke*
Broke*	.032	205	30	3-2	Broke*

*Broke when being removed from firing tool.

TABLE XI (Concluded)

NOTES: 1. 17-7 PH lap shear specimens fired at 1650°F for 10 minutes, rapidly air cooled, maintained at -100°F for 16 hours and aged at 950°F for 30 minutes.

2. Adhesive joints consisted of UI 117-63 ceramic adhesive and .008" 28 mesh 304 stainless steel wire screen.

3. Surface prepared by sandblasting.

4. All specimens tested at room temperature.

5. Bonding pressure as noted.

TABLE XII

RELATIONSHIP OF BOND THICKNESS TO
TENSILE SHEAR STRENGTHS
FIRED AT 1750°F

Bond Thickness inch	Avg. Bisque Thickness Each Coupon inch	Specimen No.	Bond Pressure psi	Firing No.	Room Temperature Tensile Shear Strength psi
.009	.014	184	50	3-6	2360
.009	.014	190	50	3-6	2220
.010	.014	178	50	3-6	1760
.010	.014	186	10	3-4	1660
.010	.014	192	10	3-4	1760
.010	.014	185	30	3-5	2100
.010	.021	221	30	3-5	2030
.011	.014	180	10	3-4	1670
.011	.014	191	30	3-5	2180
.012	.014	179	30	3-5	1970
.012	.021	227	30	3-5	1660
.012	.021	226	50	3-6	1990
.013	.032	202	50	3-6	1990
.013	.021	214	50	3-6	2040
.013	.021	220	50	3-6	1760
.014	.021	215	30	3-5	560
.014	.032	196	50	3-6	1830
.014	.032	208	50	3-6	1730
.017	.032	203	30	3-5	780
.018	.032	197	30	3-5	840
.018	.032	209	30	3-5	420
.020	.021	222	10	3-4	430
.024	.021	216	10	3-4	240
Broke*	.021	228	10	3-4	Broke*
Broke*	.032	198	10	3-4	Broke*
Broke*	.032	204	10	3-4	Broke*
Broke*	.032	210	10	3-4	Broke*

*Broke when being removed from firing tool.

NOTES: (See following page).

TABLE XII (Concluded)

NOTES: 1. 17-7 PH lap shear specimens fired at 1750°F for 10 minutes, rapidly air cooled, maintained at -100°F for 16 hours and aged at 950°F for 30 minutes.

2. Adhesive joints consisted of UI 117-63 ceramic adhesive and .008" 28 mesh 304 stainless steel wire screen.

3. Surface prepared by sandblasting.

4. All specimens tested at room temperature.

5. Bonding pressure as noted.

TABLE XIII

TEMPERATURE SURVEY
"Hevi-Duty" Furnace, Type Hot-243618-CU

Thermocouple No.

50		1-2		35		34		41		44		40	
Time	Temp.	Time	Temp.	Time	Temp.	Time	Temp.	Time	Temp.	Time	Temp.	Time	Temp.
1:18	1363	1:10	1388	1:08	1356	1:15	1394	1:13	1394	1:07	1337	1:11	1374
1:29	1390	1:22	1411	1:20	1385	1:24	1415	1:23	1402	1:21	1372	1:18	1389
1:41	1432	1:33	1459	1:33	1428	1:34	1449	1:30	1425	1:32	1411	1:30	1425
2:10	1520	1:47	1502	1:47	1472	1:46	1492	1:43	1468	1:46	1450	1:42	1470
2:32	1578	2:15	1576	2:16	1560	2:15	1578	2:12	1556	2:14	1537	2:11	1558
2:59	1652	2:35	1632	2:37	1618	2:36	1627	2:33	1614	2:34	1591	2:32	1618
3:12	1685	3:03	1704	3:06	1694	3:05	1708	3:01	1689	3:02	1671	2:59	1689
3:18	1700	3:12	1724	3:10	1709			3:10	1715	3:09	1694	3:10	1712

Thermocouple No.

45		39		33	
Time	Temp.	Time	Temp.	Time	Temp.
1:12	1372	1:09	1342	1:14	1372
1:23	1394	1:19	1371	1:25	1389
1:32	1433	1:31	1405	1:35	1425
1:45	1473	1:44	1449	1:48	1461
2:12	1555	2:14	1536	2:16	1547
2:33	1617	2:35	1591	2:37	1605
3:00	1689	3:02	1672	3:07	1681
3:08	1708			3:14	1698

Furnace Temperatures °F

Time	Back of Oven	Front of Oven
1:35	1500	1465
1:50	1540	1500
2:17	1610	1575
2:38	1700	1620
3:18	1750	1750
3:33	1750	1750

NOTES:
1. This survey was run on August 12, 1958, from 1 to 5 p.m.
2. Furnace loaded with pressure firing fixture containing 21 lap shear specimens.
3. Time is for total time in furnace.
4. Location: Aeronca Manufacturing Corporation, Ceramics Laboratory.

