

41385

PLAINTIFF'S
EXHIBIT
AL-1293

DESIGN OF 56SH NAILS FOR ASBESTOS SIDING

Memoranda written on June 18th and November 22nd, 1937, give reports of some experiments in attempting to produce an aluminum alloy nail equivalent to the cadmium or tin plated brass nail now in use. Mr. Jones of the Chicago Office had done some work on this problem and had felt that it would be possible for us to use a nail which had a larger outside diameter than .093", which, we had previously been advised, was the maximum. The present series of tests were run to show the effect of various types of grooves and various root diameters on the holding properties and driving properties. These tests were conducted in accordance with the tests outlined by Johns Manville Company specifications.

OBJECT OF THE INVESTIGATION:

The object of this investigation was, first, to determine what type of groove marking on the shank would be most suitable for the use of aluminum nails to obtain a satisfactory holding power, and, second, what size wire and what root diameter of the grooves would be required to obtain a nail with driving properties equivalent to brass nails.

MATERIAL USED:

The material used was 17S .095" diameter for the preliminary shape of the groove tests, and 56SH wire stabilized for 4 hours at 250°F. The sizes of 56SH wire were .083", .087", .091", and .095" diameter. The mechanical properties of the 56SH stabilized wire were as follows:

ATC 0034869

<u>Diameter</u>	<u>Tensile Strength</u>
.083"	60,050 lbs. per sq. in.
.087"	58,150 " " " "
.091"	60,180 " " " "
.095"	58,200 " " " "

The original tests were made on yellow pine blocks taken from the Carpenter Shop. Due to non-uniform results of this material, some seasoned yellow pine was obtained for later tests.

PROCEDURE:

A series of 17ST nails 1" long were made up with the following types of grooves or indentations on the shank:

- (1) Straight knurl 32 pitch
- (2) Straight knurl 40 pitch
- (3) Diamond knurl 40 pitch (both sharp and flat)
- (4) A drive screw thread
- (5) A drive screw thread with a diamond knurl (lower half drive screw thread, upper half diamond knurl)
- (6) 40 pitch standard thread (both sharp and flat threads)
- (7) 30 pitch annular grooves similar to the brass nails (both sharp and flat)
- (8) Plain nail
- (9) Nail caustic dipped 1-1/2 minutes to roughen surface

These nails were driven in the unseasoned yellow pine for 1/2" and the pressure required to push them and pull them from the wood was obtained from the testing machine in the Laboratory.

The 56SH nails were made with annular grooves and rolled to various root diameters on the various sizes of nails. The sides of the nails were in some cases filed flat to approximate a barbed nail. In some cases the nails were made with approximately 1/4" near the point plain without grooves, to start them into the wood more easily. Further study was made of the effect of the shape of the point upon driving by filing the point by hand to different angles ranging from 60° to 140° included angles. A round point, instead of the usual square point, was included for comparison.

The nails were made 1-1/2" and 2" long, as the 2" nail would be worse for driving qualities, while the 1-1/2" long nail was specified to be used in Johns Manville test specifications. A copy of these specifications is included in the Appendix.

The 56SH nails were tested by hand driving in the yellow pine, by measuring the load required to push the nails 1" into the wood and the load to pull the nails out of the wood, on the testing machine in the Laboratory, and were tested for bending strength on a special jig made up according to Johns Manville specifications.

RESULTS:

Figures given in Table I show the results obtained upon driving and pulling the 17ST nails of various types. The only type which had holding power equivalent to the annular groove nail was the 40 pitch thread which gave a smaller root diameter, and therefore would be more prone to bend in driving than the standard nail with annular grooves. Flattening the top of the annular groove nail by opening up the dies did not seriously affect the holding power, and it also gave a somewhat larger root diameter.

Results on the 56SH nails are somewhat confusing, as in the unseasoned yellow pine a good many of the nails bent on being pushed into the wood, even on the Laboratory testing machine. Table II shows the results of tests on these nails as compared with the standard brass nails. The holding power of the aluminum nail with the full grooves seems fully equal to that of the brass nail. Tables IIIa and IIIb show tests made at different times, so that changes in the wood may have taken place. The results are not directly comparable to those in the main table.

Table III shows the results of holding power tests in sea-

soned yellow pine of .091" diameter nails with the grooves formed to shallow depths to improve the driving characteristics. Results on full grooves with the sides ground off to approximate a barbed condition are included.

The bending tests on the stabilized nails given in Table IV indicate it will be necessary to use at least .091" diameter wire to obtain satisfactory results. Even at this size, the results are somewhat toward the low side of the specification. The difference between stabilized and unstabilized nails is very considerable as shown by the test, the unstabilized nails having a bending load about 12% greater than the unstabilized nail.

Table V shows the effect of varying the point angle on the driving characteristics of the nails. The values given are the average depth to which the nails could be driven by hand into unseasoned yellow pine. It is apparent that there is not much possibility of improving the driving characteristics by change in this direction.

Driving tests made by hand hammering the nails into either seasoned or unseasoned yellow pine were so variable as to be worthless as a basis of comparison. Even the standard brass nails usually bent before driving completely.

DISCUSSION OF RESULTS:

From the results it is apparent that nothing but the annular type of groove is suitable. Neither diamond or straight knurls gave a holding power comparable to that obtained with the grooves. A drive screw thread tended to turn when pulled from the wood, with very little pressure being required. In addition, this nail was not

stiff enough and bent during driving on the Laboratory machine. The use of annular grooves which had been filed off, producing a condition similar to that which would be obtained by barbing the wire on rolls, did not have holding power greater than 75% of that obtained on the standard brass nail.

The shallow grooves, which were in effect little more than lines in the surface of the nail, seemed to have a holding power very close to that of the full depth of groove. Even with the penetration of the dies giving a root diameter only about .002" lower than the outside diameter of the nail, the holding power was somewhat over 75% of the holding power of the brass nail driven into the same wood. The use of such a shallow groove would indicate that a burr on the surface was quite important in improving the holding power. The possibilities of using this type of grooving are very attractive because of the larger root diameter which would give a stiffer nail, and also because the outside diameter of the nail would not be greatly increased. The customer's dies for forming their asbestos shingles are made with a hole size of between .093" and .103". A nail larger in outside diameter than .093" would necessitate their redesigning their dies to make a larger hole. This would increase the differential in cost between the aluminum and the brass nail which they would have to have before they would consider changing to an aluminum nail.

From the results of the bending test it is apparent that a wire diameter of .091" or .095" will be required to produce a nail to meet their specification. Both of these sizes are on the borderline of meeting the bending requirement. This bending test was put in to insure a nail with satisfactory driving characteristics. Since the individual driving the nails will have a marked effect on their tendency to bend, it is felt that the only satisfactory way of seeing

whether the nails can be driven is for the customer themselves to run tests.

All of the tests on 56SHnails with the various types of grooves were made on stabilized material. This would have a yield strength of around 48,000# per sq. in. as compared to around 53,000-55,000# per sq. in. on unstabilized material. It is felt that if these nails are on the borderline of being acceptable to the customer, that an unstabilized nail should be satisfactory.

Reduction in the length of the groove from about 1" to 3/4" did not affect the holding power of the nail appreciably. The point of the nail was made smooth for about 1/4-3/8" to provide a pilot for starting the nail through the asbestos shingle. The holding power of these nails was very little different from that of the nails which had the grooves clear up to the point. However, the nail with grooves extending along the shank only 1/4" from the point did not give holding power equal to the fully grooved nail, so that it would not be possible to leave the larger section of the shank plain, with grooves just at the point, to increase the holding power.

CONCLUSIONS:

The results of the test in this report show that no other type of grooving will give a satisfactory holding power as the annular type of grooves specified by the customer.

The diameter of wire for an aluminum nail to obtain equal results to that of the brass nail must be at least .091". Unstabilized material is superior to stabilized material so far as the driving characteristics of the nail are concerned.

Superior driving characteristics can be obtained by back-

ing the dies off, so that the depth of groove is only about .001" to .002" compared to .007" or .008" on the brass nail. The holding power of such a nail is equivalent to about 80% of the holding power of the standard brass nail.

RECOMMENDATIONS:

It is recommended that samples of the stabilized nails of .091" diameter and .095" diameter with the shallow grooves be sent to the customer for their tests. If these are on the borderline of being satisfactory, some unstabilized nails which have aged at least a month since the fabrication of the wire should be prepared and sent for further trial.

A. HARTWELL

May 5, 1938.

TABLE I

HOLDING POWER OF .093" DIAMETER 17ST NAILS

<u>Description</u>	<u>Push IN</u>		<u>Pull OUT</u>		<u>Ratio of Pull to Push Average</u>
	<u>Range</u>	<u>Average</u>	<u>Range</u>	<u>Average</u>	
Plain	67-75#	72#	26-39#	33#	.46
Dipped	63-87#	72#	43-61#	50#	.69
Grooves Sharp	110-112#	111#	64-111#	89#	.80
Grooves Intermediate	86-92#	89#	76-84#	79#	.89
Grooves Flat	67-84#	76#	84-88#	86#	1.13
40 Threads Sharp	103-126#	111#	58-99#	77#	.69
40 Threads Intermediate	78-106#	92#	40-83#	66#	.72
40 Threads Flat	89-124#	103#	48-91#	64#	.61
Straight 40 Tooth Knurl	61-85#	74#	31-34#	33#	.45
Straight 32 Tooth Knurl	77-120#	93#	34-58#	47#	.50
32 Tooth Diamond Knurl					
Sharp	76-98#	89#	51-58#	55#	.62
32 " " " Flat	86-99#	92#	43-54#	49#	.53

Sharp indicates dies were pressed into full depth to form metal up to a sharp point.

Flat indicates dies were backed off to leave top of knurl or groove flat.

Intermediate indicates dies were backed off somewhat from sharp grooves but not as much as on the flat ones.

TABLE II

DATE ON HOLDING POWER OF 56SH STABILIZED GROOVED NAILS - UNSEASONED YELLOW PINE

Description	No. per lb.	Diameter		Push Range	Average	Pull Range	Average
		O.D.	Root				
.083 x 1-1/2 S	1250	.093	.078	148-207	178 2 of 3 bent		240 head pulled off
.083 x 2 S	948	.093	.078	71B-89B	80	All bent	
.083 x 2 R		.090	.081	91B-75B	83	All bent	
.087 x 1-1/2 S	1160	.095	.083	195-196	195	204-220	212
.087 x 2 S	887	.095	.083	111B-117B	114	All bent	
.087 x 2 R		.093	.085	103B-141B	125	All bent	
.087 x 2 P		.087		183-205	197	90-137	128
.087 x 2 D		.086		165-198	175	148 only 1 not bent	
.087 - 2 S $\frac{1}{4}$				133-142	140	All bent	
.091 x 1-1/2 S	1024	.100	.087	197-214	212	266-302	290
.091 x 2 S	840	.100	.086	114-206	154	All bent	
.091 x 2 R		.099	.089	227-242	234	314-316 all heads broke	
.091 x 2 P		.092		241-254	248	121 the 1 bent at 241	
.091 x 2 D		.091		210-256B	233	170 the 1 bent at 256	
.091 x 2 S $\frac{1}{4}$				235-254	244	149-197	173
.095 x 2 S	745	.104	.086	228-251	242	163-254	208
.095 x 2 R		.102	.090	189-270	218	218 all but 1 bent	
.095 x 2 P		.095		164-245	204	92-126	109

TABLE II - Cont'd.

Description	No. per lb.	Diameter		Push		Average		Pull		Average	
		U.D.	Root	Range	Range	Range	Range	Range	Range	Range	Range
.095 x 2 S $\frac{1}{4}$				167-185		178		126-197		162	
.095 x 2 B				157-240		198		137-177		152	
.095 P unstabilized nails											
Brass Nail Standard		.092	.078	201-231		212		198-345		273	
TABLE II-a											
.095 x 2 B				176-311		226.4		184-190		187.5	
.095 x 2 DrC				143-182		166.4		All bent			
.095 x 2 DrF				158-173.5		165.7		All bent			
Standard Brass Nails				149-273.5		215		Mostly bent		257.5	
TABLE II-b											
.091 x 2 R				189-288		241.9		197-311.5		254.5	
Samples from Amer. Steel & Wire (unstabilized)				129.5-189.5		167.5		153.5-280.5		205	
Standard Brass				127-201.5		174.6		139-255.5		191.2	

S - smooth grooved - Dies forced in to produce sharp grooves.
 R - rough grooved - Dies backed off to leave flat on top.
 P - Plain
 DrC - Drive threads with coarse knurl above to prevent turning in pulling out.
 DrF - Drive threads with fine knurl above to prevent turning in pulling out.

D - dipped.
 S $\frac{1}{4}$ - Grooved only $\frac{1}{4}$ " at bottom near point. Smooth grooves.
 B - Grooves filed off on side to approximate a barb.

TABLE III

SEASONED YELLOW PINE BLOCKS USED

<u>Description</u>	<u>O. D.</u>	<u>Root Dia.</u>	<u>Push</u>	<u>Average</u>	<u>Pull</u>	<u>Average</u>
.095 x 2 VS	.0975	.093	213-256	238	288-308	298
.095 x 2 SC	.0965	.095	238-290	262.1	259-303	275
.095 x 2 L	.0975	.094	240-281	254	244-284.5	262
.091 x 2 VS	.094	.086	235-256.5	243.3	279-296.5	287
.091 x 2 VS#1	.094	.089	200-242.5	217.6	240.5-304.5	265
.091 x 2 SC	.094	.090	241.5-254.5	245.8	241.5-248.5	245
.091 x 2 L	.094	.092	232-255.5	242	225.5-249.5	240.5
.091 x 2 R	.099	.089	252.5-255	254.3	229.5-330	301.5
.095 x 2 SB	.104	.086	194-244	222.6	200-233	214.6
.091 x 2 SB	.100	.086	195-228	212	184-214.5	197.6
Stand. Brass			193.5-202	202.6	290-364.5	326

VS - Very shallow grooves.

SC - Scratches.

L - Lines.

Vs#1 - There is a difference of .003" in the root diameter but they appeared the same, so they were listed separately.

R - Rough grooves with no lead.

SB - Smooth grooves with sides filed off to bottom of groove to approximate a barbed nail.

TABLE IV

BENDING FORCE TO PRODUCE .100 DEFLECTION

Description Specification	O.D.	Root Dia.	Force in Lbs. to deflect .100" Range	Average 30 minimum
Brass Nail	.092"	.078"	34.7-36.	35.7
.083" S	.093"	.078"	22-22.5	22.3
.083" R	.090"	.081"	23-23.5	23.2
.087" S	.095"	.083"	24-24.5	24.3
.087" R	.093"	.085"	24.5-26.5	25.3
.087" D	.086"		27.5-28.	27.7
.091" S	.100"	.086"	30-35.	31.6
.091" R	.099"	.089"	29.5-30.5	30.0
.091" P	.092"		33.3-34.	33.6
.091" D	.091"		32.5-33.	32.6
.095" S	.104"	.086"	32.7-33.5	33.1
.095" R	.102"	.090"	34.7-41.5	39.0
.095" P	.095"		37.5-38.5	37.9
.095" P Unstabilized			41.5-45.5	43.3
.095" DrC			33-36.	34.8
.095" DrF			34-34.	34.0
.091" Am. St. & Wire (Unstabilized)			32.5-36.5	34.6
.095" VS	.0975"	.093"	35.5-36.5	36.1
.095" Sc	.0965"	.095"	38-39.	38.3
.095" L	.0975"	.094"	37.5-38.	37.7
.091" VS	.094"	.086"	32-33.	32.6
.091" Vs#1	.094"	.089"	33.2-34.	34.6
.091" Sc	.094"	.090"		33.5
.091" L	.094"	.092"		33.5

TABLE V

EFFECT OF POINT ANGLE ON DEPTH OF DRIVING WITH HAMMER

<u>Shape</u>	<u>Included Angle of Point</u>	<u>Average Depth-Inches</u>
Standard Square Point	58°	4/8
Round Point	66°	5/8
Standard Square Point	66°	3/8
" " "	90°	3/8
" " "	100°	3/8
" " "	110°	7/16
The chisel point	66°	2/8
Points blunter than 140°		1/8

SPECIFICATION NUMBERS 65024-1 M-W-A-F-G 1 1/4" x #14 GAUGE
 65025-1 M-W-A-P-G 1" x #14 "
 65026-1 M-W-A-P-G 1 3/4" x #14 "
 65027-1 M-W-A-F-G 1 1/2" x #14 "
 65029-1 M-W-A-F-G 2" x #14 "

Page 1 of 3 pp

JOHNS-MANVILLE CORPORATION
 PURCHASING SPECIFICATIONS
 FOR TIN PLATED BRONZE CASING NAILS
 APPROVED DECEMBER 10, 1937.

I. Composition:

The nails shall be manufactured from Bronze wire.

II. Dimensions:

(a) Length: The lengths as measured from under side of head to end of point shall be:

1", 1 1/4", 1 1/2", 1 3/4", 2"

(b) Gage: The nails shall be #14 Gage; Stubs' (-0.083").
 Measurement shall be made prior to plating the nails.

(c) Shape of Point: Diamond point - Standard.

(d) Shape of Head: Flat button, 5/32" diameter, 1/64" thick (Min.)

Variations from the above values shall not be of greater magnitude than that allowed by the U. S. Government, Federal Specification FF-N-101 for Standard Brass Nails of similar sizes.

(e) Count per pound:

<u>Nail length</u>	<u>Count /lb.</u>
1"	550 Min.
1 1/4"	470
1 1/2"	400
1 3/4"	320
2"	280

III. Finish

(a) Formation:

Barbing of the annular ring type 30 ± 2 threads/inch, shall extend along the shank of the nail, 1/8" from the head end of the diamond point formation, to 1/4" from the head.

(b) Color:

Dull Tin.

ATC 0034882

(c) Coating:

The bronze nails shall be Tin plated either prior to or following the barbing operation. No subsequent acid treatment or coating shall be used to increase the holding power.

The coating thickness shall be .0003" min.

IV. Workmanship:

Nails shall be true to shape, well finished and reasonably free from imperfectly formed or misshapen pieces, and shall not vary from the specified tolerances. The nails shall be free from corrosion, reasonably smooth, free from either excessive deposits of Tin or uncoated spots.

V. Performance Requirements:(a) Bending:

Nails less than 1 1/2" long shall not be subjected to bending test.

Nails shall be supported on 3/8" diameter rollers, placed 1.125" apart (center distances). A bending load shall be applied at the center of the span through a 1/4" diameter rod or bar. During the load application, the center deflection shall be measured to the nearest one-thousandth of an inch (0.001"). The nails shall meet the following requirements:

1. Minimum ultimate load for any one nail - 30 lb.
2. Average ultimate load - 35 lbs. minimum.
3. Maximum center deflection at 30 lb. load - 0.1".

(b) Holding Power:

Nails shall be driven through a hole in a tin metal plate into a 2" x 2 1/2" x 3 1/2" seasoned yellow pine block. The hole in the metal plate shall be approximately 0.005 inches larger in diameter than the diameter of the barbing on the nail shank.

Following the nailing operation, both metal plate and wood block shall be gripped in suitable holders and mounted in a tensile testing machine. Load shall be applied at the rate of 150 lb. per minute in such a manner as to exert a direct tensile pull on the nails.

Nails are to be tested for holding power when driven across the grain, perpendicular to the annular rings of seasoned yellow pine.

The nails to be tested shall be 1 1/2" long and shall show the following characteristics:

1. The head of the nail shall in no case break free from the shank.
2. The nail shall have at least 75% of the holding power of the standard nail.* The average of not less than five tests with the standard nail* shall be construed as the holding power of the length of wood chosen for test use.

* Standard Nail - 1 1/2" Hassall Tin plated Bronze Casing Nails
- #14 gage - Ring barbing as described by the manufacturer is
"threaded with 30-V annular rings for one inch."

VI. Inspection:

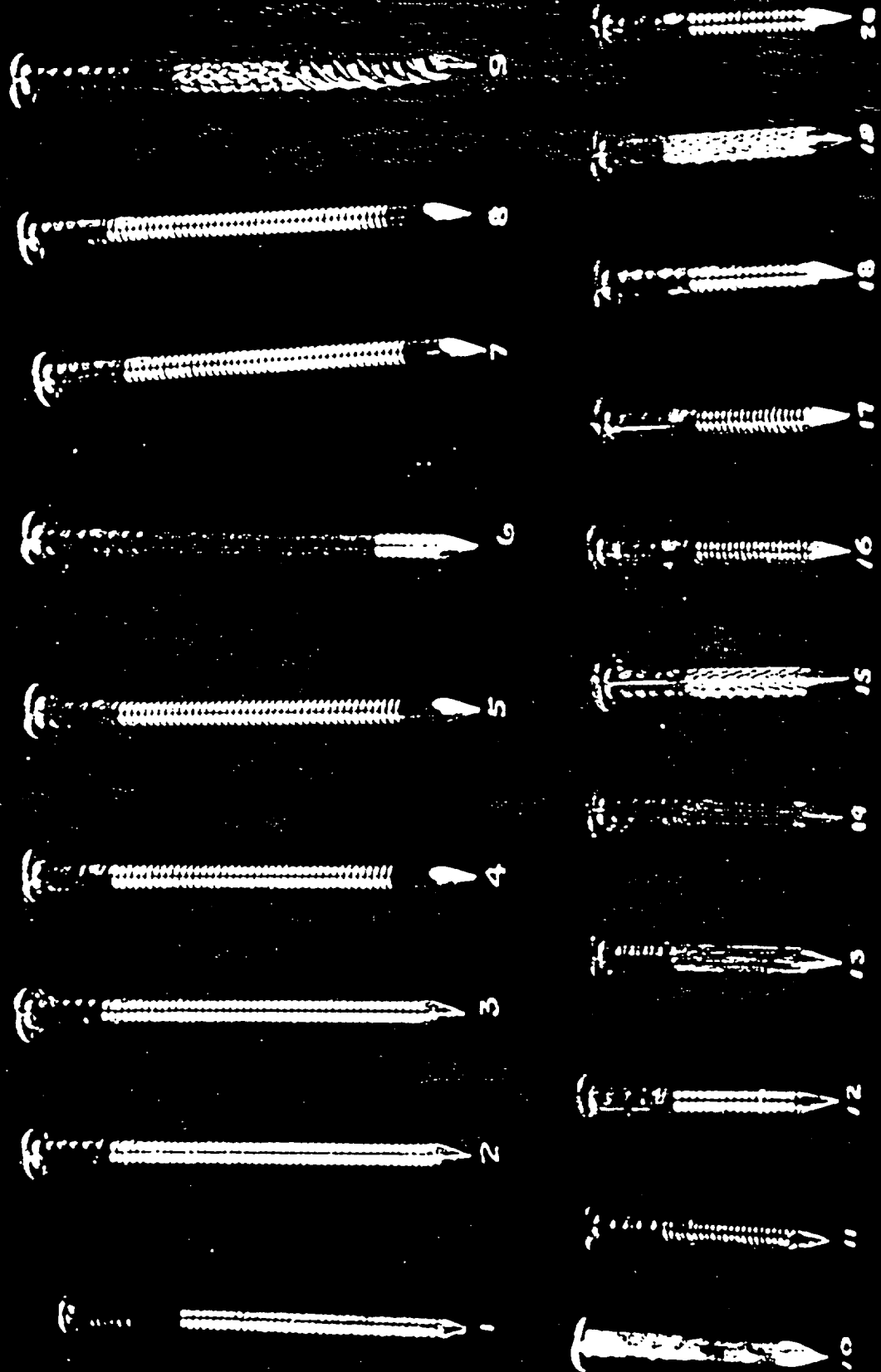
Four per cent (1 box out of every 25 boxes) of the nails received shall be inspected for the following characteristics:

- a. Type
- b. Class
- c. Material
- d. Size
- e. Finish
- f. Workmanship
- g. Net Weight
- h. Count

All specifications covering this particular material will be issued under serial numbers indicated above with a designating number following. The designating numbers will be changed with each revision of the specifications, while the specification numbers will remain the same. Reference to these specifications on requisitions and in correspondence must always include the designating number, else the reference is incomplete and the letter or requisition should be referred back for completion.

KEY TO NAILS PHOTOGRAPH

1. Brass Nail.
2. Smooth Grooved Nail.
3. Rough Grooved Nail.
4. Shallow-grooved with Pilot Point.
5. Shallow-grooved with Pilot Point- Large root diameter.
6. Smooth Grooves for 1/4" near End.
7. Scratches on Surface of Nail.
8. Lines on Surface of Nail.
9. Drive Screw Thread and Diamond Knurl.
10. Dipped 17ST Nail.
11. Sharp 40 Pitch Thread.
12. Sharp Grooves.
13. 40 Pitch Knurl.
14. 32 Pitch Knurl.
15. Flat Diamond Knurl.
16. Intermediate 40 Pitch Thread.
17. Flat 40 Pitch Thread.
18. Intermediate Grooves.
19. Sharp Diamond Knurl.
20. Flat Grooves.



ATC 0034886

INTERNAL CORRESPONDENCE

August 23, 1938.

FROM JOHN W. HOOD
METALLURGICAL DEPT.
EDGEWATER WORKS

To MR. R. L. TEMPLIN
NEW KENSINGTON

714
SEP 2 1938
Hartwell

RESEARCH
AUG 23 '38

RECEIVED

24/AUG 26 1938

24/AUG 26 '38

24

RE: DESIGN OF 56SH NAILS FOR ASBESTOS SIDING

This is to thank you for your letter of August 15th suggesting the use of 14S nails for this application.

You may be interested in the reasons why we have not previously tried this alloy for this purpose.

When the question as to the development of these nails first came up, we discussed the matter with Mr. Bossert as to the alloy to be used, including 14S, 27S, and 56S. At that time, Mr. Bossert felt that if 56S had satisfactory upsetting characteristics, it would be the better alloy from the corrosion standpoint, and we accordingly did no work on 14S. This question of corrosion continues to be important as the customer desires to have the nails stay light colored and not turn dark as long as possible. We believe that probably the high copper alloy would turn considerably darker than 56S.

The second factor which held us from using the 14S nails was the conclusion reached in Research Report PT34-11, of February 14th, 1934. 14ST nails were compared with 17ST and 27ST at that time, and the conclusion was reached that these nails were too brittle for general use. The mechanical properties quoted in that report on 14S were: Yield Strength 56,300#/sq.in., Tensile Strength 69,080#/sq.in. It is probably the Yield Strength which is the deciding factor in the bending of the nail during driving. We were obtaining a Yield Strength of around 56,000#/sq.in. before stabilizing in 56SH, and on the last lot of material we obtained a Yield Strength of 61,700#/sq.in. without stabilizing. Room temperature aging would drop the first material to

(by F.V.H.)

around 51,000#/sq.in. and the second to about 56,000-57,000#/sq.in. The material has been sent to the customer for test. With this material there would apparently be no appreciable advantage from the use of 14S, as the yield strengths would be about the same.

This question is a cost proposition throughout, and the use of a heat-treatable alloy would add about 5¢ a pound to the price of the nails, particularly if special equipment, such as a salt bath, were required for the 14S material. There would also be the further difficulty of the possible bending and distortion of the nails during heat treating. Although neither 50SH nor 14S are standard alloys for wire products in the .091" diameter size involved, it is probable from the comparison of 17S and 50S $\frac{1}{2}$ H prices, that there will be a further cent or two a pound difference in the material cost. We do not have a sufficient margin to enable us to pay these extra 6¢ a pound and still obtain the business, from present indications.

You may be interested to know that while the customer had stated that the material described in our report was about 40% under in bending tests, they ran some preliminary tests on unstabilized material which came through quite well, much to their surprise. They have sent these samples to their Development Division, but do not expect to be able to give us a report for at least two months, due to the vacation problem in the summer.

DICTATED BY
A. HARTWELL:S


JOHN W. HOOD

CC: Mr. E. H. Grotefend
Mr. T. W. Bossert

ATC 0034888

NOT RELEASED FOR PUBLICATION

ALUMINUM COMPANY OF AMERICA
Aluminum Research Laboratories
Chemical Metallurgy Division
New Kensington, Pa.

40847

No. XF-289

October 10, 1949

PERFORMANCE OF ALUMINUM ALLOY NAILS IN ASBESTOS CEMENT SIDING

INTRODUCTION:

Several years ago, when aluminum alloy nails were introduced to the building trades, tests were started to evaluate the merits of these nails for specific applications. One of these involved lathers' nails for plaster and another pertained to nails for asbestos cement siding. In both cases, the trade was concerned over the ability of the nails to resist the alkaline materials. In this connection, there is an erroneous belief that all alkaline materials are particularly corrosive to aluminum alloys. This is certainly true for caustic solutions and certain other alkaline materials, such as sodium carbonate or trisodium phosphate, but there are important exceptions to this. For example, aluminum alloys are resistant to ammonium hydroxide, calcium carbonate, and may have adequate resistance to other alkaline materials under the conditions encountered in service. This is apparent from the satisfactory use of aluminum alloys in contact with alkaline materials such as plaster and concrete. It is known that alkaline solutions of many compounds, such as sodium carbonate or trisodium phosphate, are not corrosive to aluminum alloys when sodium silicate is present (Figure 1). Asbestos cement siding also contains silicates and these might favorably influence the chemical properties of this material.

ATC 0034889

OBJECT:

To present laboratory tests and outdoor exposure data on the performance of aluminum alloy nails when used in asbestos cement siding; and to augment these with information on the resistance of aluminum alloys to other alkaline materials.

PROCEDURE AND RESULTS:

Commercial lots of asbestos cement siding representing six different manufacturers were secured on the open market for these tests. Chemical analysis revealed that all of these materials were alkaline in nature. They contained from 0.08% to 0.13% free lime⁽¹⁾ and a hot water leach of them had a pH of about 11.0 (at 25°C.). The tests employed 61S nails conforming to Federal Specification QQ-A-325.

Accelerated Laboratory Corrosion Tests

A number of tests were made in the laboratory. To simulate service conditions, most of the specimens used 61S siding nails driven through the asbestos siding and into yellow pine.

Some of these assemblies were continuously immersed and others were alternately immersed (1-1/2 minute cycles) in distilled water and tap water. A set of nails removed after 10 days' exposure were found to be in good condition. The nail heads and the shanks in contact with the siding were only slightly corroded. This is a relatively severe test because

(1) Method by Snell and Biffens, "Commercial Methods of Analysis", Chapter 13, P.277: hot alcohol and glycerol solution with titration using an alcoholic solution of ammonium acetate.

any water soluble alkali in the 2" x 6" panels of siding used were allowed to concentrate in the solutions, whereas, in service there is free drainage of water.

Other assemblies were exposed in a humidity chamber where continuous condensation occurs at a temperature of 125°F. In this test, the asbestos siding and the pine wood backing were thoroughly saturated with water. After 10 days in this test, the 61S siding nails were not corroded significantly. In an earlier test using roofing nails of 61S, no corrosion occurred even after 3 months' exposure. In this same test were galvanized steel siding nails. These suffered complete failure of the galvanized coating and rusting of the nails. Cadmium plated bronze nails endured this test as well as did the 61S nails.

A test panel containing a number of 61S siding nails was supported in an almost vertical position and tap water was allowed to flow continuously, but at a slow rate over the surface of the siding and, of course, the nail heads. Several nails were removed after 10 days' exposure and were found to be in good state of preservation. The surfaces in contact with the siding were only superficially etched.

Atmospheric Exposures

The encouraging results obtained with the preceding accelerated corrosion tests are being confirmed by outdoor exposures in progress on 61S nails driven in the six lots of asbestos cement siding.

The nails were driven through the siding and into yellow pine. One set was exposed to the industrial atmosphere at New Kensington, Pa., and a duplicate set was exposed to the seacoast atmosphere just 300 feet from the ocean at Point Judith, R.I. The high rates of attack shown in Figure 8 for mild steel demonstrate the relatively severe corrosive conditions existing at both of these locations. The assemblies were exposed at an angle of 45° which allowed rainfall and dew to flow over the asbestos cement siding and the nails.

A visual inspection after about two years' exposure showed that the 61S nails were in excellent condition at both test sites. The attack was too slight to be visually discernible. The surfaces exposed to the atmosphere showed only mild weathering and the under surface of the heads and the shank contacting the asbestos cement siding appeared to be free from attack. This is apparent from photographs in Figures 2, 3 and 4.

Microscopic examination was also made of longitudinal sections of these nails. The sections in Figure 5, and 6 are typical. These illustrate the excellent condition of the nails, especially of the heads and the shanks which had been adjacent to the asbestos cement siding. The depth of attack measurements made at 100 diameters are as follows:

Alloy	Exposure	Period	Depth of Attack			
			Head Exposed to the Weather		Head and Shank Contacting Siding	
			Avg.	Max.	Avg.	Max.
61S	Industrial Atmos.	2 yr.	0.0009"	0.0017"	0.0003"	0.0005"
61S	Seacoast Atmos.	2 yr.	0.0010	0.0021	0.0004	0.0008

It is apparent from a comparison of the above data with that in Figure 10 which were obtained on freely exposed aluminum alloys, that the asbestos cement siding has certainly not had any harmful effects. Also note from Figure 10 that the rate of weathering of aluminum alloys decreases to a very low value after about two years. This results from film formation. It is unlikely, therefore, that continued exposure will cause any real changes to occur in the already low rate of weathering of the 61S nails in asbestos cement siding.

In support of these findings is the satisfactory performance of 61S siding nails which were used to attach asbestos cement siding in six to eight large housing developments in the San Francisco area. The installations were made about 1-1/2 years ago and the aluminum alloy nails are reportedly in excellent condition.

Some Fundamental Considerations

In a dense material, such as asbestos cement siding, the rate at which alkali can be leached by water from the siding is of more importance than the total amount of alkali present in the product. The alkali would be expected to be leached more

rapidly from the surface layers than from the interior of the siding. The depleted surface layers would provide a diffusion barrier through which the alkali leach would have to diffuse before it could reach the surface of the siding and the nail heads. Hence, the total amount of alkali present is not of as much importance as the rate at which the alkali can be dissolved from the siding.

Two of the more important materials likely to be present in leaches of asbestos cement siding are lime ($\text{Ca}(\text{OH})_2$), and calcium carbonate (CaCO_3). Each of these compounds was added to distilled water to form a saturated solution and provide an excess as a fine powder. Specimens of commercially pure aluminum (2S-H14) were exposed to these saturated solutions, with the results given in the following table. For purposes of comparison, the rate of attack by sodium hydroxide (caustic) solution of about the same pH as the above solutions are included.

<u>Solution</u>	<u>pH</u>	<u>Depth of Attack inches per year</u>
Sat. $\text{Ca}(\text{OH})_2$	12.7	0.0105
4 g/l NaOH	12.8	1.6700
Sat. CaCO_3	9.3 - 9.5	0.0017
0.016 g/l NaOH	9.5	0.0040

The corrosion was uniform and the rates of attack in inches penetration per year were calculated from weight loss data. The rate of attack in the saturated solutions of the materials likely to be leached from the siding were low, being 0.0105 inches per year for saturated lime and 0.0017 inches per year for saturated solutions of calcium carbonate.

These rates are much lower than that in sodium hydroxide solutions having the same pH values. The significance of these data are at least two fold: (1) the alkaline nature of a solution as measured by pH is not an accurate criterion of the corrosiveness of a solution to aluminum alloys; and (2) the rate of attack in a saturated lime solution, although measureable, is not marked, evidently because of the formation of a protective film over the metal. Furthermore, lime solutions may have their pH reduced by absorption of carbon dioxide from the air with the formation of calcium carbonate. Consequently, part of the leachable lime on the surface of the siding may be converted to calcium carbonate. If this conversion occurs, the leach would be even less corrosive than a lime solution as shown by the above data.

A number of tests were made on a single lot of commercial asbestos siding to indicate the effect of repeated leaching on the alkalinity. This material had a free lime content of 0.05%, and a water leach of it had a pH of about 12.0. A 2" x 6" panel of the siding was coated with wax, except for the face intended for exposure to the weather. The panel was immersed in 150 ml of distilled water, and the pH of the solution was measured at various intervals from one minute to 22 hours. This water was not changed, but at the different periods, the panel was temporarily removed, rinsed, and immersed for one minute in fresh distilled water and the pH of this solution was measured. These data are given in Table I.

The data reveal that a fresh surface of asbestos cement siding provides enough alkali to increase the pH to about 11 during the first minute and continued leaching gradually increasing the pH to a value of about 12.2 in four hours. The intermediate immersions for one minute in fresh water, on the other hand, showed a gradual decrease in the pH to that of a neutral solution (pH 7.0), indicating a reduction in readily available alkali on the surface of the siding. This was especially true when the siding had been leached for four hours (Table I, Run 1). After 22 hours' immersion, the panel was transferred to fresh distilled water and allowed to remain until a pH of 10.0 was obtained. This required 40 minutes, whereas, only one minute was needed initially.

This relatively slow rate of leaching was demonstrated in another test in which water slowly flowed over a 12" high panel of the siding. Tap water with a pH of 7.5 was used. After two minutes, the drainage had a pH of about 10.0; after five minutes, the pH was 8.6. It then dropped to 7.8 in 90 minutes; and thereafter a pH of 7.5 was maintained, this being the same as that of the incoming tap water.

The immersion test and the flowing water test do demonstrate that the alkali on the skin of the asbestos siding is likely to leach away after a few rains, etc., and that thereafter water draining over the surface of the siding is not likely to pick up any significant amount of alkali. The density of the asbestos cement siding and the expected slow

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rate of diffusion of alkali in this hard material probably accounts for this favorable condition.

In view of the preceding results, it was reasoned that since diffusion of the alkali is evidently sluggish in this siding, it is unlikely that the shanks of the nails would be affected by more than the amount of alkali present in a narrow annular ring about the nail. It is apparent from the various corrosion tests that this has had very little effect. To illustrate this more quantitatively, the following test was conducted. The material (1.891 g.) from a 1/2" circle of the siding was powdered and mixed with 25 ml of water. Nails of 61S were then exposed to a number of these prepared solutions (pH 12.0). Two nails were removed after 4, 8, 24, 48, 72, and 96 hours' exposure, cleaned, and loss in weight determined. The results are given in Figure 7.