TABLE XIV

EFFECT OF SURFACE TREATMENT ON
TENSILE SHEAR STRENGTH OF UI 117-63 BOND

GENERAL INFORMATION

Adhesive used: UI 117-63 (includes carbonyl iron).
Adhesive applied by dipping.
.008" 28 mesh, 304 stainless steel mesh screen used in bond joint.
17-7 PH stainless steel lap specimens tempered to RH 950 condition.

Series 1.

Surface preparation - light sandblasting.

Vapor degrease, light sandblast at 45° to and 6" from surface. Two cross passes.

Sample No.	Bond Thickness inch	Failing Load lbs.	Assumed Area sq. in.	Ultimate Tensile Shear Strength psi	
229	--	700	.50	1400	
230	.013	838	.50	1680	Avg. = 1460 psi
231	.009	658	.50	1320	

Series 2.

Surface preparation - complete alkaline cleaning and light sandblasting.

Vapor degrease, immerse in Matawan 48W, 6 to 8 oz./gal. for 10 minutes at 160°F under electric current. Follow by a 10-minute cold-water rinse. Light sandblast at 45° to and 6" from surface. Two cross passes.

Sample No.	Bond Thickness inch	Failing Load lbs.	Assumed Area sq. in.	Ultimate Tensile Shear Strength psi	
232	.013	728	.50	1450	
233	.013	880	.50	1760	Avg. = 1650 psi
234	.010	866	.50	1730	

Series 3.

Surface preparation - etching in cold hydrochloric acid.

Vapor degrease. Immerse in Matawan 48W 6 to 8 oz./gal. for 10 minutes at 160°F under electric current. Follow by a 10-minute cold-water rinse. Immerse at room temperature in 20% HCl solution for 5 minutes. Rinse for 10 minutes in cold water.

TABLE XIV (Continued)

Sample No.	Bond Thickness inch	Failing Load lbs.	Assumed Area sq. in.	Ultimate Tensile Shear Strength psi	
235	.016	450	.50	900	
236	.012	612	.50	1220	Avg. = 1190 psi
237	.012	726	.50	1450	

Series 4.

Surface preparation - based on Aeronca method of cleaning 17-7 PH steel.

Vapor degrease. Immerse in Matawan 48W 6 to 8 oz./gal. for 10 minutes at 160°F under electric current. Rinse for 10 minutes in cold water. Etch for 15 minutes at room temperature in chemical bath consisting of 20% by volume H_2SO_4, sp.gr. 1.84, 10% by volume HNO_3 conc., 2% by volume HF, 68% by volume of water, and 1.0% by weight of $Na_2Cr_2O_7 \cdot 2H_2O$. Rinse for 10 minutes in cold water.

Sample No.	Bond Thickness inch	Failing Load lbs.	Assumed Area sq. in.	Ultimate Tensile Shear Strength psi	
238	.010	780	.50	1560	
239	.008	674	.50	1350	Avg. = 1460 psi
240	.009	740	.50	1480	

Series 5.

Surface preparation - based on Franklin Institute WS-22 cleaning of steel (WADC TR 55-87, Appendix I).

Vapor degrease. Immerse in Matawan 48W 6 to 8 oz./gal. for 10 minutes at 160°F under electric current. Etch for 10 minutes at 150-160°F in bath consisting of:

H_2SO_4 sp.gr. 1.84	10% by volume
Activol 57-X	0.5% by weight of H_2SO_4
Water	90% by volume

Rinse for 10 minutes in cold water. Immerse for 10 minutes at room temperature in a bath consisting of:

HNO_3 conc.	10% by volume
HF	2% by volume
Water	88% by volume

Rinse in cold water for 10 minutes.

TABLE XIV (Continued)

Sample No.	Bond Thickness inch	Failing Load lbs.	Assumed Area sq. in.	Ultimate Tensile Shear Strength psi	
241	.011	686	.50	1370	
242	.009	Broke in Handling			Avg. = 1350 psi
243	.010	664	.50	1330	

Series 6.

Surface Preparation - based on Franklin Institute WS-4 method of cleaning steel (WADC TR 55-87, Appendix I).

Vapor degrease. Immerse in Matawan 48W 6 to 8 oz./gal. for 10 minutes at 160°F under electric current. Rinse in cold water for 10 minutes. Immerse for 15 minutes at 120°F in bath consisting of:

H_2SO_4 sp.gr. 1.84	1.0 liter
Saturated sodium dichromate sol.	35.0 ml.

Rinse in cold water for 10 minutes.