The weight loss data in Figure 7 show that the corrosion virtually stopped after about 48 hours' exposure, in spite of the fact that the solutions remained decidedly alkaline. Evidently, a protective film was formed on the 61S. Visual inspection disclosed that the nails had remained in very good condition, the corrosion being only superficial. This was confirmed by weight loss data showing that the corrosion had penetrated an insignificant 0.00046".

Aluminum Alloys Versus Other Alkaline Building Materials ATC 0034897

Aluminum alloys are also resistant to the action of other alkaline building materials, such as plaster, concrete,

magnesium oxychloride, magnesia block insulation. Some test data are briefly given to illustrate this.

Plaster

Laboratory tests have revealed that neither lime-base plaster or gypsum-base plaster had any harmful effect on 61S aluminum alloy nails. These plasters caused only superficial etching of the aluminum alloy nails and this served to increase the adhesion of the plaster to the nails. The nails had been driven in plasterboard, then covered with 3/8" thick layer of the above plasters, and a finishing coat containing 1 part plaster of Paris and 2 parts lime. One set was cured for 10 to 21 days and examined; a duplicate set was subsequently exposed to 100% relative humidity at 125°F for 7 days, at which time the plaster separated from the plasterboard. The corrosion in all cases was of a superficial etching type, the maximum attack being no greater than 0.0002". It might be added that large quantities of 61S lathers nails have been used for this application during the past two years and no corrosion problems have been reported.

Concrete

ATC 0034898

Freshly prepared concrete causes some uniform mild surface attack of aluminum alloys but this corrosion does not seem to continue after the concrete has hardened and cured. The data in Tables II, III and IV illustrate this. It is obvious from Table II that 3S-0 was only corroded to a depth of 0.0005" during a 4-1/2 day setting period. The oil coating on alloys 4S-0 and 52S-H38 was of no benefit, but again the corrosion was

superficial in nature. A second test in which concrete was poured into boxes made of 52S-H32 and the assemblies exposed to humid conditions or immersed in water showed (Table III) that the corrosion was slight after 6 months. The corrosion was not appreciably greater than that expected from the initial action of the wet concrete. Another test utilized 1" diameter rods of 53S-T6 imbedded in blocks of concrete. Some of these were stored indoors up to 8 years and others were exposed outdoors for 8 years to the industrial atmosphere at New Kensington, Pa. The data in Table IV disclose that the bond strength improved with time of curing and that the exposure did not have any corrosive effect that would be reflected in a decrease in strength. Sections of the rods were examined microscopically at 100 diameters and the depth of corrosion measured. These data in Table IV illustrate that the corrosion was shallow. Note that the metal imbedded in concrete was less corroded than those portions of the bars which had been freely exposed to the weather.

Magnesium Oxychloride Cement

Magnesium oxychloride cement prepared from magnesium oxide and magnesium chloride is alkaline in nature and a water leach of it had a pH of about 9.5. Laboratory tests have shown that aluminum alloys, including 61S-T6 have an inherently high resistance to corrosion by this material and one superior to galvanized steel or plain steel. Like concrete and plaster, this product causes superficial etching of aluminum during the casting and setting period, but subsequent exposure to alternately

ATC 0034899

wetting and drying conditions does not cause any further corrosion of significance.

Corrugated Asbestos Cement Roofing

A sample of commercial grade corrugated asbestos cement roofing about 1/4" thick was placed in contact with 61S-T6 and exposed for 3 months to 100% relative humidity at 125°F. This material contained 3.8% free lime and a water leach of it had a pH of 11.6. Although the material was alkaline in nature, it did not cause any corrosion of 61S-T6 in the three month period, even though the specimens were continually wet during this time.

85% Magnesia Insulation

Small blocks of 85% magnesia insulation were sandwiched between panels of several aluminum alloys, including 3S-H14 and 4S-H14 and exposed for 6 months in the 100% humidity chamber at 125°F. The samples were continuously wet and visual inspection revealed that the panels were in excellent condition and free from corrosion. The magnesia insulation was alkaline, a water leach of it having a pH of 9.9.

Resistance to Atmospheric Weathering of Aluminum Alloys

The data in Figures 8, 9, and 10 are included to illustrate the high inherent resistance to corrosion of aluminum alloys when exposed to a wide variety of weathering conditions, including seacoast, industrial, urban, rural, and tropical. There is very little difference in the resistance to weathering of aluminum alloys 2S, 3S, 4S, 52S, 53S, and 61S; consequently, the data for one alloy may be used to indicate the expected

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performance of the other alloy. It is apparent from the data that aluminum alloys weather at a slow rate and that the rate decreases to a very low value after about two years. Obviously, data obtained within two years can give a good measure of the ultimate performance expected of these aluminum alloys with regard to their resistance to weathering. The aluminum alloys were, of course, outstandingly superior to steel (Figure 8), and comparable to copper (Figure 9). In some strongly industrial atmospheres rich in sulphur bearing products, aluminum alloys would be expected to be superior to copper.

The surface appearance of aluminum alloys will vary with the nature of the environment. Aluminum will generally weather to a light gray, which should blend well with asbestos cement siding. In industrial atmospheres, the accumulation of industrial soil will cause some darkening of the nail heads; this is true of not only aluminum nails but nails of any other commercial alloy. The siding may also be darkened somewhat so that the nail heads should not be conspicuous.

CONCLUSIONS:

Since we understand that the six lots of asbestos cement siding are representative of commercial production, it is believed that the following conclusions are justified.

- (1) Accelerated laboratory corrosion tests and exposures for two years to relatively severe industrial and seacoast ATC 0034901 atmospheres show that aluminum alloy (61S) nails are suitable for use in asbestos cement siding. The 61S nails revealed a

high inherent resistance to weathering, and contact with the siding of six different manufacturers did not have any corrosive effect on the nails.

(2) Furthermore, 6LS nails employed 1-1/2 years ago in several large asbestos cement siding installations in San Francisco are reportedly giving excellent service.

(3) Laboratory tests show that this favorable performance might be attributed to the following: the dense nature and stability of asbestos cement siding actually resists the leaching of appreciable amounts of alkali from the siding; and, in addition, 6LS nails are resistant to any alkali leached from the siding under service conditions, because of a protective film which the metal develops.


C. J. WALTON

CJW:mcc

TABLE I
EFFECT OF LEACHING THE FACE OF ASBESTOS CEMENT SIDING
ON THE RESIDUAL ALKALINITY OF THE SURFACE

Run	Distilled Water Not Changed ⁽¹⁾		Panel Temporarily Immersed ⁽²⁾ In Fresh Distilled Water	
	Exposure Period	pH	Exposure	pH
1	1 min.	11.0	1 min.	10.0
2	5 min.	11.6	1 min.	9.9
3	10 min.	11.4	1 min.	9.7
4	15 min.	11.5	1 min.	9.7
5	30 min.	11.7	1 min.	8.7
6	1 hr.	11.7	1 min.	9.6
7	2 hr.	11.5	1 min.	9.2
8	4 hr.	12.2	1 min.	7.0
9	6 hr.	12.2	1 min.	7.2
10	22 hr.	11.5	1 min. (3)	7.0
11			5 min. (3)	7.0
12			10 min. (3)	8.6
13			15 min. (3)	9.1
14			25 min. (3)	9.8
15			30 min. (3)	9.9
16			40 min. (3)	10.1

- (1) Panels 2" x 6" entirely waxed except for the face which is intended for exposure to the weather, immersed in 150 ml of distilled water, the initial pH of which was 5.5. The gradual increase in pH was caused by the leaching of alkali from the siding.
- (2) Panel was temporarily removed from the solution (1) above, rinsed, and immersed for 1 minute in fresh distilled water, and the pH measured.
- (3) After 22 hours of leaching in solution (1), the panel was placed in fresh distilled water and allowed to remain to determine the time required to again produce a pH of about 10.0.

CJW:mcc

ATC 0034903

TABLE II
RESISTANCE OF ALUMINUM ALLOYS TO CORROSION
BY CONCRETE DURING THE SETTING PERIOD

<u>Alloy</u>	<u>Surface Oiled</u>	<u>Setting Period</u>	<u>Depth of Attack</u>	
			<u>Average</u>	<u>Maximum</u>
3S-0	No	4.5 da.	0.0005"	0.0005"
4S-0	Yes	4.5 da.	0.0008	0.0008
52S-H38	Yes	3.5 da.	0.0003	0.0004

Note: (1) Specimens were 0.064" x 4" x 6".

(2) Portlant cement: SiO₂ 21.7%, Al₂O₃ 4.9%, Fe₂O₃ 4.2%, CaO 61.4%, MgO 4.4%, SO₃ 1.6%, Ignition Loss 0.73%.

(3) Mix used: 1 part cement, 2 parts sand, 4 parts gravel.

CJW:mcc

TABLE III

RESISTANCE TO CORROSION OF 52S-H32 ALUMINUM
ALLOY IN CONTACT WITH CONCRETE AND EXPOSED
UNDER MOIST OR WET CONDITIONS FOR SIX MONTHS

<u>Alloy</u>	<u>Exposure</u>	<u>Depth of Attack(1)</u>		<u>Appearance</u>
		<u>Average</u>	<u>Maximum</u>	
52S-H32	100% relative humidity at 30°C	0.0011"	0.0014"	Uniform etching
52S-H32	Alternately to 100% humidity at 52°C and to refrigerator at 4°C	0.0008	0.0019	Uniform etching
52S-H32	Half immersed in tap water	0.0005	0.0009	Uniform etching

Note: (1) Corrosion of surface in contact with the concrete.

(2) Bubble slag concrete had been poured into shallow
boxes formed from 52S-H32 sheet 0.032" thick.

CJW:mcc

ATC 0034905

TABLE IV

53S-T6 ALUMINUM ALLOY RODS (1" DIA.) IMBEDDED IN CONCRETE(1) AND
STORED INDOORS OR EXPOSED OUTDOORS AT NEW KENSINGTON, PA. FOR EIGHT YEARS

Location of Sections Examined(2)	Depth of Attack(2)				Bond Strength - psi		
	Stored Indoors		Exposed Outdoors		Stored		Exposed
	Avg.	Max.	Avg.	Max.	Indoors	Outdoors	Outdoors
In concrete	0.0017"	0.0050"	0.0019"	0.0038"	28 da.	249	336
Concrete-air interface	0.0013	0.0021	0.0020	0.0034	1 yr.	334	518
Away from concrete (exposed to Atmos.)	0.0006	0.0012	0.0038	0.0064	8 yr.	402	

Note: (1) Unprotected 1" diameter rods set in 8" dia. x 8" deep blocks of concrete.
(2) Determined by microscopic examination of cross sections at the different zones listed.

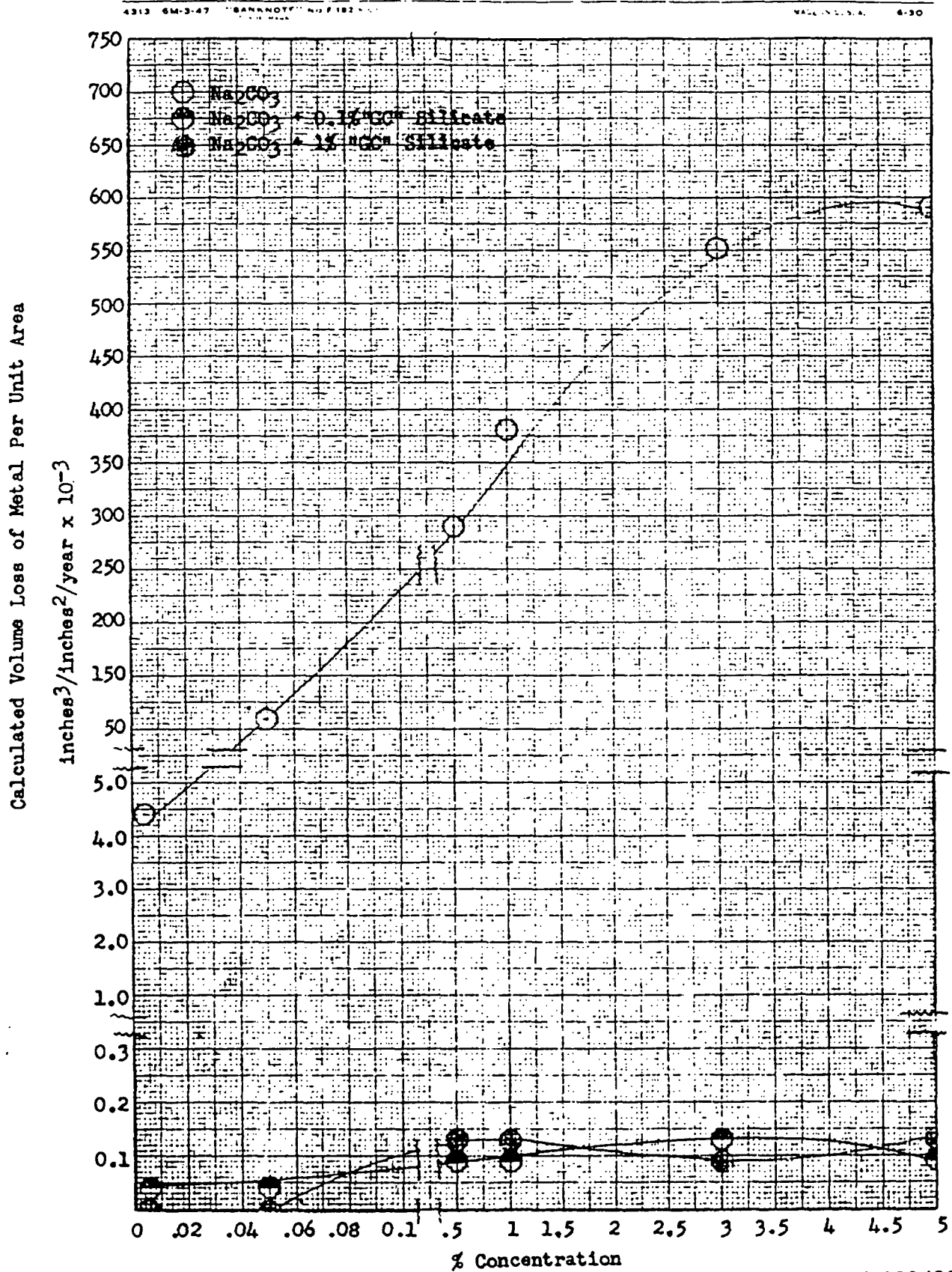
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Figure 1

Tests were made on aluminum alloy 2S-H14. The solutions were alkaline; the pH of the sodium carbonate ranged from 9.0 to 11.4. The pH of the sodium carbonate - sodium silicate solutions ranged from 10.3 to 11.3. The straight sodium carbonate solution was corrosive, whereas, the sodium carbonate - silicate solutions were not significantly corrosive even though alkaline.

REPORT NO. _____ DATE _____

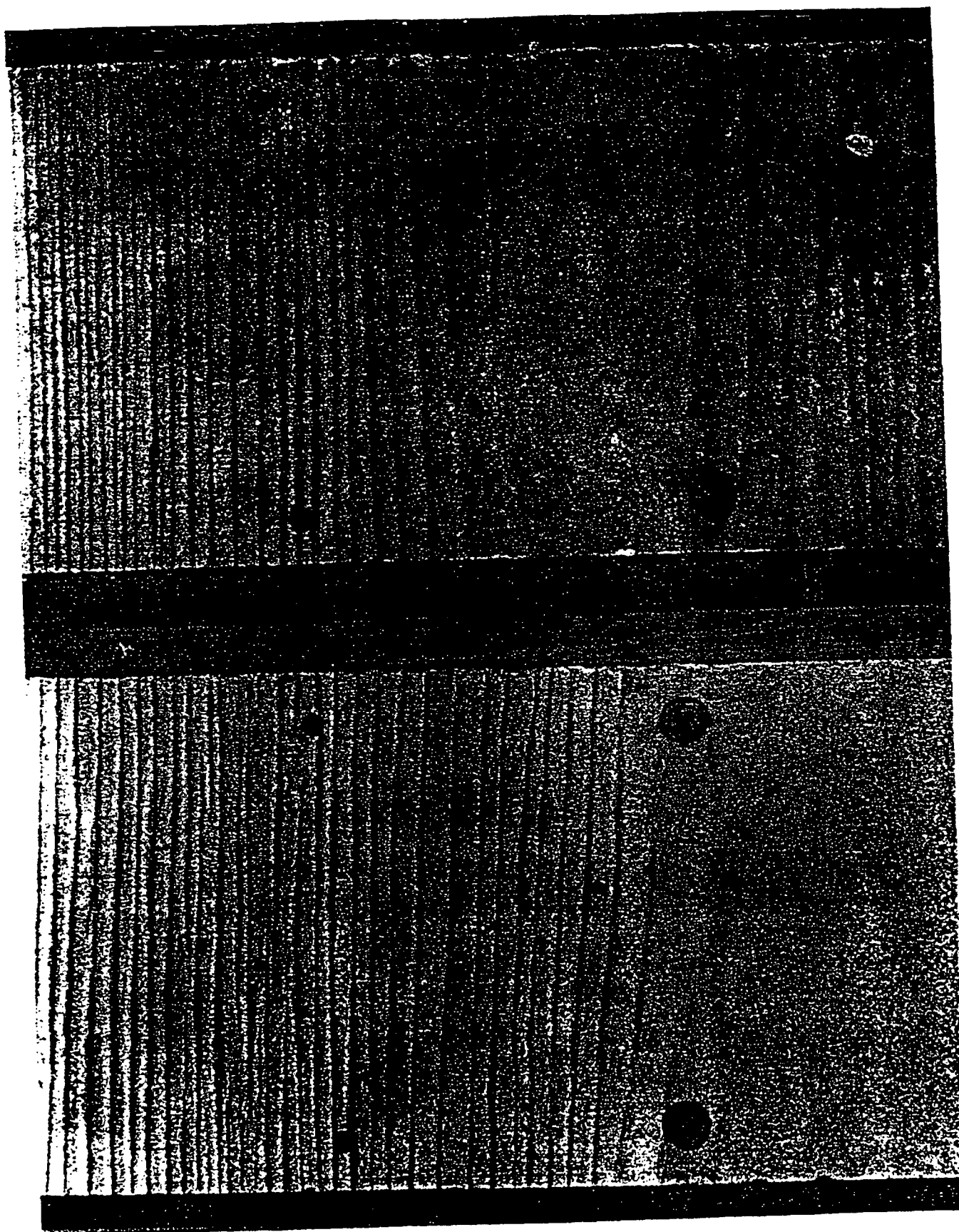
FIGURE NO. 1

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Figure 2

Neg. No. 56410C

Photograph showing condition of nails and asbestos cement siding after two years' exposure to either the sea-coast atmosphere at Point Judith, R.I., or the industrial atmosphere at New Kensington, Penna.



ATC 0034910

Figure 3

Neg. No. 56445A

Photograph showing good condition of 61S nails
as removed from asbestos cement siding after two years'
exposure to the industrial atmosphere at New Kensington,
Penna.

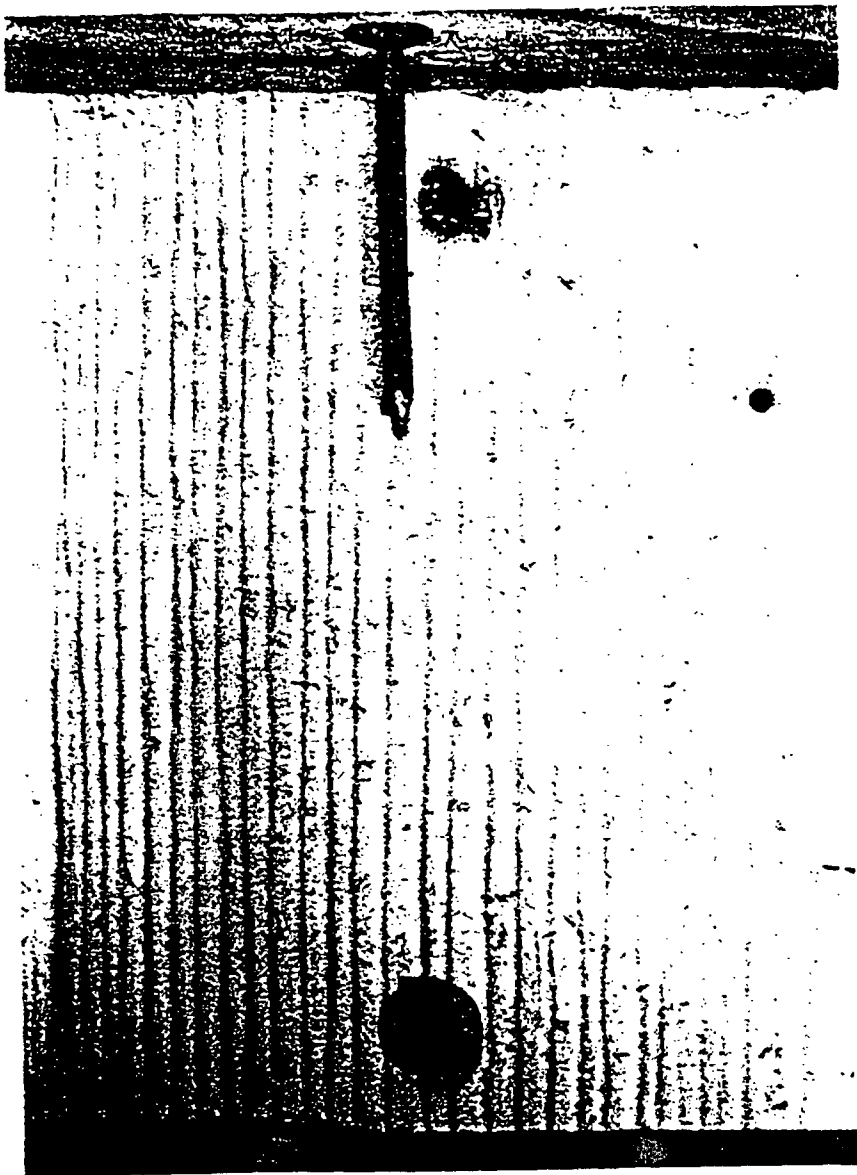


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Figure 4

Neg. No. 56344A

Photograph showing good condition of 61S nails as removed from asbestos cement siding after two years' exposure to the seacoast atmosphere at Point Judith, R. I.



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Figure 5

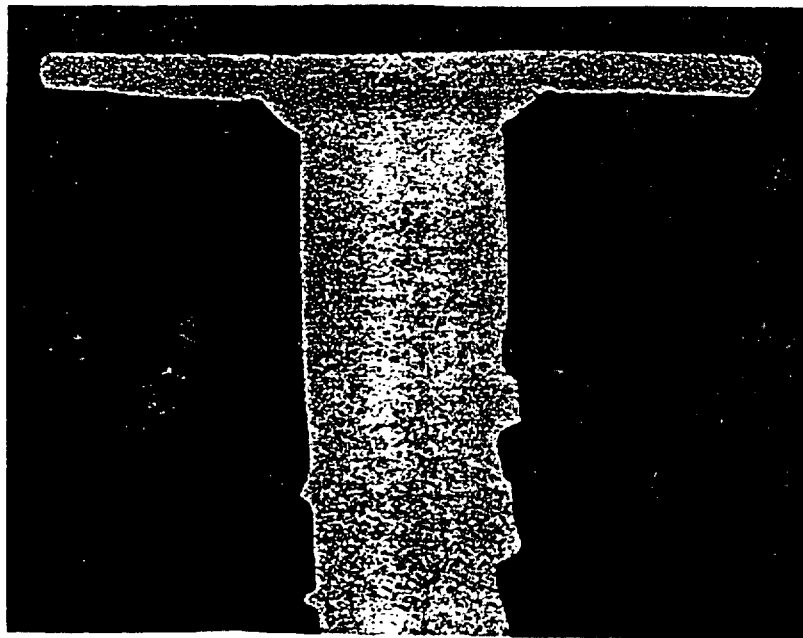
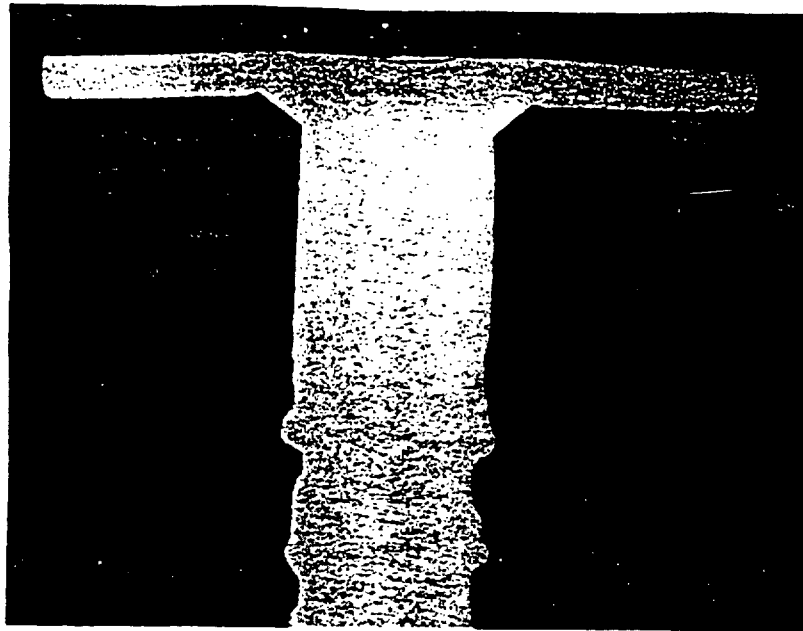
Neg. No. 60309
Mag. about 8X

Photomicrograph of longitudinal section of head and shank of 61S nail (roofing) driven in asbestos cement siding and exposed two years to industrial atmosphere at New Kensington, Penna. No significant corrosion had occurred. Sliver on top, center of head and ridges and valleys of shank are mechanical in origin.

Figure 6

Neg. No. 60310
Mag. about 8X

Photomicrograph of longitudinal section of head and shank of 61S nail (roofing) driven in asbestos cement siding and exposed two years to seacoast atmosphere at Point Judith, R.I. No significant attack was evident. Ridges and valleys of shank were mechanical.



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FIGURE NO. 7

42-1 6M-6 CV

BANKNOTE NO. 7182-810

K 20

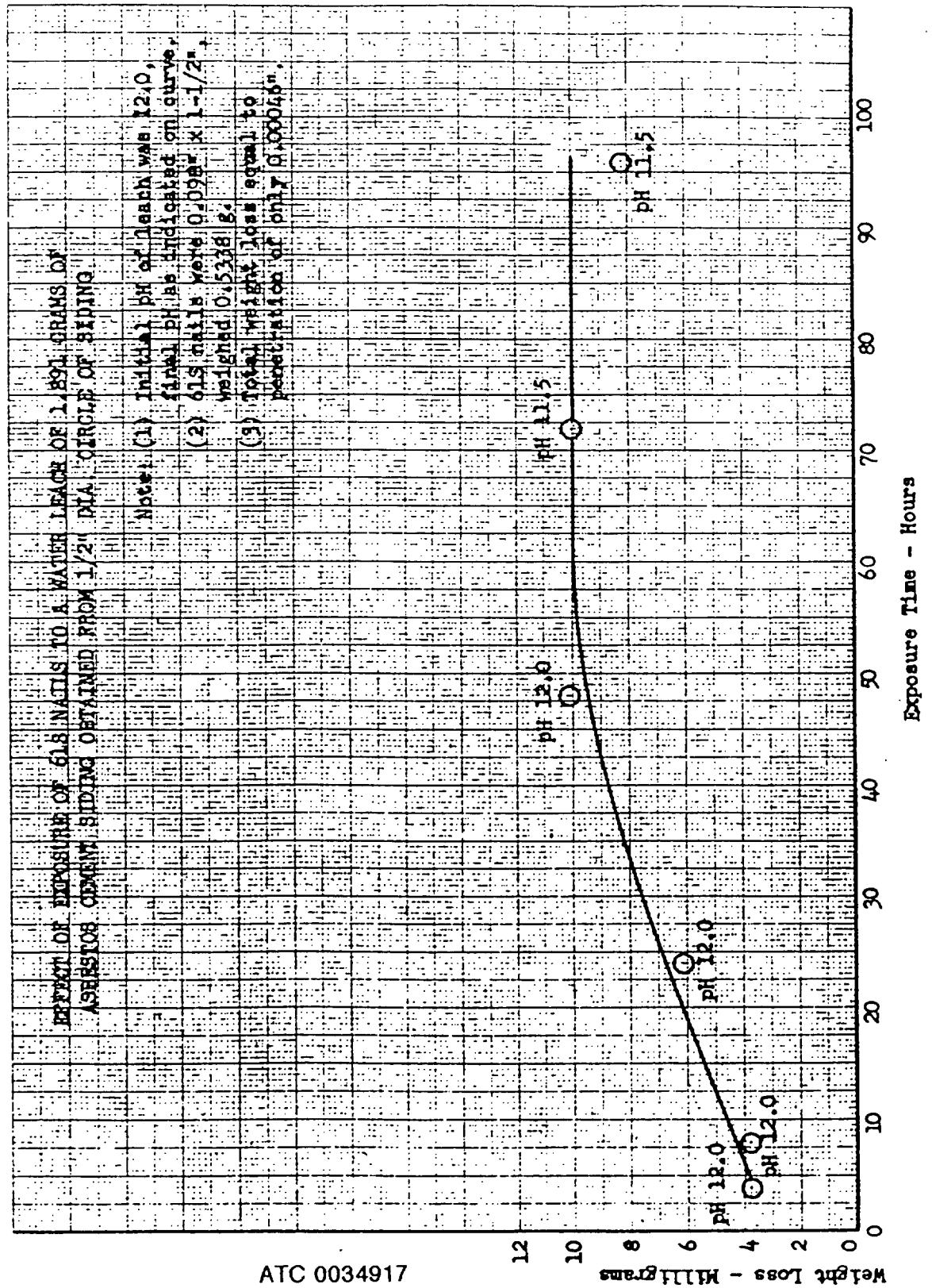


Figure 8

Data shows the relative severity of the test sites at New Kensington and Point Judith. The severity of them is indicated by their effect on mild steel. Note the high inherent resistance to weathering of the aluminum alloys, which include 61S. Also, that the rate of weathering decreases with time. Loss in tensile strength is used as a critical measure of extent of corrosion.

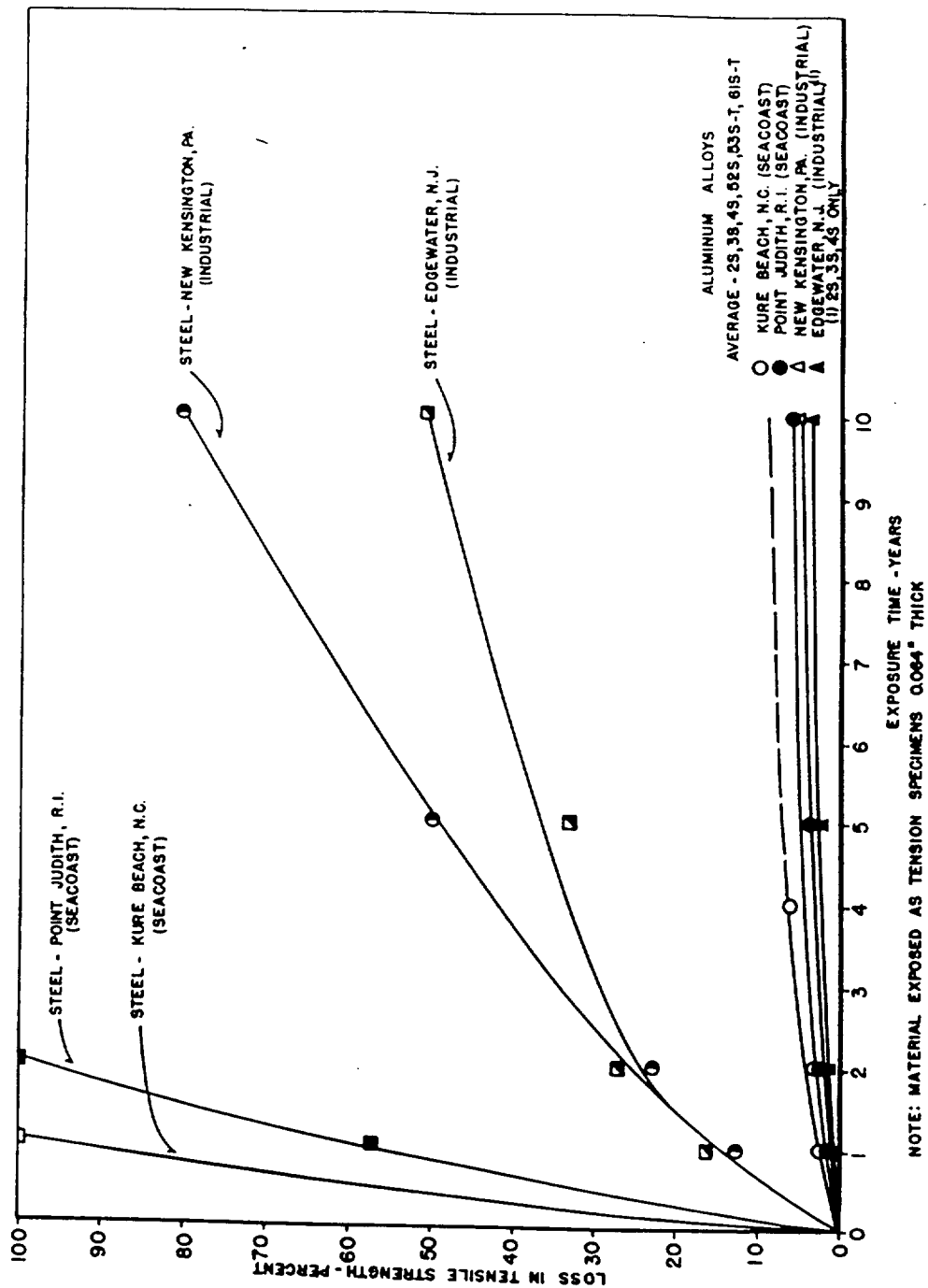


Figure 9

Data obtained from A.S.T.M. tests showing that aluminum alloys (2S-H14 [1/2H] and 3S-H14 [1/2H]) weather at a slow rate in a variety of atmospheres and were equal to copper. (No real differences exist between 61S-T6 and either 2S or 3S in atmospheric exposures.)

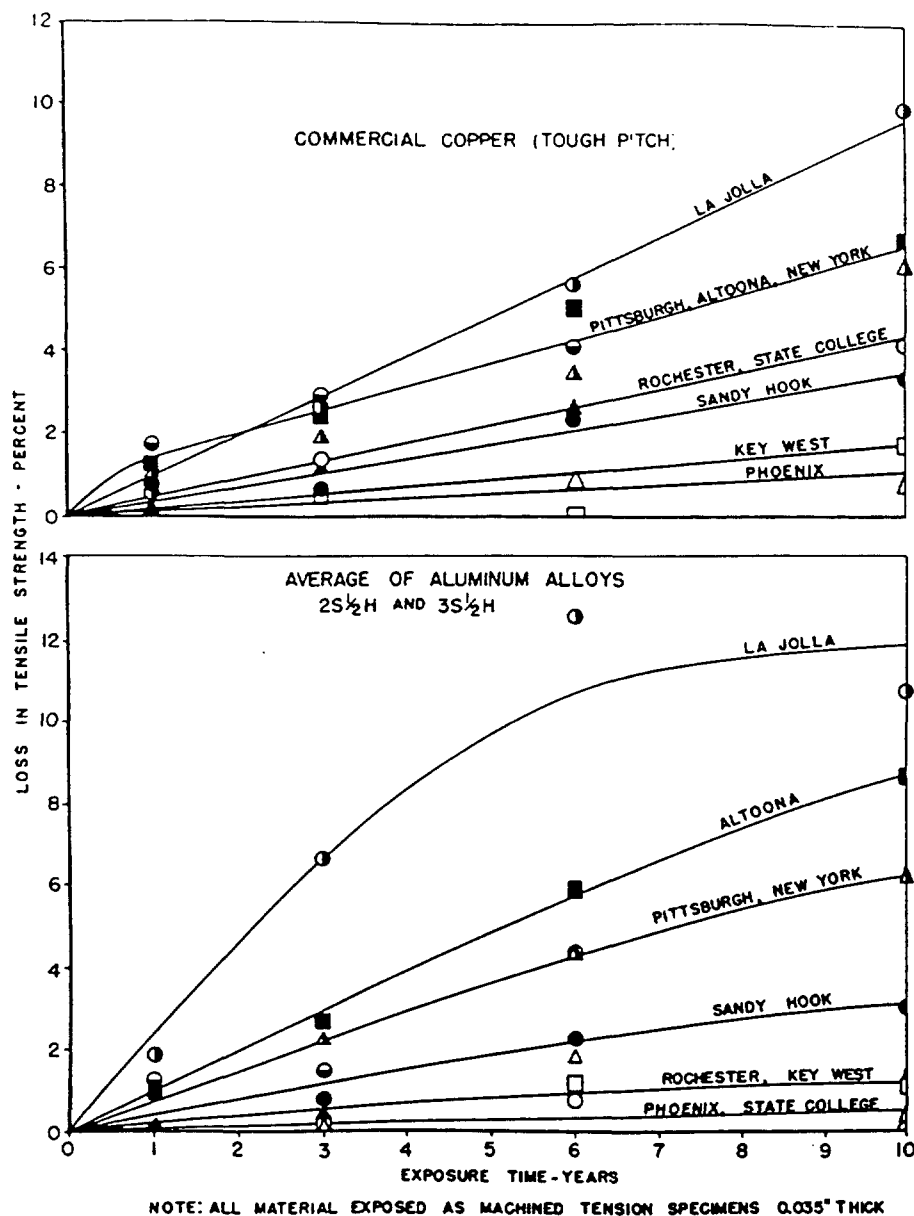
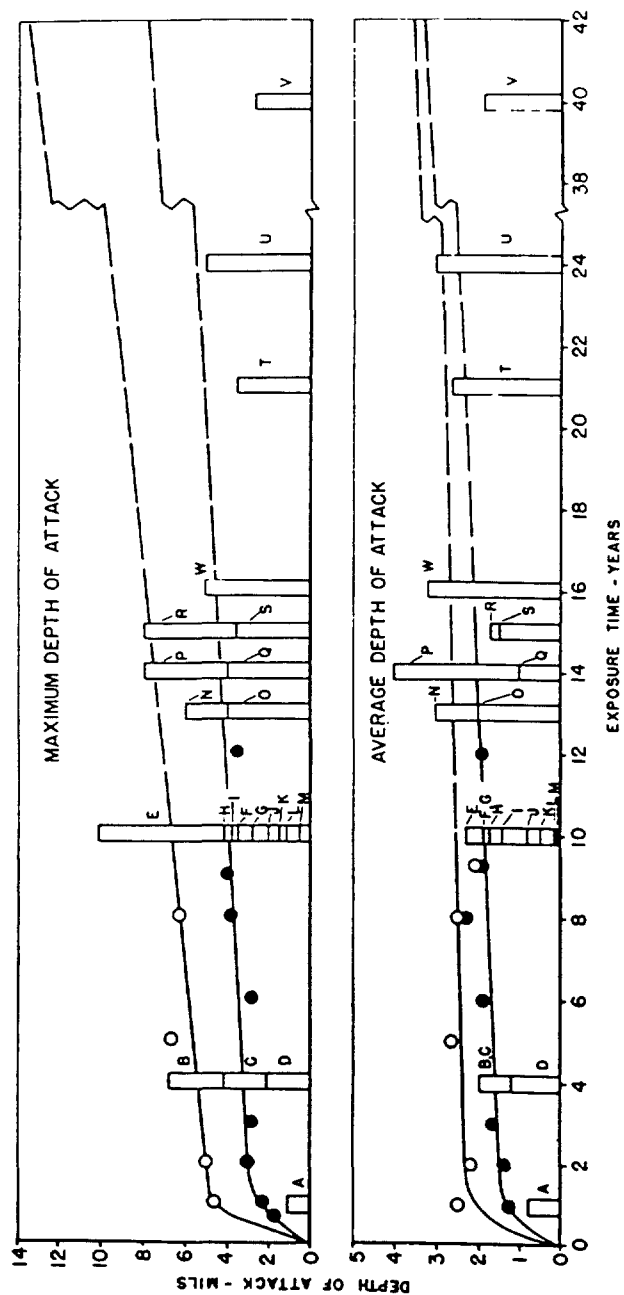


Fig. 9

Figure 10

The curves show the rate of attack of aluminum alloys over a period of 10 years to the seacoast atmosphere at Point Judith, R.I., and over 14 years to the industrial atmosphere at New Kensington, Penna. These curves are extrapolated to a period of 42 years. Note that the attack is shallow and that the rate of weathering of aluminum alloys decreases markedly after about two years; also, that aluminum alloys exposed at many other localities in this country generally were not appreciably affected.