Sample No.	Bond Thickness inch	Failing Load lbs.	Assumed Area sq. in.	Ultimate Tensile Shear Strength psi	
244	.012	516	.50	1030	
245	.012	474	.50	950	Avg. = 1040 psi
246	.010	570	.50	1140	

TABLE XV

EFFECT OF SURFACE TREATMENT ON
TENSILE SHEAR STRENGTH OF UI 1067-1 BOND

General Information

Adhesive used: UI 1067-1
Adhesive applied by spraying.
No screen used in lap joint.
17-7 PH stainless steel lap specimen tempered to RH 950 condition.
All surface preparation preceded by vapor degreasing, immersion in Matawan 48W, 6 to 8 oz./gal. for 10 minutes at 160°F under electric current and rinsed in cold water for 10 minutes.

Series 1.

Based on Franklin Institute WS-22 cleaning (WADC TR 55-87, Appendix I). Etched in two baths:

Bath 1

H_2SO_4 sp.gr. 1.84	10% by volume
Activol 57-X	0.5% by weight of H_2SO_4
Water	90% by volume

Bath 2

HNO_3 conc.	10% by volume
HF	2% by volume
Water	88% by volume

Immerse in Bath No. 1 for 10 minutes at 150-160°F. Rinse in cold water for 10 minutes. Immerse in Bath No. 2 for 10 minutes at room temperature. Rinse in cold water for 10 minutes.

Sample No.	Bond Thickness inch	Failing Load lbs.	Area sq. in.	Ultimate Tensile Shear Strength psi	
268	.003	578	.53	1090	
269	.004	840	.52	1610	
270	.004	682	.52	1310	
271	.004	732	.53	1380	
272	.003	756	.53	1430	Avg. = 1420 psi
273	.003	914	.52	1760	
274	.004	646	.51	1270	
275	.005	678	.52	1300	
276	.005	796	.50	1590	

TABLE XV (Continued)

Series 2.

Based on Shell Methods "B" and "C", (WADC TR 53-126, Part IV, pages 38-40). Etched in two baths:

Bath 1 (Shell Method "B")

HCl sp.gr. 1.18	500 gms.
H_2O_2 - 30%	20 gms.
Formaldehyde solution - 37%	100 gms.

Bath 2 (Shell Method "C")

H_2SO_4 sp.gr. 1.84	65 gms.
$Na_2Cr_2O_7 \cdot 2H_2O$	37 gms.
Water	1122 gms.

Immerse in bath #1 for 10 minutes at 140-160°F. Rinse in cold water for 10 minutes. Immerse in bath #2 for 10 minutes at 160-170°F. Rinse in cold water for 10 minutes.

Sample No.	Bond Thickness inch	Failing Load lbs.	Area sq. in.	Ultimate Tensile Shear Strength psi	
277	.004	676	.52	1300	
278	.004	740	.53	1400	
279	.004	620	.52	1190	
280	.004	710	.53	1340	
281	.004	504	.52	970	Avg. = 1230 psi
282	.004	638	.53	1200	
283	.003	684	.52	1320	
284	.002	618	.52	1190	
285	.005	594	.51	1160	

Series 3.

An etch designed for this work.
Etched for 5 minutes at 150-160°F in a bath consisting of:

HNO_3 conc.	20% by volume
HF	4% by volume
Water	76% by volume

Rinse in cold water for 10 minutes.

TABLE XV (Concluded)

Sample No.	Bond Thickness inch	Failing Load lbs.	Area sq. in.	Ultimate Tensile Shear Strength psi	
286	.003	874	.52	1680	
287	.003	568	.53	1070	
288	.003	706	.51	1380	
289	.002	796	.54	1470	Avg. = 1390 psi
290	.004	754	.54	1400	
291	.003	866	.54	1600	
292	.004	664	.52	1280	
293	.004	740	.52	1420	
294	.003	634	.52	1220	

TABLE XVI

SURFACE PREPARATION STUDY
EFFECT OF SURFACE TREATMENT ON ADHESION

GENERAL INFORMATION

1. .032" x 3" x 8" 17-7 PH stainless steel sheets used.
2. Sheets cleaned as indicated below.
3. UI 1067-1 adhesive applied by spraying. The bisque thickness was maintained at .013" ± .002".
4. After coating, the sheets were subjected to 1750°F for 5 minutes.
5. After coating, the coated sheet was bent 90° by pliers in approximately a 1" radius corner. Best adhesion indicates no spalling of the coating during bending. Good adhesion indicates spalling at the radius only. Fair adhesion indicates spalling back one-half inch from the radius. Poor adhesion indicates a spalling of the coating as soon as the sheet started to bend. Spalled coatings flaked from the sheet during the cooling.

DATA

SURFACE PREPARATION	DESCRIPTION OF SURFACE PREPARATION
Best adhesion with this preparation.	Note 1. Immerse in Matawan 48W, 6 to 8 oz./gal. for 10 minutes at 160°F under electric current. Follow by a 10-minute cold-water rinse.
1. Vapor degrease, alkaline clean per Note 1, heat scale at 1700°F for 30 minutes. Etch per Note 2 for 30 minutes. Neutralize per Note 3.	Note 2. Chemical composition of bath: HNO_3 20% by volume HF 4% by volume Water 76% by volume Immerse for time stated at 150-160°F. Follow by a 10-minute cold-water rinse.
Good adhesion with these preparations.	
2. Vapor degrease, sandblast per Note 4.	
3. Vapor degrease, sandblast per Note 4, alkaline clean per Note 1.	
4. Vapor degrease, sandblast per Note 4, etch per Note 2.	Note 3. Immerse in NaCN, 6 to 8 oz./gal. for 5 minutes. Follow by a 10-minute cold-water rinse.
5. Vapor degrease, sandblast per Note 4, etch per Note 2, neutralize per Note 5.	Note 4. Light sandblast at 45° to and 6" from surface. Two cross coat passings.

TABLE XVI (Continued)

SURFACE PREPARATION	DESCRIPTION OF SURFACE PREPARATION
6. Vapor degrease, sandblast per Note 4, etch per Note 2, neutralize per Note 3.	Note 5. Immerse in Matawan 48W, 6 oz./gal. for 5 minutes. Follow by 10-minute cold-water rinse.
7. Vapor degrease, alkaline clean per Note 1, heat scale at 1700°F for 30 minutes etch per Note 2 for 5 minutes.	Note 6. Shell "B" solution - chemical composition of bath: HCl 1.18 sp.gr. — 500 gms. H_2O_2 30% — 20 gms. Formaldehyde sol. 37% — 100 gms. Water — 450 gms.
Fair adhesion with these preparations.	Immerse for 10 minutes at 140-160°F. Follow by 10 minutes in cold-water rinse.
8. Vapor degrease, etch per Note 6, etch per note 7.	
9. Vapor degrease, alkaline clean per Note 1, etch per Note 8.	
10. Vapor degrease, alkaline clean per Note 1, etch per Note 6, etch per Note 7, neutralize per Note 3.	Note 7. Shell "C" - chemical composition of bath: H_2SO_4 sp.gr. 1.84 — 65 gms. $Na_2Cr_2O_7 \cdot 2H_2O$ — 37 gms. Water — 1122 gms.
11. Vapor degrease, alkaline clean per Note 1, etch per Note 2 for 30 minutes, neutralize per Note 3.	Immerse for 10 minutes at 160-170°F. Follow by 10-minute cold-water rinse.
Poor adhesion with these preparations.	
12. Vapor degrease, alkaline clean per Note 1, etch per Note 2 for from 5 to 60 minutes.	Note 8. Franklin Institute WS-22 chemical composition of Bath No. 1: H_2SO_4 sp.gr. 1.84 — 10% by volume Activol 57-X — 0.5% by wt. H_2SO_4 Water — 90% by volume
13. Vapor degrease, alkaline clean per Note 1, heat scale 1750°F for 10 minutes.	Chemical composition of Bath No. 2:
14. Vapor degrease, etch per Note 6, neutralize per Note 3.	HNO_3 conc. — 10% by volume HF — 2% by volume Water — 88% by volume

TABLE XVI (Concluded)

SURFACE PREPARATION	DESCRIPTION OF SURFACE PREPARATION
Spalled with these preparations.	Note 8. (Cont.) Immerse in Bath No. 1 for 10 minutes at 150-160°F. Rinse for 10 minutes in cold water. Immerse in Bath No. 2 at room temperature for 10 minutes. Follow by 10-minute cold-water rinse.
15. Vapor degrease only.	Note 9. Immerse in 30% HCl solution for 5 minutes at room temperature. Follow by 10-minute cold-water rinse.
16. Vapor degrease, alkaline clean per Note 1.	Note 10. Chemical compostion of bath: H_2SO_4 sp.gr. 1.84 — 20% by volume; HNO_3 conc. — 10% by volume; HF — 2% by volume; $Na_2Cr_2O_7 \cdot 2H_2O$ — 1.0% by weight; Water — 68% by volume. Immerse for 15 minutes at room temperature. Follow by 10-minute cold-water rinse.
17. Vapor degrease, alkaline clean per Note 1, etch per Note 9.	Note 11. Franklin Institute WS-4 chemical composition of bath: H_2SO_4 sp.gr. 1.84 — 1.0 liter; Sat. $Na_2Cr_2O_7$ solution — 35.0 ml. Immerse in bath for 15 minutes at 120°F. Follow by 10-minute cold-water rinse.
18. Vapor degrease, alkaline clean per Note 1, etch per Note 10.	Note 12. Aqua regia. Chemical composition of bath: 3 parts HCl conc.; 1 part HNO_3 conc. Immerse for 5 minutes at room temperature. Follow by 10-minute cold-water rinse.
19. Vapor degrease, alkaline clean per Note 1, etch per Note 11.	
20. Vapor degrease, alkaline clean per Note 1, etch per Note 12.	