SEACOAST STATIONS	INDUSTRIAL ATMOSPHERE STATIONS	RURAL ATMOSPHERE STATIONS	FROM INSTALLATIONS IN SERVICE
A-2S PITCAIRN ISLAND, BRITISH OCEANA	C-2S, 3S, 4S ST LOUIS, MO.	L-2S, 3S, STATE COLLEGE, PA.	N-3S ROOF - MOENGO, DUTCH GUANA
B-2S, 3S, 4S, KURE BEACH, N.C.	F-2S, 3S, ALTOONA, PA.	M-2S, 3S, PHOENIX, ARIZ.	O-3S ROOF - PITTSBURGH, PA.
D-2S, 3S, 4S, MIAMI, FLA.	G-2S, 3S, 4S, EDGEWATER, N.J.		P-992% AL MOLDING - CINCINNATI, OHIO
E-2S, 3S, LA JOLLA, CALIF.	H-2S, 3S, NEW YORK CITY		Q-3S SIDING - EAST ST LOUIS, MO.
I-2S, 3S, SANDY HOOK, N.J.			R-3S COAL CONVEYER COVER MAHANOY CITY, PA.
J-2S, 3S, KEY WEST, FLA.			S-4S SIDING, CLEVELAND, OHIO
K-2S, 3S, 4S, GEORGETOWN, B.C.			T-3S SHINGLES, NEW KENSINGTON, PA (SIMULATED SERVICE)
W-2S, 3S, 4S, SAN FRANCISCO, CALIF.			U-3S ROOFING - NEW KENSINGTON, PA.
			V-98.4% AL CHURCH ROOF - ROME, ITALY

Figure 10

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ALUMINUM RESEARCH LABORATORIES

NOTED

JUN 27 1942 Page 1

PROGRESS REPORT

E. H. D., JR.

PROBLEM NO. CL-64 - INVESTIGATION OF THE PERMANENT MOLD PROCESS

OPERATOR W. A. Dean DATE 5-20-42

1809452

CONCLUSIONS

SUGGESTIONS

EXPERIMENTS

REMARKS

(Cast Iron and Aluminum Molds Cast to Finished Dimensions)
Asbestos-Cement Mold

Synopsis

Attention has been given to materials for permanent molds. One phase of this problem is to produce cast iron permanent molds quicker and cheaper than the present method. Another is to produce molds from materials other than cast iron. In the present report an attempt was made to cast cast iron mold halves to finished dimensions. Surfaces were smooth and of satisfactory quality for a production mold but the castings were warped and had to be reworked to a greater extent than was desired. A better molding sand, one of which is now under consideration, might eliminate this warping. The mold halves were finished to print dimensions and have been used to produce commercial castings.

The second phase of this problem was investigated by preparing molds from the same pattern equipment as the cast iron mold but of aluminum and of an asbestos cement mixture. Thus materials of higher and of considerably lower thermal conductivity than cast iron were available. It appeared from these preliminary experiments that aluminum alloy molds might be made to final dimensions and used in production provided the same freedom from warping encountered in the cast iron mold, can be achieved. After some preparatory experiments a set of mold halves of an asbestos-cement mixture was made. The dies cracked during these experiments and the mold halves were also warped. After a thermal treatment to expel moisture it appeared that castings of satisfactory external appearance also could be produced from such a mold.

A microscopic examination of castings from the three molds revealed that the one of asbestos-cement produced castings of internal appearance similar to a sand casting. Solidification of castings in this mold was much slower than in the metallic mold. There was no significant difference in micro appearance of the castings produced in the uncoated cast iron and aluminum alloy molds. A light spray of wash on the metallic molds reduced the chill to small but noticeable extent in each case.

These experiments indicate the need for further attention to the problem of casting permanent molds to finished dimensions whether from cast iron, aluminum or other materials.

Introduction

ATC 0034924

The present report is a discussion of a phase of an investigation of materials for permanent molds. The occasion for the particula

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ALUMINUM RESEARCH LABORATORIES

PROGRESS REPORT

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PROBLEM NO. CL-64 - INVESTIGATION OF THE PERMANENT MOLD PROCESSOPERATOR W. A. Dean DATE 5-20-42

CONCLUSIONS	SUGGESTIONS	EXPERIMENTS	REMARKS
experiments described in this report was a request from Mr. G. Kohl of the permanent mold plant that we attempt to cast mold halves to dimensions or at least so close to the final contour of the desired mold that finishing operations would be relatively simple and inexpensive. The particular job was a small bracket for Vought Sikorsky Aircraft Corp., two of which were to be poured from a central gate. Three sets of mold halves were prepared from the pattern equipment, namely, a set of cast iron, one of Al32 alloy and one of a cement-asbestos mixture. A microscopic study has been made of brackets cast in each of the three molds. <u>Experimental</u> Several attempts to make the iron mold halves in green sand some of which had been obtained from the Cleveland Cooperative Stove Co., were not successful. The iron (scrap Meehanite melted in R Plant) washed and cut the green sand. A dry sand mold was used with some success but the core sand mold was not hard enough for the iron. The mold halves were warped slightly and had to be reworked in the machine shop. The Al32 alloy mold halves were cast in green sand and were warped to a greater extent than the iron. Better results would undoubtedly be obtained by casting the aluminum mold halves in dry sand. Al32 was selected because of its low coefficient of expansion and because of its high resistance to abrasion. Although the metal was fluxed with chlorine the gas content of the castings was high. The use of aluminum alloy molds in production would require greater attention than is given to iron molds. For example, while warming up the mold, the temperature must be kept below 950°F to prevent melting. Any wash applied to the surface should be easy to remove since scraping or the use of a wire brush will rapidly impair the sharpness of contour. One of the salt binder type washes soluble in water might be used. An attempt was made to eliminate the warp from the Al32 mold halves by heating them in gas fired furnace under a load of 100 pounds. Some improvement was effected but the warp was not completely eliminated. The Al32 alloy mold was not machine finished to the customer's print and was not used in production. The third mold, one of cement-asbestos was made only after a preliminary investigation which will be described in some detail now. Interest in mold materials of relatively low thermal conductivity has been expressed from time to time by different individuals familiar with the permanent mold process. For example Transite has been suggested as a material which was hard, very smooth and perhaps could be used without the application of a wash. Mr. L. W. Kempf discussed with			

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CONCLUSIONS	SUGGESTIONS	EXPERIMENTS	REMARKS
Mr. R. H. Heilman of the Mellon Institute, the use of molding mixtures similar to Transite. In a letter from Mr. G. H. Wagner to Mr. L. W. Kempf (November 10, 1941 Re: Mixtures for Molding Transite) a mixture was suggested consisting of 15% asbestos fibre and 85% Portland cement by weight. Lumnite cement, because of its higher thermal stability was reported to be preferable to Portland cement. Directions also were given for the preparation of such a mixture. A supply of Lumnite cement was obtained and also three samples of asbestos recommended by Mr. Fleck of Johns-Manville Co. These were identified as follows 4H - Shingle fibre (0.0-5.0-10.0-1.0) - \$73.00/ton 6D - Waste, Stucco or Plaster fibre (0.0-0.0-7.0-9.0)-\$30.00/ton 7D - Refuse or Short fibre (0.0-0.0-5.0-11.0)-\$30.00/ton All prices F.O.B. the mine The designation of the different grades in parenthesis represents the ounces of material from a 16 ounce sample left on three screens and collected in the bottom of the pan. 4H therefore, is coarsest and 7D the finest. The first investigation was carried out in a sub-press used in the Amsler machine which produced a cylindrical specimen of 0.50 square inches in area. A sample of each grade of asbestos was soaked in water over night and at the start of the experiment Lumnite cement was added to each to make a 15% asbestos-85% cement (by weight) mixture. Excess water was decanted, the sub-press cavity filled with the mixture and specimens produced by applying pressures of 1000, 2000, 5000, 10,000 and 20,000 p.s.i. There was little difference in the appearance of specimens made from the different grades of asbestos except that those from 4H were slightly rougher than the others. All were readily scratched with a pen knife. Somewhat harder surfaces would have developed had the specimens been tempered with water during the aging period following their formation. An attempt was made to Brinell the specimens. Many of them cracked but in some instances readings were obtained. Figure 1 shows the specimens, the pressures at which they were formed and the Brinell readings where failure did not occur. The 4H (long fibre) specimen were strongest and as a group the 7D specimens were next. As would be expected, resistance to failure increased with pressure at which the specimens were formed. An attempt was made to determine the best ratio of cement to asbestos. Grade 6D was examined since it offered greater room for improvement than either 4H or 7D. At two pressures of 2000 p.s.i. and 10,000 p.s.i. mixtures containing 10, 15, 25, 35 and 50 per cent asbestos by weight were formed into specimens. These specimens were			

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CONCLUSIONS	SUGGESTIONS	EXPERIMENTS	REMARKS
tempered with water 4 hours after they were formed and received three additional applications of water at hourly intervals. The results, shown in Figure 2 indicate the optimum concentration of asbestos for maximum hardness is in the neighborhood of 15 to 25 per cent. From these experiments it was concluded that desirable conditions for the proposed cement-asbestos mold would be a mixture of 15 to 25 per cent of long fibre asbestos and a high forming pressure (10,000 p.s.i. or greater). A preliminary experiment showed that reduction in volume of a 16-2/3% asbestos-84-1/3% cement mixture at a pressure of 10,000 p.s.i. was approximately 4 to 1. With this information a boiler plate rectangular frame was made. The sides were of 1" plate, the ends of 1/2" stock and the frame was held together with 1-3/8" threaded bolts (drawing CDR-D-10636). The bottom of the box was closed with a hard maple board 1" thick. Pressure in the press was to be transmitted through a built up hard maple block that fit into the boiler plate frame with 1/8" clearance on the sides and 1/4" on the ends. A view of the frame with bottom maple board and maple block are shown in Figure 3. A side view of the equipment is shown in Figure 4. The inside dimensions of the box were roughly 7" deep, 14" long and 4-1/2" wide. Plaster casts were taken from the patterns from which the cast iron and Al32 mold halves already described were made. These plasters were used as patterns to make a pair of dies, also of Al32 alloy, which were to be used subsequently to form the asbestos-cement mixture. The dies were machined to establish a 1/16" clearance when fitted in the box. Figure 5 shows these dies after they were used to make the asbestos-cement mold halves. The gate, both risers and the two bracket castings are apparent. Both dies cracked during the experiment. The 1500 ton press at the forge shop was used for these experiments. The boiler plate form with 1" hard maple bottom was placed on the lower die of the press. The frame was filled to within 2" of the top with a mixture of 16-2/3% asbestos and 83-1/3% Lumnite cement (by weight). Then one of the two Al32 alloy dies was placed on top of the mixture. The built up maple block was placed on top of the aluminum die and pressure applied from the upper die of the press. At first a light pressure was applied to the assembly and the mixture squirted out at the top. It was apparent that the intentional clearance in the equipment were not enough to permit the water to squeeze out. The press die was then very slowly drifted into contact with the assembly and allowed to drift until the top of the boiler plate frame was contacted. The assembly was raised on two blocks and the contents			

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of the box slowly pressed out of the bottom. Light tapping permitted the Al32 alloy die to be drawn from the asbestos-cement mixture. The die had cracked, it was thought, during the initial application of pressure. Both maple blocks were warped somewhat. When the other half of the asbestos-cement mold was made the upper die of the press was drifted into contact with the assembly and this time none of the mixture squirted out. On removing the aluminum die it was found to have cracked also. Both asbestos-cement mold halves were warped because of the cracked dies. Figure 6 shows the asbestos-cement mold halves. These experiments indicated the need for more substantial base and follow-up block than hard maple. Also dies with greater ductility than Al32 alloy are needed. With some experience in seating the dies in the asbestos-cement mixture (a vibrator might be useful) perhaps cracking of the dies and the resulting warping of the mold halves can be prevented. The experiments showed the need for the slowest possible application of pressure to permit the excess water to be squeezed from the mixture. The mold halves were tempered with water, 4, 11 and 24 hours after they were pressed. The halves were dressed with a file to reduce the extent of the warp. The mixture may be filed readily provided no fine particles of steel or iron are encountered. These probably are present as impurities in the cement. To further investigate these mold materials castings of B195 alloy were made in each mold. The pouring temperature was about 1350°F. This alloy is used to produce the Vought Sikorsky bracket commercially. For the first experiment the cast iron and aluminum molds were used without a wash and the asbestos-cement mold had been aged only at room temperature. All three were heated to about 250°F after which a series of about 15 castings were made. The iron and aluminum molds were then sprayed lightly with Silocel and in the meantime the asbestos-cement mold was aged for 3-1/2 hours at 300°F to drive out moisture, which before this treatment, had bubbled into the gate of the casting giving it the appearance of having been made in green sand of too high a moisture content. This thermal treatment effectively stopped the evolution of moisture when castings were poured into the mold subsequently. Castings made in the asbestos-cement mold solidified far slower than those in the metallic molds. As mentioned previously only the cast iron mold was finished to the print for the casting. Both the Al32 and asbestos-cement molds produced castings of considerably thicker section than required. The only sections of comparable thickness in castings made in the three molds were the ribs. These were used for micro examination. Castings selected for examination were those poured after it was thought the mold			

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was at operating temperature. Figures 7 and 8 are microsections of B195 alloy castings made in the asbestos-cement mold before and after the aging treatment at 300°F. There is little difference in the appearance of the micro-constituents of the two castings or in their dendritic patterns. Both are quite coarse and are of similar appearance to a sand casting. Figures 9 and 10 show the microstructures of castings made in the cast iron mold used with and without a coating of mold wash. The constituent size and dendritic patterns are much finer than those produced in the asbestos-cement mold. Of interest is the observation that the light spray of wash decreased the chill, for the structure in Figure 10 is slightly coarser than that in Figure 9. Figures 11 and 12 show the structures of castings made in the Al32 alloy mold. Castings made in the iron and aluminum molds which had not been sprayed with wash were of very similar micro appearance. The effect of the light wash on the aluminum mold reduced chill to a greater extent than a similar wash on the iron mold as may be seen from a comparison of Figures 10 and 12. This difference in the effectiveness of a wash on the iron and aluminum molds should not be regarded as significant in view of the limited information since the wash on the aluminum mold may have been slightly heavier than that applied to the iron mold. It is well known that light layers of wash considerably reduce chill but subsequent applications of the same thickness do not bring about a correspondingly further reduction in chill. <u>Discussion and Conclusions</u> 1. An attempt has been made to cast cast iron mold halves directly from patterns of the finished casting. The mold halves warped and had to be worked in the machine shop to a greater extent than was desired before they could be used to produce commercial castings. It is thought that the dry sand mixture was not proper for this job. Another sand has been recommended by Mr. G. Gardner of the G.T.L. and this will be used when an experiment of this kind is tried again. The surface of the iron mold halves as cast were very smooth and of a quality that could have been used without further finishing had the molds not warped. It is the opinion at this time that molds cast to dimensions can be made successfully if warping can be overcome. 2. A set of mold halves were also made of Al32 alloy from the same equipment. These too warped badly and were never finished for the production foundry. Their surfaces were also very smooth and it is thought that molds of a suitable aluminum alloy might well be used in the foundry particularly for jobs where the number of castings required is limited. Such molds, as is pointed out in the body of this report, would require more than normal care during preheating and cleaning after use to prevent			

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melting in the first case and to preserve their sharpness in the second. Further work on this phase of the permanent mold problem appears warranted. 3. An investigation has been made of a relatively poor heat conducting material, namely an asbestos-cement mixture. To obtain mold halves which would produce castings similar to those made in the cast iron and aluminum molds already described, Al32 alloy die halves, a boiler plate frame and other equipment was prepared to press mold halves from a 16-2/3% asbestos-83-1/3% cement mixture in the 1500 ton press at the Forge Shop. During the experiments the Al32 alloy dies cracked and the mold halves were warped. The mold halves were dressed so that castings could be produced. The surfaces of these mold halves were also smooth. To drive moisture from the mold a treatment at 300°F was found desirable. 4. Solidification of the casting in the asbestos-cement mold was much slower than in the metallic molds. Before the asbestos cement was given a thermal treatment moisture bubbled from the mold^{and} contaminated the casting. A microscopic examination of corresponding sections from castings made in the three molds showed the structure of the ones made in the asbestos-cement mold had the appearance of a sand casting. Those made in the two metallic molds were of fine structure characteristic of the permanent mold process. There appeared to be no difference in the structures of castings made in the uncoated iron and aluminum molds. The effect of a light layer of wash on both of the metallic molds was apparent in the reduction of chill which the wash produced. The wash on the aluminum mold was more effective but that on the aluminum mold may have been slightly thicker than the layer sprayed on the cast iron mold. 5. While these experiments do indicate that molds of simple design can be made from asbestos-cement mixtures the structure of castings made in such a mold would be expected to be more like that of castings made in sand rather than in a permanent mold.			
cc-Mr. E. H. Dix, Jr. Mr. F. Jardine Mr. G. Kohl Mr. T. D. Stay			
ATC 0034930			