TABLE XVII

EFFECT OF ELECTROLYTIC ALKALINE CLEANING ON TENSILE SHEAR STRENGTHS

Specimen No.	Cleaning Cycle Included Electrolytic Alkaline Cleaner	Load lbs.	Average sq. in.	Tensile Shear Strength psi
A-4	Yes	750	.52	1440
A-5	Yes	792	.54	1470
A-6	Yes	692	.54	1280
			Avg. =	1400
B-4	No	694	.53	1310
B-5	No	740	.52	1420
B-6	No	778	.53	1460
			Avg. =	1400

NOTES:

1. 17-7 PH steel lap shear specimens heat treated and aged to the RH 950 condition.

2. UI 1067-1 ceramic adhesive used.

3. Specimen prepared by vapor degreasing with or without electrolytic alkaline cleaning and rinse, heat scaled at 1700°F for 30 minutes, etch in 20% HNO_3, 4% by volume solution for 5 minutes at 150-160°F, water rinsed, neutralized in 6 oz./gal. NaCN solution at room temperature for 5 minutes, rinsed for 10 minutes and air dried.

4. Electrolytic alkaline cleaning, when used, consisted of immersing in Matawan 48W, 6 to 8 oz./gal. for 10 minutes at 160°F under electric current.

5. Bonding pressure - 50 psi.

6. All bond thicknesses were approximately .005" thick.

TABLE XVIII

DEVELOPMENT OF CORE COATING TECHNIQUES

1st SET

Slip Used: UI 117-50
Characteristics of Slip: 51% solids, 4000 cps viscosity
Method of Application: Dip
Position of Core During Firing: Flat
Firing Temperature: 1700°F
Time at Firing Temperature: 10 minutes
Methods of Cleaning for Individual Specimen:

Specimen 1. Degreased.

Specimen 2. Degreased, Matawan 48W electrolytic alkaline cleaned, water rinsed, dipped in 4N HCl, water rinsed.

Specimen 3. Core in "as received" condition.

Specimen 4. Degreased and prefired at 1500°F for 5 minutes.

Specimen 5. Degreased, Matawan 48W electrolytic alkaline cleaned, water rinsed, dipped in 20% HNO_3, water rinsed.

Specimen 6. Degreased, Matawan 48W electrolytic alkaline cleaned, water rinsed, dipped in 20% HNO_3, water rinsed, prefired at 1500°F for 5 minutes.

Results in all cases: Core oxidized to the point of crumbling.

2nd SET

Slip Used: UI 1067-1
Characteristics of Slip: 2000 cps viscosity
Method of Application: Dip
Position of Core During Firing: Flat
Firing Temperature: 1700°F
Time at Firing: 7 minutes
Method of Cleaning Individual Specimens:

Specimen 7. Degreased, dipped in 2 parts HNO_3, 1 part H_2O, for 2 minutes, water rinsed.

Specimen 8. Degreased, dipped in 1 part HNO_3, 1 part H_2O, for 2 minutes, water rinsed.

Results in both cases: Glass drained to the bottom of the core leaving little or no glass on 65% of the cell wall.

TABLE XVIII (Continued)

3rd SET

Slip Used: UI 1067-1
Characteristics of Slip: 2000 cps viscosity
Method of Application: Dip
Position of Core During Firing: Vertical
Firing Temperature: 1700°F
Time at Temperature: 7 minutes
Method of Cleaning:

Specimen 9. Degreased, dipped in 1 part HNO_3, 1 part H_2O for 2 minutes, water rinsed.

Results: Glass coated rather evenly, no puddling noted in core. Vertical firing apparently brought about this improvement. The glass pulled to the center walls leaving the edges of the cell with very thin coat of glass. Fifteen to 20% of the cell walls were exposed to oxidation because of lack of protection.

4th SET

Slip Used: UI 1067-1
Characteristics of Slip: 3500 cps viscosity, solids 33%
Method of Application: Dip
Position of Core During Firing: Vertical
Firing Temperature: 1700°F
Time at Firing Temperature: 5 minutes
Method of Cleaning:

Specimen 10. Degreased, Matawan 48W electrolytic alkaline cleaned, water rinsed, etched in 20% HCl, water rinsed.

Specimen 11. Degreased only.

Results: Coating on specimens 10 and 11 are generally as described for 3rd Set, Specimen 9.

5th SET

Slip Used: UI 1067-1
Characteristics of Slip: 3500 cps viscosity, solids 33.5%
Method of Application: Spray
Position of Core During Firing: Vertical
Firing Temperature: 1750°F
Time at Firing Temperature: 5 minutes

TABLE VIII (Concluded)

5th SET (Continued)

Method of Cleaning Specimen:

Specimen 12. Degreased, Matawan 48W electrolytic alkaline cleaned, water rinsed, etched in aqua regia, water rinsed.

Results: Glass formed a smooth coating. Edges protected on 75% of the cross sectional area. Cell wall protected over 90-95% of the total area.

6th SET

Slip Used: UI 1067-1
Characteristics of Slip: 3200 cps viscosity, specific gravity 1.16
Method of Application: Spray
Position of Core During Firing: Vertical
Firing Temperature: 1750°F
Time at Firing Temperature: 3-1/2 minutes
Method of Cleaning:

Specimen 13. Degreased, Matawan 48W, electrolytic alkaline cleaned, water rinsed, etched in 20% HNO_3, 4% HF at 150°F for 5 minutes, water rinsed.

Specimen 14. Same as No. 13 except in addition was given a final dip in 6 oz./gal. NaCN at room temperature and water rinsed.

Results: Glass formed a smooth coating. Edges protected on almost total cross section. Cell walls protected over 95-98% of total area. No perceptible difference between Specimen 13 or 14.

TABLE XIX

EFFECT OF ADDITIONS TO
50% UI 117 AND 50% UI 1067 MIXED SLIP
ON TENSILE SHEAR STRENGTH

Specimen No.	Bond Thickness inch	Load lbs.	Area sq. in.		Tensile Shear Strength psi
50% UI 117 & 50% UI 1067 - No addition					
124	.0004	1080	.54		2000
125	-.0002	932	.52		1790
126	.0003	1146	.53		2160
				Avg.	1980
307	.0018	840	.51		1650
308	.0005	810	.46		1760
309	.0017	892	.49		1820
				Avg.	1740
50% UI 117 & 50% UI 1067 - Plus 1% (-100 mesh) 304 PC stainless steel powder					
298	.0018	636	.51		1250
299	.0021	690	.52		1330
300	.0022	672	.54		1240
				Avg.	1270
50% UI 117 & 50% UI 1067 - Plus 1% Fe_2O_3, plus 10% (-100 mesh) 304 PC stainless steel powder					
310	.0043	612	.51		1200
311	.0047	458	.52		880
312	.0035	432	.52		830
				Avg.	970

NOTES:

1. 17-7 PH stainless steel lap shear specimens, heat treated and aged to the RH 950 condition.
2. All surfaces prepared and adhesive applied by recommended procedure.
3. All tests at room temperature.
4. Bonding pressure - 50 psi.
5. All weights based on 100% frit in slip.

TABLE XX

EFFECT OF ADDITIONS TO
UI 117-50 ADHESIVE
ON TENSILE SHEAR STRENGTHS

Specimen No.	Bond Thickness inch	Load lbs.	Area sq. in.	Tensile Shear Strength psi
UI 117-50 - No additions				
121	.0003	634	.54	1170
122	.0003	912	.52	1750
123	.0001	836	.52	1610
			Avg. =	1510
UI 117-50 - Plus 1% Fe_2O_3				
313	.0013	834	.53	1570
314	.0011	582	.52	1120
315	.0016	610	.52	1170
			Avg. =	1290
UI 117-50 - Plus 1% Fe_2O_3, plus 10% (-100 mesh) 304 PC stainless steel powder				
316	.0032	760	.51	1490
317	.0028	726	.52	1400
318	.0028	538	.53	1020
			Avg. =	1300
UI 117-50 - Plus 15% (-100 mesh) 304 PC stainless steel powder				
304	.0012	938	.52	1800
305	.0020	972	.52	1870
306	.0012	972	.57	1700
			Avg. =	1790

NOTES:

1. 17-7 PH stainless steel lap shear specimens, heat treated and aged to the RH 950 condition.
2. All surfaces prepared and adhesives applied by the recommended procedure.
3. All tests at room temperature.
4. Bonding pressure - 50 psi.
5. All weights based on 100% frit in slip.

TABLE XXI

EFFECT OF ADDITIONS TO
UI 1067-1 SLIP
ON TENSILE SHEAR STRENGTH

Specimen No.	Bond Thickness inch	Load lbs.	Area sq. in.	Tensile Shear Strength psi
UI 1067-1 - No Addition				
319	.0006	595	.49	1210
320	.0013	496	.48	1020
321	.0013	776	.50	1550
				Avg. = 1260
UI 1067-1 - Plus 10% (-100 mesh) 304 PC stainless steel powder				
322	.0039	912	.50	1820
323	.0036	576	.51	1130
324	.0033	824	.50	1650
				Avg. = 1530
UI 1067-1 - Plus 1% Fe_2O_3				
325	.0015	1034	.51	2030
326	.0016	1112	.51	2180
327	.0021	632	.53	1200
				Avg. = 1800
UI 1067-1 - Plus 2.6% Fe_2O_3, plus 26% (-100 mesh) 304 PC stainless steel powder				
130	.0025	1092	.51	2140
131	.0018	1040	.53	1960
132	.0013	754	.53	1420
				Avg. = 1840
UI 1067-1 - Plus 13% Fe_2O_3, plus 26% (-100 mesh) 304 PC stainless steel powder				
127	.0022	760	.54	1410
128	.0024	816	.54	1510
129	.0019	824	.54	1530
				Avg. = 1480
UI 1067-1 - Plus 2.6% Co_2O_3, plus 26% (-100 mesh) 304 PC stainless steel powder				
295	.0060	1092	.54	2020
296	.0025	866	.56	1550
297	.0050	1052	.53	1980
				Avg. = 1850