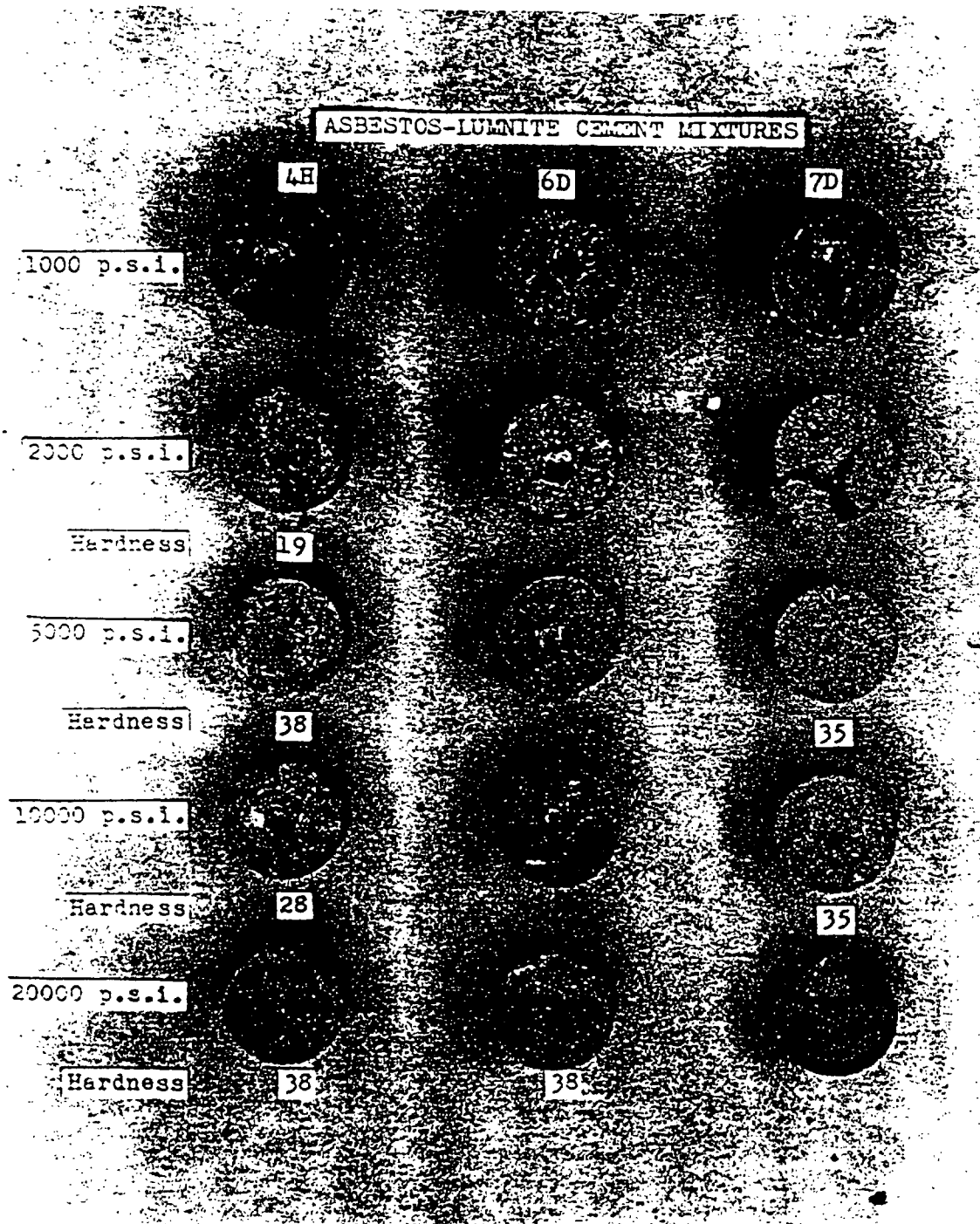


Fig. 1



2.3.1.

Mariners

10000 p.s.i.

Hardness

Fig. 2

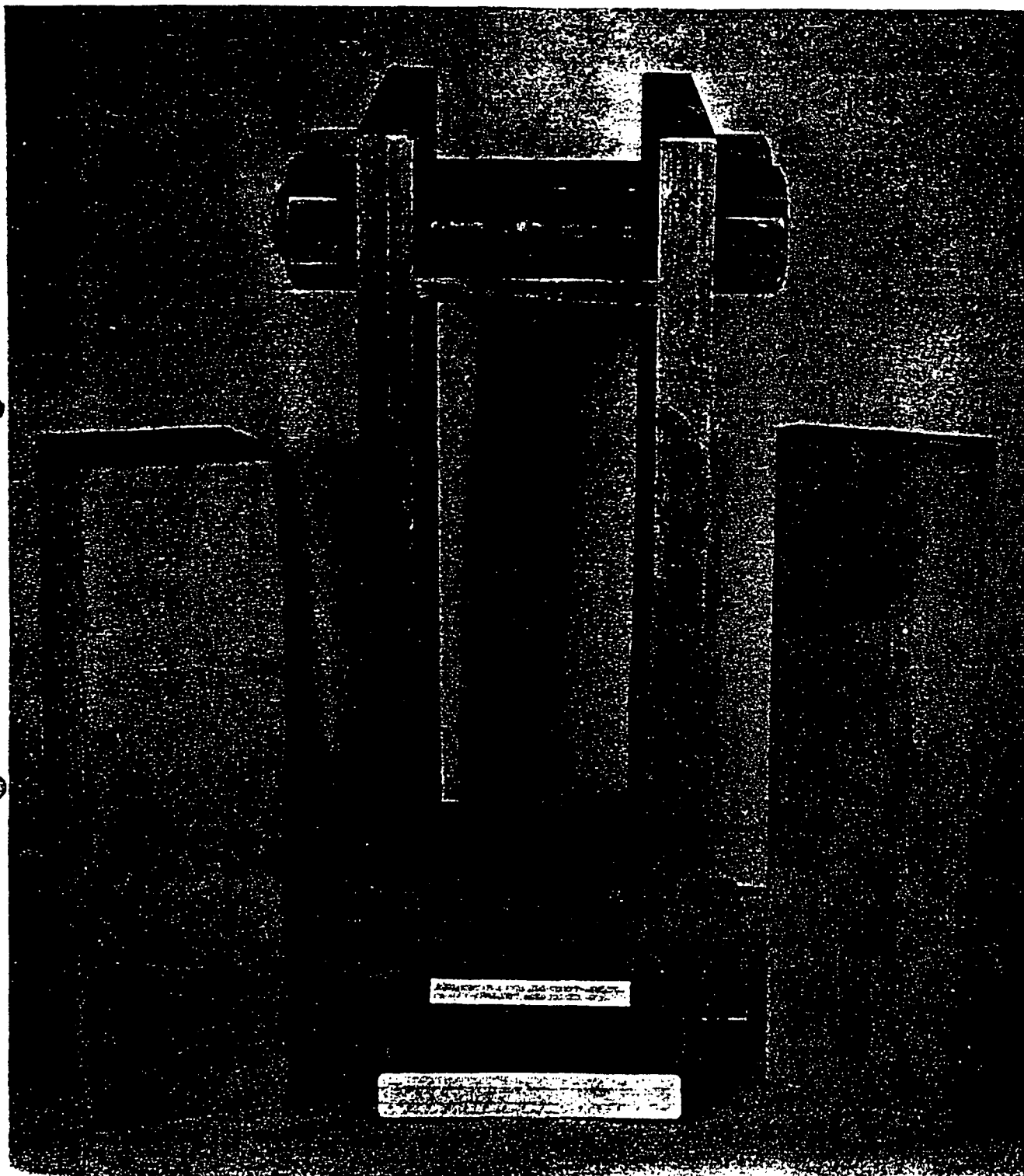


Fig. 3

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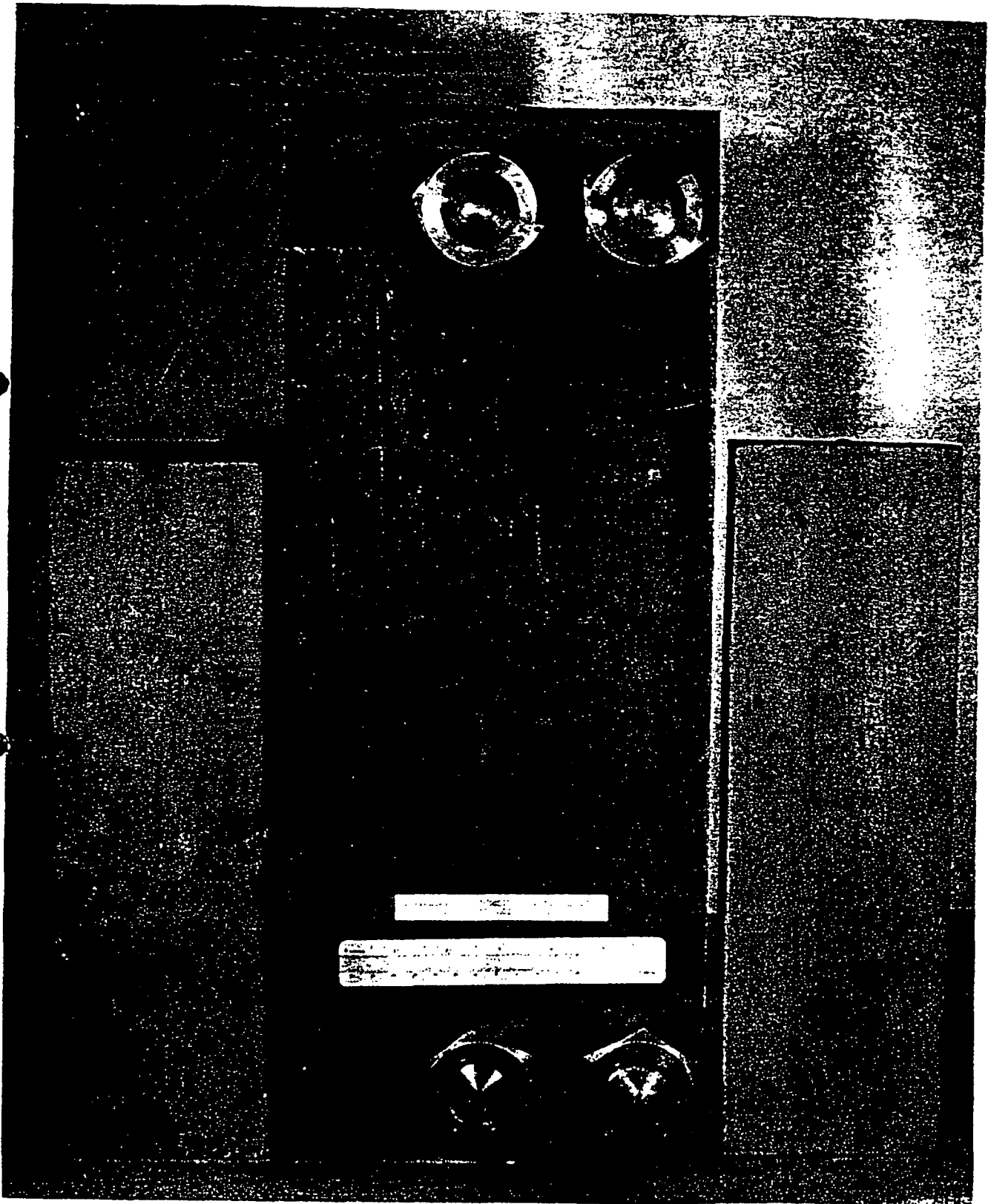


Fig. 1



Fig. 5

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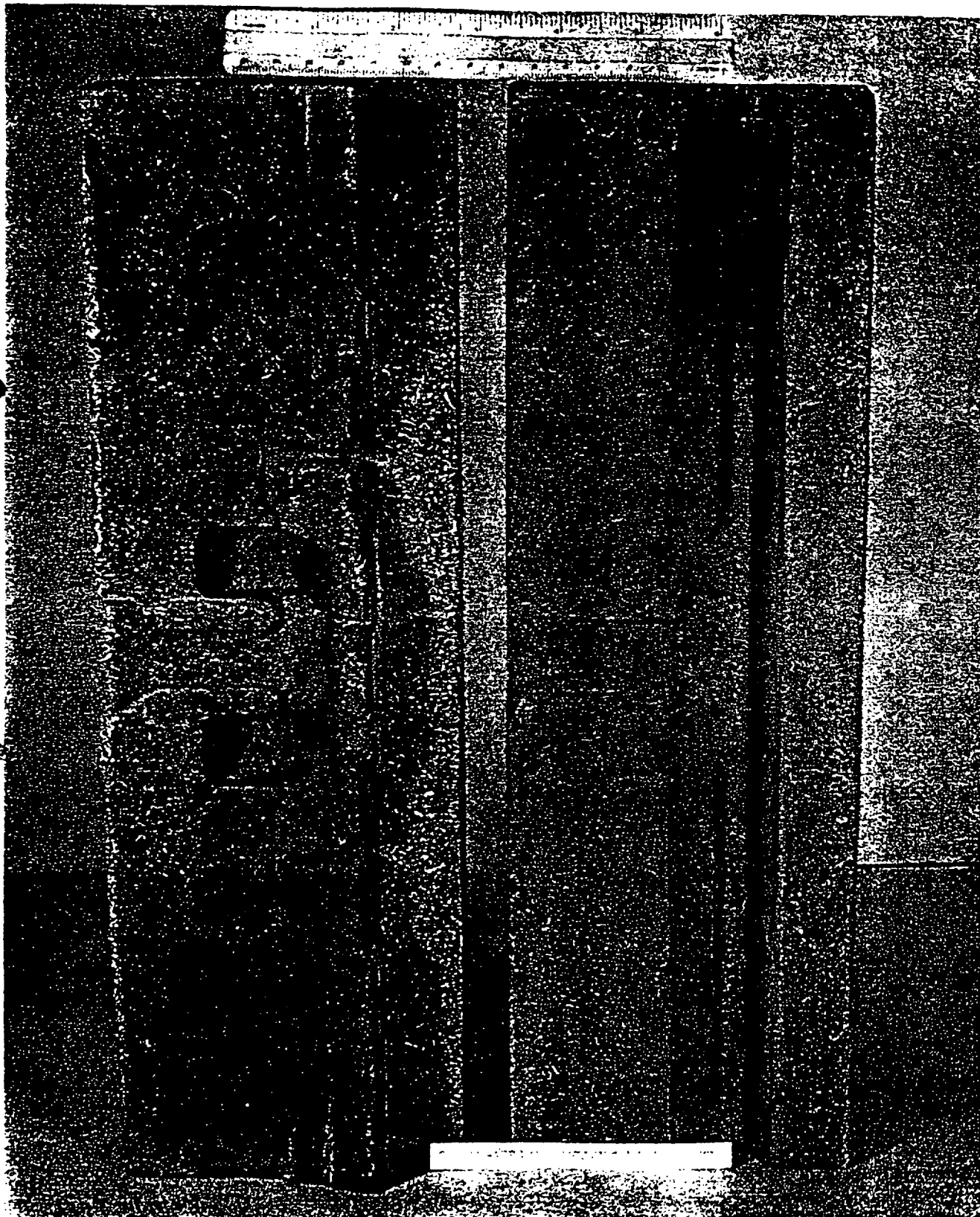


Fig. 6

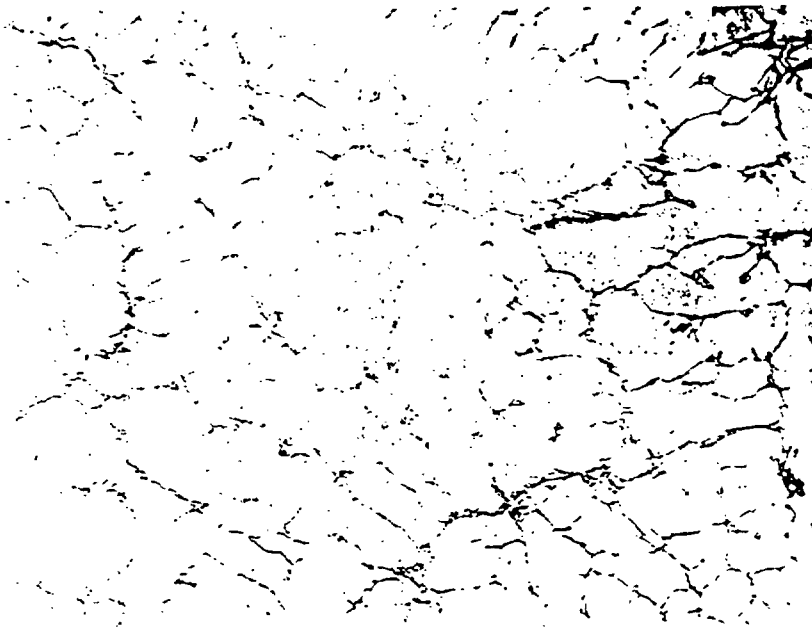


Fig. 7
Asbestos-Cement Mold-Dried at room temperature. Microstructure of 13th casting at 100X etched with 0.5% HF. Negative A3222.

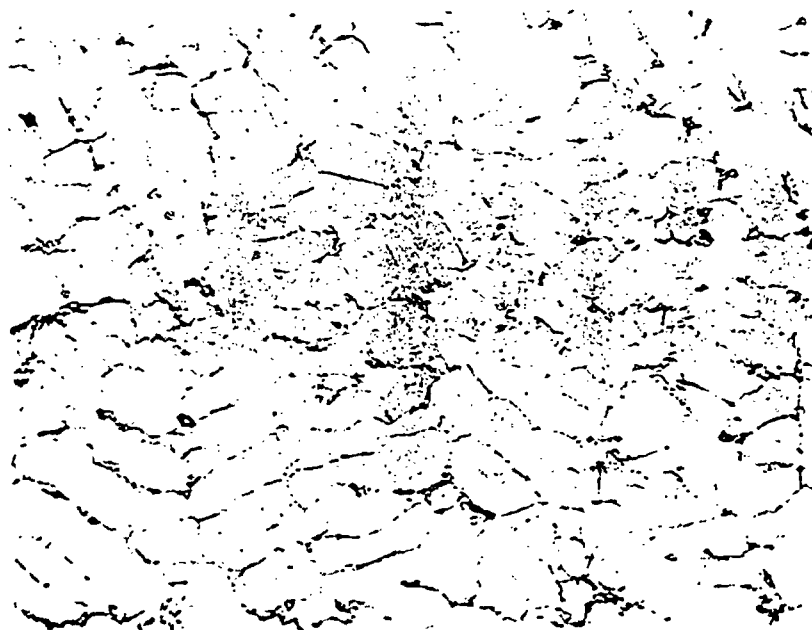


Fig. 8
Asbestos-Cement Mold-heated 3-1/2 hours at 300°F. Microstructure of 11th casting at 100X-etched with 0.5% HF. Negative A3220.

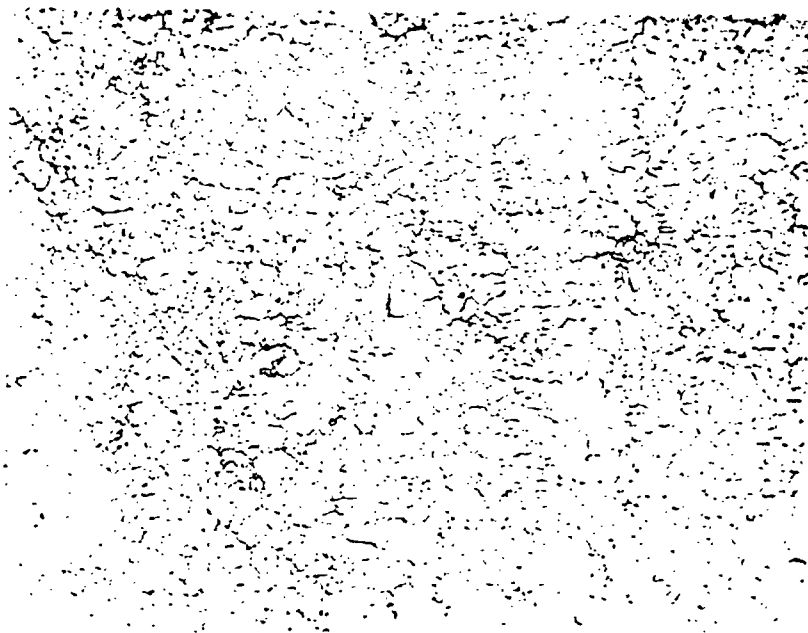


Fig. 9
Cast iron mold - no wash - microstructure of
8th casting at 100X - etched with 0.5% HF
Negative A3218.



Fig. 10
Cast iron mold - light wash - microstructure
of 14th casting at 100X - etched with 0.5% HF
Negative A3241.



Fig. 11

Al32 Alloy Mold - No wash - Microstructure of
11th casting at 100X - etched with 0.5% HF
Negative A3221.



Fig. 12

Al32 Alloy Mold - light wash - Microstructure
of 11th casting at 100X - etched with 0.5% HF
Negative A3219

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PROBLEM NO. CDX-5791, Cl.C.T. 287 DETERMINATION OF SUITABILITY OF GRAPHITE-TREATED ASBESTOS GASKET FOR USE WITH MAGNESIUM ALLOYS OPERATOR M. Daugherty DATE 6-8-40

CONCLUSIONS	SUGGESTIONS	EXPERIMENTS	REMARKS
Ref: (1) Letter R. T. Wood to M. W. Daugherty 3/28/40, Re: Determination of Suitability of Graphite-Treated Asbestos Gasket for Use With Magnesium Alloys. (2) Book 2778, p. 34, 6/5/40 (3) Progress Report 10/23/35, Cl-54, Cl.C.T.109, Determination of the Electrolytic Corrosion Characteristics of Magnesium Alloys in Contact with Ferrous and Non-Ferrous Alloys. (4) Progress Report 5/13/37, CDX-5725, Cl.C.T.178, Evaluation of General Motors Cork and Fabric Insulating Strip and DuPont Insulating Tape. (5) Progress Report 2/10/39, CDX-4989, Cl.C.T. 229, Vallutex Gasket Material for Magnesium Base Alloys. A graphite-treated asbestos gasket was obtained from the Bendix Aviation Corp. to test its corrosive effect on magnesium. The gasket is used as a carburetor throttle body gasket, and is .032" thick. Two similar pieces, each with a single surface area of about one square inch, were cut from the gasket, superimposed, and clamped firmly between two machined pieces of AM265-C alloy. Each piece of the alloy was 3/16" x 3" x 2", and had not been coated. They were cleaned with casing head gasoline before assembly. To avoid metallic contacts the clamping was done by means of rubber bands. The clamped assembly was continuously immersed in a vertical position in tap water at room temperature for 8 weeks, with a daily change of water, since previous similar tests had been made in that manner. After the 8 week exposure the AM265 alloy surfaces which had contacted the gasket material were cleaned with chromic acid and photographed (Figure 1). As shown in Figure 1 each specimen was severely pitted where in contact with the gasket, particularly along the gasket edges. The maximum depth of pitting was approximately .03 inches. This indicated electrolytic action probably between the graphite and magnesium. Comparison of this result with previous results with other gasket materials, as reported under Cl.C.T. 109, 178, and 229, indicates that the graphite-treated asbestos is a decidedly unsuitable gasket material for use in contact with magnesium. <u>SUMMARY</u> Pieces of a graphite-treated asbestos gasket were clamped between machined surfaces of AM265-C alloy, and continuously immersed in tap water for 8 weeks. After exposure the magnesium surfaces which			

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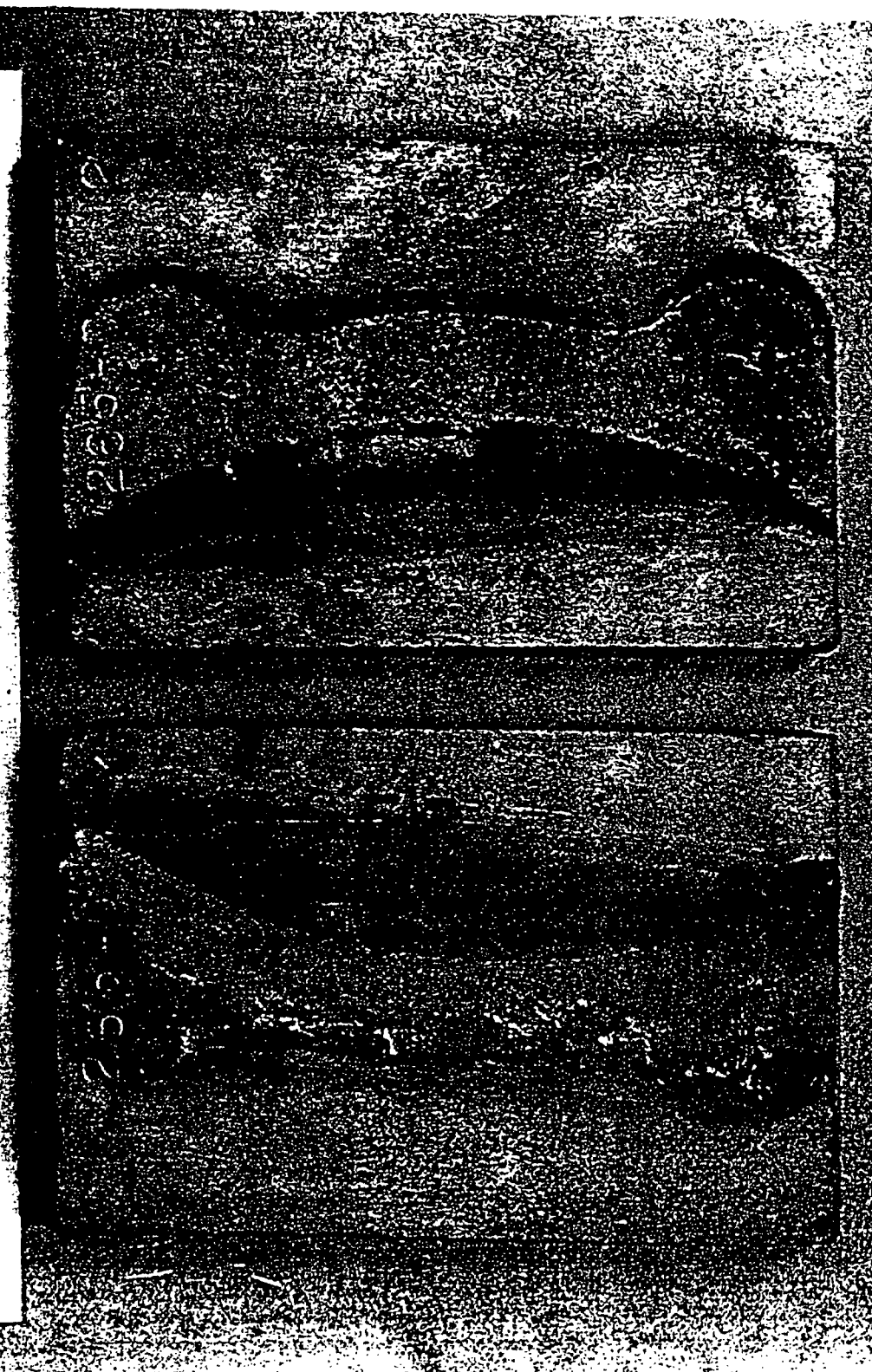
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PROBLEM NO. CDX-5791, C.I.C.T. 287 DETERMINATION OF SUITABILITY OF GRAPHITE-
TREATED ASBESTOS GASKET FOR USE WITH MAGNESIUM ALLOYS OPERATOR M. Daugherty DATE 6-6-40

CONCLUSIONS	SUGGESTIONS	EXPERIMENTS	REMARKS
had contacted the gasket were found severely pitted, particularly along the gasket edges, probably indicating electrolytic action between the graphite and magnesium. It was concluded that the gasket material is unsuitable for use in contact with magnesium.			
cc-Mr. R. T. Wood Mr. H. Menking Mr. W. G. Harvey Mr. R. L. Haskell			
<i>M. W. Daugherty</i> <i>June 6, 1940</i>			

ATC 0034941

Fig. 1, Cl.C.T. 287, 6/5/40
Continuously Immersed 8 Weeks in Tap Water,
In Contact with Graphite-Treated Asbestos



FROM R. A. CHRISTINI
CHEMICAL PRODUCTS & PROCESSES
ALCOA TECHNICAL CENTER - B

TO VITO CEDRO
CHEMICAL PRODUCTS & PROCESSES
ALCOA TECHNICAL CENTER - C

ALCOA PRIVATE INFORMATION

1995-11-07

RE: EVALUATION OF CAMERON-FIAT ITALIAN Mg PROJECT

Letter Report No. 09-95-39

Abstract

The Cameron-Fiat Italian Mg project proposes to use serpentine (asbestos) as part of the Mg ore feed. Mass and energy balances were calculated and used to calculate costs to compare to Northwest Alloys' costs. The major conclusions follow.

1. Cameron's process using serpentine (asbestos) as a partial MgO feed source does not produce a cost advantage even with no costs attributed to the serpentine.
2. Due to CaO requirements in the slag, dolime and/or lime must be fed, thus reducing the serpentine portion to only 10-18% of the total oxide feed.
3. Larger slag volumes are required which will reduce productivity. These slag compositions have little sales value due to the high SiO₂ content.
4. Five raw materials must be fed which requires more capital for the feed system. (NWA has only 3 feed tanks per furnace.)
5. Higher power requirements will increase costs by \$0.01-.03/lb Mg due to increased amounts of raw materials and slightly higher operating temperatures.

Recommendations

1. Continue to follow Cameron's process to assess the technical advantages of a plasma atmospheric process.
2. Do not participate at this point unless Cameron can show a way to improve the economics.

Roy A. Christini

R. A. CHRISTINI

ms/3065

cc: ID-D
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ALCOA

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Introduction

As part of our on-going magnesium processes evaluation study, Cameron's plasma Magnetherm process⁽¹⁾ using asbestos for a feedstock was evaluated. Cameron has proposed that his high temperature atmospheric pressure version of Magnetherm using a plasma arc could utilize asbestos as a feedstock. Asbestos or serpentine is a MgO-SiO_2 ore containing 45-50% MgO on a dried basis. It is actually slightly richer than dolime which is about 41% MgO . However, the high concentrations of SiO_2 (45-50%) are a detriment to a normal Magnetherm operation feeding FeSi since slag chemistry control is critical for good operation.

Cameron has never published detailed material balances for his proposed process. It was decided that mass balances would be calculated for various raw material feeds and several slag compositions: (1) normal NWA composition, (2) a mid-level MgO slag composition from Cameron's patents, and (3) a high level MgO slag composition from Cameron's patents.

Calculations

Simplified raw material compositions were assumed by ignoring low level impurities. Thus serpentine was assumed to be 50% MgO and 50% SiO_2 . Lime and magnesite were assumed to be 100% CaO and 100% MgO , respectively.

Mass balances were calculated for four cases: (1) A standard Magnetherm case was done for comparison, (2) serpentine was used to produce a normal NWA slag since this is a process that definitely would operate, (3) and (4) two optimized balances producing Cameron slags were calculated (see Table I).

All four were normalized to the same crude Mg production of 30,000 lbs which is the current upper limit for one cycle production at NWA.

A refine recovery of 91.7% was assumed for recovery of good Mg (final product) from crude Mg . This is fairly typical for NWA.

Standard NWA raw material costs were assumed for calculating Table II. Since serpentine (asbestos) tailings have zero value, it was assumed the Cameron-Fiat plant would have very little cost involved in that raw material.

Raw material costs include calcination costs and transportation costs for magnesite, lime, dolime, and bauxite.

Raw material costs:

	<u>\$/lb</u>
Serpentine	0
Magnesite	0.10
Lime	0.05
Dolime	0.05
Bauxite	0.10
Al	0.70
Ferrosilicon (75%)	0.40

Energy balances were also calculated based on the mass balances of Table I. Dolime was assumed to enter the furnaces hot at 1000°C. All other raw materials are at room temperature. Both the Magnetherm and AN-6 case (Magnetherm slag using serpentine) were assumed to operate at 1550°C. From Cameron's patent, the ACL-4 mid-level MgO slag (13% MgO) operates at 1700°C. The ACH-8 high level MgO slag (25% MgO) operates at 1900°C.

Results

Various combinations of raw materials were tried for each final slag composition. Due to the high concentrations of CaO needed in the slag, either dolime or lime must be added to the mass balance in large quantities. Since serpentine brings significant quantities of nonreactive SiO₂ that hinders the FeSi reaction, large quantities of serpentine cannot be used. Thus, serpentine only amounted to 10-18% of the total oxide feed.

The second significant aspect of the mass balances is the slag weight. The slag is 26-40% larger for the three serpentine cases compared to Magnetherm. Since slag volume in the reduction furnace can be a capacity constraint, these large slag volumes could reduce productivity proportionally. Since none of these slags has a sales value, the larger volumes are just more material that needs to be landfilled.

Finally, the three new mass balances all require 5 different feed materials to balance the slag composition. NWA has just 3 feed tanks per furnace and feeds 4 materials by preblending two of them. Five materials would either require even more blending (with potential inaccurate feed rates) or more feed tanks which means higher capital.

Note that none of the mass balances use bauxite instead of Al as the Al₂O₃ source for the slag. Several mass balances were calculated using bauxite but the slag volume increase was substantial and the FeSi requirement was very large. The costs for all the bauxite mass balances were over \$1.00/lb Mg and were not included here.

The costs at the bottom of Table II show that both AN-6 and ACL-4 are comparable to Magnetherm. However, as mentioned above, all three serpentine cases have larger slag

weights and thus lower furnace throughput. This lower productivity will lead to higher costs due to poorer asset utilization and the distribution of fixed costs over fewer pounds.

Energy usage was examined for two reasons: (1) the higher slag weights means more raw materials have to be heated to high temperature and (2) both of the Cameron processes run at higher temperatures: ACL-4 at 1700°C and ACH-8 at 1950°C. The energy increases are significant from 2.94 kwh/lb Mg for Magnetherm up to 4.24 kwh/lb Mg for ACH-8. At NWA's current power rate of \$0.026/kwh, the incremental power cost is \$0.01 to \$0.03/lb Mg. The power cost will not be the deciding factor but it is important to consider.

Discussion

The one arena in which the Cameron-Fiat process is important occurs if the Italian government is willing to subsidize the process to use it to dispose of asbestos tailings or to provide jobs. In a recent conversation, Cameron said the Italian government would provide half the capital to build the plant to provide jobs since the closing of the asbestos mine had hurt that area.

Reference

1. A. M. Cameron, "Magnesium Production," U.S. Patent 5,090,996, 1992 February 25.

Table I. Mass Balances

Serpentine (Asbestos) Feed Material

<u>In</u>	<u>Magnetherm</u>		<u>AN-6</u>		<u>ACL-4</u>		<u>ACH-8</u>	
			<u>NWA Slag</u>		<u>Cameron Slag</u>		<u>Cameron Slag</u>	
					<u>Mid MgO</u>		<u>High MgO</u>	
Serpentine - MgO	--		15.4		15.7		6.8	
SiO ₂	--		15.4		15.7		6.8	
Magnesite - MgO	10.9		--		--		36.7	
Lime - CaO	--		30.8		7.8		--	
Dolime - CaO	62.8		58.5		75.4		56.2	
MgO	44.4		42.3		54.6		40.7	
Al	6.8		9.8		7.5		10.1	
Si	13.4		10.9		12.9		10.7	
Fe	<u>4.4</u>		<u>3.7</u>		<u>4.2</u>		<u>4.0</u>	
	142.7		186.8		193.8		172.0	
<u>Out</u>								
Slag - CaO	62.8	58%	89.2	58.7%	83.2	52.5%	56.2	41.0%
- MgO	5.4	5%	7.7	5.1%	20.4	12.9%	34.2	25.0%
- Al ₂ O ₃	13.0	12%	18.8	12.4%	14.1	8.9%	19.2	14.0%
- SiO ₂	<u>27.1</u>	25%	<u>36.3</u>	23.9%	<u>40.7</u>	25.7%	<u>27.4</u>	20.0%
	108.3		152.0		158.4		137.0	
Crude Mg	30.0	--	30.0	--	30.0	--	30.0	--
Good Mg	--	27.5	--	27.5	--	27.5	--	27.5
Resid Si	1.3		1.1		1.2		1.1	
Fe	<u>4.4</u>		<u>3.7</u>		<u>4.2</u>		<u>4.0</u>	
	144.0		186.8		193.8		172.1	

Table II. Costs

<u>Raw Material</u>	<u>Magnetherm</u>	<u>AN-6</u> NWA Slag <u>Composition</u>		<u>ACL-4</u> Cameron Slag <u>Mid MgO</u>		<u>ACH-8</u> Cameron Slab <u>High MgO</u>		
Serpentine	--	30.8		31.4		13.7		
Magnesite	10.9	--		--		36.7		
Lime	--	30.8		7.8		--		
Dolime	108.0	100.8		130.0		96.8		
Bauxite	--	--		--		--		
Al	6.8	9.8		7.5		10.1		
Fe Si	17.9	14.6		17.1		14.6		
Slag	108.0	152.0		158.0		137.0		
Mg	27.5	27.5		27.5		27.5		
Residual	5.7	4.8		5.5		5.1		
	lb/lb	\$/lb	lb/lb	\$/lb	lb/lb	\$/lb	lb/lb	\$/lb
Serpentine/Mg	--	--	1.12	0	1.14	0	.50	0
Magnesite/Mg	.40	.04	--	--	--	--	1.33	.13
Lime/Mg	--	--	1.12	.05	.28	.01	--	--
Dolime/Mg	3.93	.20	3.67	.18	4.73	.24	3.52	.18
Bauxite/Mg	--	--	--	--	--	--	--	--
Al/Mg	.25	.18	.36	.25	.27	.19	.37	.26
FeSi/Mg	.65	.26	.53	.21	.62	.25	.53	.21
Total		.68		.69		.69		.78

Table III. Energy Balances

	<u>Magnetherm</u>		<u>AN-6</u>		<u>ACL-4</u>		<u>ACH-8</u>	
	<u>lbs</u>	<u>kwh</u>	<u>lbs</u>	<u>kwh</u>	<u>lbs</u>	<u>kwh</u>	<u>lbs</u>	<u>kwh</u>
Serpentine MgO	--	--	15.4	7.48	15.7	8.37	6.8	4.15
SiO ₂	--	--	15.4	3.62	15.7	4.05	6.8	2.01
Magnesite	10.9	5.29	--	--	--	--	36.7	22.44
Lime	--	--	30.8	11.15	7.8	3.09	--	--
Dolime CaO	62.8	15.64	58.5	14.57	75.4	20.59	56.2	17.60
MgO	44.4	14.96	42.3	14.26	54.6	20.18	40.7	17.26
Al	6.8	1.82	9.8	2.62	7.5	2.19	10.1	3.40
Si	13.4	5.45	10.9	4.44	12.9	5.76	10.7	5.47
Fe	4.4	1.66	3.7	.62	4.2	.77	4.0	.84
Al reaction		8.17		11.76		9.87		15.25
Si reaction (.9)		<u>27.95</u>		<u>22.73</u>		<u>29.50</u>		<u>28.07</u>
		80.93		93.25		104.37		116.49
Good Mg	27.5		27.5		27.5		27.5	
kwh/lb Mg		2.94		3.39		3.79		4.24

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PROGRESS REPORT

SPECIFIC METHODS OF REDUCING THE RISER VOLUME OF ALUMINUM
AND MAGNESIUM CASTINGS

TITLE

OPERATOR E. E. Stonebrook PROBLEM NO. CL-291 DATE 6-9-49



1809449

SEVENTH REPORT: RISER REDUCTION ON NADLER
FOUNDRY AND MACHINE CO. CASTING NO. M-6-A
BY ASBESTOS-SILOCEL TYPE INSULATION

JUL 15 1949

SYNOPSIS

Previous riser reduction tests (1,2,3,6) had established the effectiveness of Hi-Temp, an asbestos-silocel type material, when used as insulation around blind risers. This study has now been extended to include riser reduction tests on another production casting with Hi-Temp insulation.