TABLE XXI (Concluded)

Specimen No.	Bond Thickness inch	Load lbs.	Area sq. in.	Tensile Shear Strength psi
UI 1067-1 - Plus 13% Co_2O_3, plus 26% (-100 mesh) 304 PC stainless steel powder				
301	.0016	662	.51	1300
302	.0037	578	.64	900
303	.0020	612	.60	1020
				Avg. = 1070
UI 1067-1 - Plus 1% Fe_2O_3, plus 10% carbonyl iron				
328	.0015	1195	.53	2250
329	.0012	1230	.51	2410
330	.0014	1170	.52	2250
				Avg. = 2300

NOTES:

1. 17-7 PH stainless steel lap shear specimens, heat treated and aged to the RH 950 condition.
2. All surfaces prepared and adhesives applied by the recommended procedure.
3. All tests at room temperature.
4. Bonding pressure - 50 psi.
5. All weights based on 100% frit in slip.

TABLE XXII

EFFECT OF ADDITIONS TO
UI 1067-1 SLIP ON
TENSILE SHEAR STRENGTH

Specimen No.	Bond Thickness inch	Load lbs.	Area sq. in.	Tensile Shear Strength psi
UI 1067-1 - Plus 5.8% Fe_2O_3, 34% (-325 mesh) 304 PC stainless steel powder				
343	.0020	990	.50	1980
344	.0017	1110	.50	2220
345	.0012	1062	.51	2080
				Avg. = 2090
UI 1067-1 - Plus 1.5% Fe_2O_3, 20% (-325 mesh) 304 PC stainless steel powder				
340	.0020	1018	.50	2040
341	.0022	638	.51	1250*
342	.0020	1146	.51	2250
				Avg. = 2150

*Sample dropped in handling; not included in average.

Specimen No.	Bond Thickness inch	Load lbs.	Area sq. in.	Tensile Shear Strength psi
UI 1067-1 - Plus 2.5% Fe_2O_3, 20% (-325 mesh) 304 PC stainless steel powder				
349	.0017	1128	.48	2350
350	.0015	1240	.51	2430
351	.0012	1090	.50	2180
				Avg. = 2320

NOTES:

1. 17-7 PH stainless steel lap shear specimens, heat treated and aged to RH 950 condition.
2. All surfaces prepared and adhesives applied by the recommended procedure.
3. All tests at room temperature.
4. Bonding pressure - 50 psi.
5. All weights based upon 100% frit in slip.

TABLE XXIII

TENSILE SHEAR STRENGTH
OF UI 1067-1A JOINTS
AT VARIOUS TEST TEMPERATURES

Specimen No.	Bond Line Thickness inch	Test Temperature	Tensile Shear Strength psi
360	.0025	Room	1780
361	.0014	Room	1700
Average		Room	1740
352	.0020	600°F	1830
353	.0011	600°F	1500
354	.0015	600°F	1750
Average		600°F	1690
355	.0010	800°F	1940
356	.0017	800°F	1960
357	.0012	800°F	2000
Average		800°F	1970
358	.0012	1000°F	2250
359	.0007	1000°F	2770
363	.0017	1000°F	2350
Average		1000°F	2460

NOTES:

1. Specimen No. 362 broke while being removed from firing jig.
2. 17-7 PH stainless steel specimens, heat treated and aged to the RH 950 condition.
3. Surface preparation and application of adhesive in accordance with recommended practice for facing sheets as reported in "Recommended Procedures for Fabricating Sandwiches".
4. UI 1067-1A is adhesive recommend for use in honeycomb sandwiches. Consists of UI 1067-1 with 2.0% Fe_2O_3 and 20% 304 PC stainless steel powder (-325 mesh).
5. No space screen used.
6. Fired at 1750°F for 10 minutes under 50 psi pressure.

TABLE XXIV

FLEXURAL STRENGTH
CERAMIC BONDED 17-7 PH STAINLESS STEEL
HONEYCOMB SANDWICH

CORE SHEAR STRESS AT FAILURE - PSI

Test Temperature °F	Core in 8" Direction				Core in 3" Direction			
	Specimen No.	10 min. Exposure	Specimen No.	192 hr. Exposure	Specimen No.	10 min. Exposure	Specimen No.	192 hr. Exposure
Room	L-15	310	-	-	W-3	330	-	-
	L-20	310	-	-	W-7	320	-	-
	average	310	-	-	average	325	-	-
600°	L-17	170	L-21	180	W-5(1)	600	W-4	230
	L-28(1)	270	L-24	200	W-6(1)	260	W-10	220
	average	220	average	190	average	430	average	225
800°	L-27	430	L-25	270	W-1	340	W-11	240
	L-16	350	L-26	420	W-8	320	W-23	310
	average	390	average	345	average	330	average	275
1000°	L-14(2)	280	L-22(2)	430	W-2(2)	250	W-12(2)	300
	L-19(2)	270	L-13(2)	390	W-9(2)	240	W-18(2)	380
	average	275	average	410	average	245	average	340

TABLE XXIV (Concluded)

(1) In these specimens, core shear failure (split across full width) initiated a ceramic bond shear failure in one facing.

(2) In these specimens, core shear instability failure was followed by a ceramic bond shear failure. The specimen also took a permanent set at this test temperature only.

(3) In the remaining specimens, only ceramic bond shear failure occurred.

NOTES:

1. Specimens fabricated from:

   a. .032" x 3" x 8" 17-7 PH stainless steel facing sheets

   b. 0.500" x 3" x 8" 17-7 PH stainless steel square cell honeycomb core, .002" foil, 1/4" cell size.

   c. UI 1067-1A ceramic adhesive.

2. Sandwiches treated per cycle to temper 17-7 PH steel to RH 950 condition.

3. Pressure during firing - 5 psi.

4. Fired at 1750°F for 10 minutes.

5. See "Recommended Procedure for Fabricating Sandwiches".

6. Exposed at test temperature for time noted in headings.

7. Tested by single point loading on a 6" span.

TABLE XXV

EDGEWISE COMPRESSION OF
CERAMIC BONDED 17-7 PH STAINLESS STEEL
HONEYCOMB SANDWICH

Test Temperature °F	EDGEWISE COMPRESSION - PSI							
	Core in 4" Direction				Core in 3" Direction			
	Specimen No.	10 min. Exposure	Specimen No.	192 hr. Exposure	Specimen No.	10 min. Exposure	Specimen No.	192 hr. Exposure
Room	E-9	76,000	-	-	E-3	84,700	-	-
	-	-	-	-	E-8	85,900	-	-
	-	-	-	-	average	85,300	-	-
600°	E-10	128,600	E-16	Broke*	E-12	112,500	E-2	100,300
	-	-	-	-	E-13	106,800	-	-
	-	-	-	-	average	109,600	-	-
800°	E-17	130,200	E-4	122,200	E-14	116,700	E-6	133,800
	-	-	-	-	E-15	118,200	-	-
	-	-	-	-	average	117,500	-	-
1000°	E-18	33,800	E-7	71,200	E-11	70,800	E-5	Broke*

*Broke during environmental exposure.

TABLE XXV (Concluded)

NOTES:

1. Each specimen 4" long, 3" wide.
2. Sandwich made of .032" facing sheets, 17-7 PH stainless steel, 0.500" 17-7 PH steel square cell honeycomb core - .002" foil, 1/4" cell size.
3. Sandwich treated per cycle to temper 17-7 PH steel to RH 950 condition.
4. Pressure during firing - 5 psi. Fired at 1750°F for 10 minutes.
5. See "Recommended Procedures for Fabricating Sandwiches".
6. Exposed at test temperature for time as noted in heading.
7. Tested in 4" direction.

TABLE XXVI

FORMULATIONS FOR FRITS
RAW MATERIAL
PARTS BY WEIGHT

	UI 1067	UI 117
Quartz	24.8	
$NaNO_3$	9.0	4.2
H_3BO_3	66.2	
Potash Feldspar		34.8
Borax		9.2
Soda Ash		16.5
$BaCO_3$		0.7
ZnO		9.2
$CaCO_3$		8.0
Na_2SiF_6		4.2
$BaO \cdot P_2O_5$		6.9
88.5% Vanadium Pentoxide		2.5
Aloxide (1)		3.8

(1) Al_2O_3 dust collector fines

TABLE XXVII

OXIDE COMPOSITIONS OF FRITS
PARTS BY WEIGHT

	UI 117	UI 1067
SiO_2	27.2	38.0
Na_2O	16.3	5.0
B_2O_3	4.0	57.0
Al_2O_3	13.0	
K_2O	5.1	
BaO	6.0	
CaO	5.4	
ZnO	11.0	
Na_2SiF_6	5.0	
P_2O_5	4.0	
V_2O_5	3.0	

TABLE XXVIII

CERAMIC ADHESIVE COMPOSITION

Parts By Weight	UI 117-50	UI 1067-1	UI 117-63	UI 1067-1A
Frit UI 117	100	100	63.7	
Frit UI 1067			36.3	100
Syloid	2	2		
Water	50	40	98	160-220
Carbonyl Iron, Type C			20	
Silicon Powder			10	
Fe_2O_3, Technical Grade				2.0
-325 304 PC stainless steel powder				20