It was found that the weight of the two risers used on Nadler Foundry and Machine Co., part No. M-6-A, could be reduced from 10.0 to 4.2 pounds, a reduction of 58%, without any sacrifice in casting quality. A cost analysis, however, indicated that it would not be economical to use this method of riser reduction on a casting having such relatively small risers, but that insulation of considerably larger risers (3) should offer some cost advantage. Another application for such riser insulation might be for the production of castings where the problem is more one of providing adequate feeding rather than riser reduction.

REFERENCES

- (1) Progress Report by W. E. Sicha and E. E. Stonebrook, CL-291, 9-12-45, First Report, "Riser Reduction by Heating or Insulation".
- (2) Progress Report by E. E. Stonebrook and W. E. Sicha, CL-291, 3-15-46, Second Report, "Riser Reduction of Wedge Castings by Heating or Insulation".
- (3) Progress Report by E. E. Stonebrook and W. E. Sicha, CL-291, 5-13-46, Third Report, "Riser Reduction by Insulation as Applied to Al32 Alloy Production Castings".

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TITLE

OPERATOR E. E. Stonebrook PROBLEM NO. CL-291 DATE 6-9-49

- (4) Progress Report by E. E. Stonebrook and W. E. Sicha, CL-291, 7-29-46, Fourth Report, "Riser Reduction Methods Applied to Large 195 Alloy Step Castings".
- (5) Progress Report by E. E. Stonebrook and W. E. Sicha, CL-291, 8-29-46, Fifth Report, "Breakdown Tests as a Gauge of the Quality of 195-T4 Alloy Castings Produced with Insulated Risers".
- (6) Progress Report by E. E. Stonebrook and W. E. Sicha, CL-291, 8-7-47, Sixth Report, "Riser Reduction on Wedge Castings by Heating or Insulation".
- (7) Notebook #5248, pages 1, 2 and 3.

OBJECT

It was desired to make riser reduction tests on a production casting using asbestos-silocel type insulators.

PROCEDURE AND RESULTS

Pattern equipment for two castings was secured from the Cleveland Sand Foundry for riser reduction tests. Both were Nadler parts. Part No. 15-C-2 is a cover plate casting. It was deemed unsuitable, however, for riser reduction tests using asbestos-silocel type insulated risers because of the irregular shape of certain portions of the casting adjoining the risers. These relatively thick-walled insulators are not readily adaptable to use around top risers unless the casting surfaces upon which the insulators rest are flat. Furthermore, it is often undesirable to have the bottom of the insulators in contact with a casting because the rate of solidification of the portions of the casting thus contacted will be reduced considerably.

Three views of the other Nadler casting, designated as No. M-6-A, are shown in Figure 31. Form risers are located on two sides of the casting and it is poured two-up as indicated in the photographs. The small letter

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markings on the Nadler casting photographs indicate the type and location of chills originally used in producing this part. It was found later that the casting could be produced without any chills, and their use was discontinued. One of these castings was poured at 1340°F with 356 alloy from heat C5007 following the practices used by the Cleveland Sand Foundry. The different parts of the casting had the following weights:

Gating	5.5 lbs.
Riser A	5.7 "
Riser B	4.3 "
Trimmed casting	17.1 "
Poured casting	<u>32.6</u> "

Radiographs showed that the casting contained general fine to medium gas porosity and areas of small to medium motting. A few small gas holes were noted in one area, but the casting was considered to be of acceptable quality.

A blind riser form was made for the top side riser (B) by molding loose asbestos-silocal cement (Hi-Temp) and baking to drive off the added water. The approximate size of the original open riser was 2-7/8 in. x 2-1/8 in. at the base and 3-7/8 in. x 2-1/2 in. at the top with a total height of 4-1/2 in. above the riser pad. The blind riser had the same dimensions at the base but was only 3 in. high with suitable draft. A squeezer was used to ram the asbestos-silocal cement in an aluminum tube. Some difficulty was experienced in removing the formed insulator from the tube and it was necessary to use the squeezer in conjunction with a ram in ejecting the insulator. A rectangular-shaped riser form was used for riser A and the resulting riser cavity was lined on four sides with 1 in. thick blocks of Hi-Temp. The top of the riser was left open. These insulated risers are shown in the A sketches of Figure 32.

Another casting was poured from heat C5078 using the insulators just described. The weights of the different parts of the casting were as follows:

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Gating	5.5 lbs.
Riser A	2.1 "
Riser B	2.4 "
Trimmed casting	<u>16.9</u> "
Poured casting	26.9 "

Radiographs showed a localized area of small to medium shrinkage porosity on one side of the bottom wall under riser A, and the casting was considered to be of unsatisfactory quality.

The insulated B riser used in this test appeared to adequately feed the one side of the casting and was retained in subsequent tests. The next test (heat C5121) involved the use of two insulated blind risers located at the parting line of the mold in place of the single A riser used in the previous test. Each of the two insulators used for the A risers was the same size as the insulator used for riser B. Figure 32B shows sketches of the locations of the three risers used.

Weights of the different parts of the casting from heat C5121 were as follows:

Gating	5.6 lbs.
Two A risers	4.4 "
Riser B	2.4 "
Trimmed casting	<u>17.1</u> "
Poured casting	29.5 "

Radiographs showed that this casting was of satisfactory quality but the weight saving was only 3.1 lbs. which would not begin to offset the cost of insulating the risers.

Accordingly, one additional casting was poured from heat C5241 using only one insulated A riser at the parting line instead of the two used in the previous test. Radiographs indicated that the part was of acceptable quality. The weights obtained with this practice were as follows:

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Gating	4.9 lbs.
Riser A	2.0 "
Riser B	2.2 "
Trimmed casting	<u>17.2</u> "
Poured casting	26.3 "

This is a total weight saving of 6.3 lbs. compared with the casting produced with standard, uninsulated risers. It was thought that this casting represented nearly the maximum riser reduction possible with insulation, and no further tests were made.

DISCUSSION

If the cost of melting and pouring one pound of aluminum is 3 cents, then the total saving resulting from the riser reduction achieved with the casting poured from heat C5241 would be about 19 cents. There would also be some saving in trimming cost since the large side riser was entirely eliminated from the experimental casting. These savings should be balanced against cost of insulators and a possible increase in molding time resulting from placing the insulators and favoring them during the ramming operation. Although accurate values for insulator costs could not be obtained from the small scale method of fabrication employed, it is doubtful whether the saving with this casting would justify the cost even if the insulators could be used several times.

In the case of castings having relatively large risers, (3) riser insulation should offer some cost advantage, particularly if the insulators can be salvaged and reused several times. There are also jobs where only a few castings are to be produced and the cost of scrapping one or more castings would far outweigh the expense of providing insulators. If added assurance is desired that the castings will be adequately fed, then insulated risers would offer one solution to the problem.

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SPECIFIC METHODS OF REDUCING THE RISER VOLUME OF ALUMINUM AND MAGNESIUM CASTINGS

TITLE _____

OPERATOR E. E. Stonebrook PROBLEM NO. CL-291 DATE 6-9-49

CONCLUSIONS

- (1) Use of asbestos-silocel type insulated risers permitted the poured weight of Nadler casting No. M-6-A to be reduced from 32.6 to 26.3 lbs. without any sacrifice in casting quality.
- (2) The cost of using insulated risers on this particular casting would be greater than the savings effected by the 6.3-lb. reduction in poured weight.
- (3) From an economic standpoint, use of asbestos-silocel insulators should be restricted to castings having relatively large, bulky risers or to short runs of relatively expensive castings for which satisfactory gating practices have not yet been developed.

E. E. Stonebrook 6-17-49

cc: Mr. M. W. Daugherty, ARL, Cleveland
Mr. E. H. Dix, Jr., ARL, New Kensington
Mr. W. E. Sicha, ARL, Cleveland
Mr. H. J. Rowe, Pittsburgh
Mr. E. V. Blackmun, Cleveland

FIGURE 31

Three Views of Nadler Casting No. M-6-A
Which Was Used for Riser Reduction Tests
with Hi-Temp Insulation.

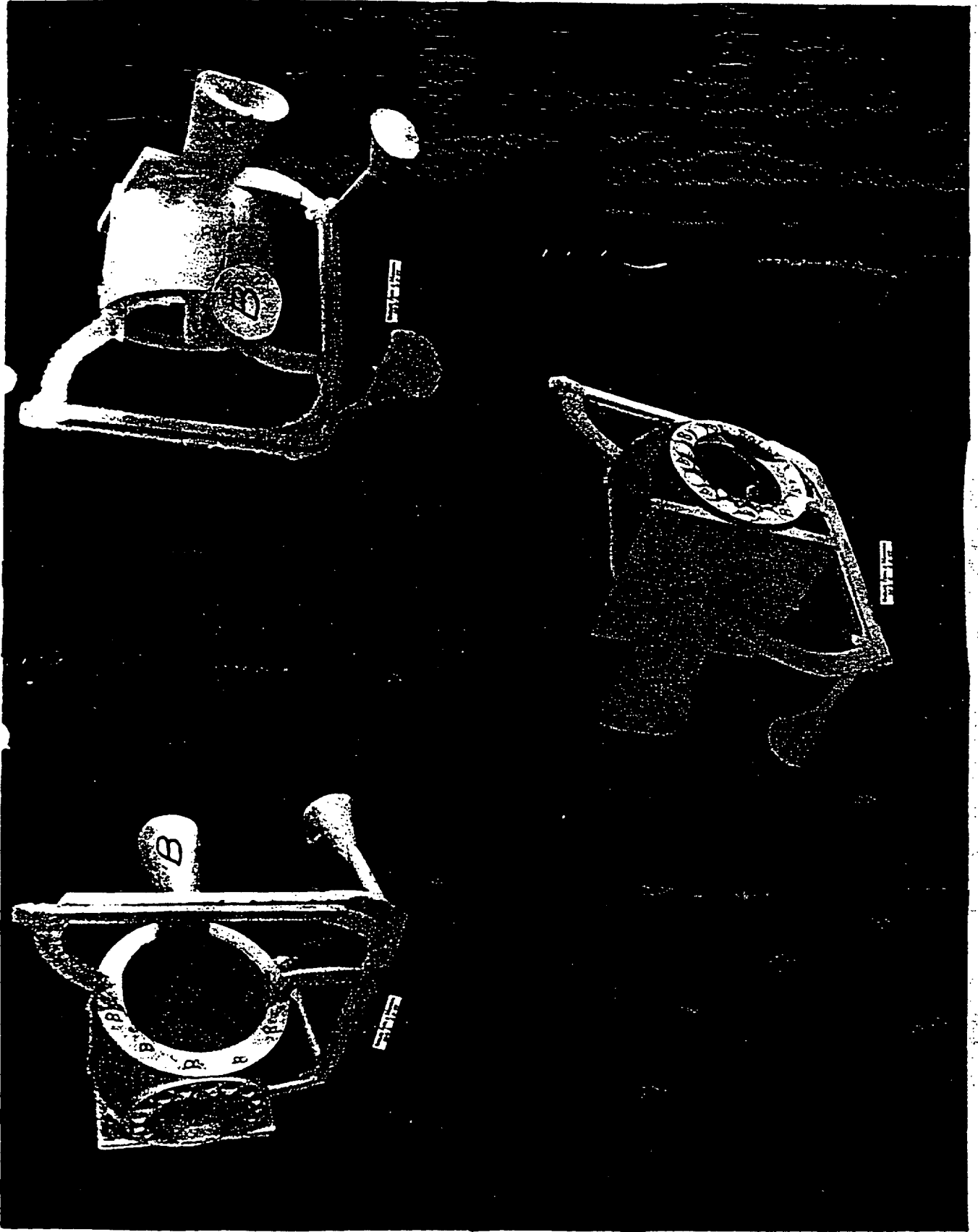
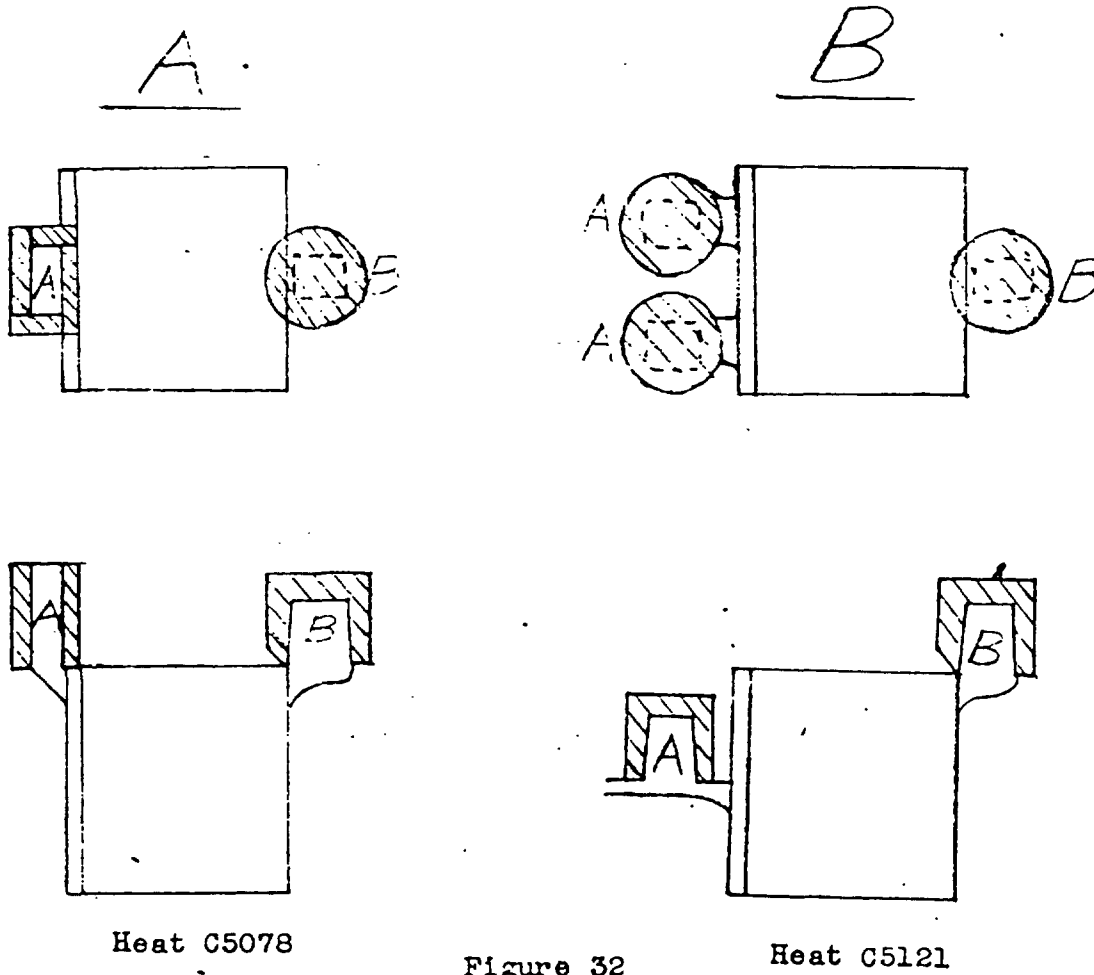


FIGURE 31



Heat C5078

Figure 32

Heat C5121

Sketches showing types of insulated risers used in producing two Nadler M-6-A castings.

163713

0053420

FROM R. A. MARRA
CHEMICAL SYSTEMS DIVISION
ALCOA TECHNICAL CENTER-E

TO MEMORANDUM

1992 March 12

RE: LETTER REPORT 6.92.28: CHARACTERIZATION OF PABCO METALTEMP

Background

Calcium silicate board materials are used as both headers and feedboards at Warrick Operations for the Horizontal Direct Chill (HDC) casting of aluminum ingots. The HDC process was originally designed to use Header Grade Molten Metal Marinite (HGMMM) for the header and feedboard. HGMMM is an asbestos fiber-reinforced calcium silicate board material. Due to the health hazards associated with this asbestos containing material, replacement materials for both the header and feedboard are being evaluated. An acceptable header replacement should have the characteristics shown in Table I. The feedboard should have similar characteristics, although the resistance in molten metal attack is less important. Since a large material area of the feedboard is exposed to molten metal contact, the moisture and carbonate content is extremely important.

Table 1
Desired Characteristics for HDS Header Material

Characteristic	Importance
Molten Metal Resistance (Low Wettability)	- Prevent metal/header reactions which degrade the header surface and result in ingot defects.
Low Thermal Conductivity	- Prevent molten metal solidification at the header surface.
High Compressive Strength	- Prevent crushing of header in the mold package.
Low Moisture/Carbonate Content	- Prevent risk of explosion when contacted with molten metal and avoid outgassing which degrades ingot quality
High Abrasion Resistance	- Prevent degradation of header surface during casting.
Low Porosity/Permeability	- Minimize lubricant absorption.
Adequate Strength (MOR)	- Allow machining and handling of header.
Low Thermal Expansion (Good Thermal Stability)	- Minimize change in header/oil ring overhang and stress build-up in mold package.

PABCO Metaltemp and BNZ Materials Metalform calcium silicate boards have been evaluated for feedboard and header replacement. These materials are boards produced by pressing slurries of lime (CaO), silica (SiO₂; typically added as a colloidal material), wollastonite (CaSiO₃), and alkali resistant glass fibers. The pressed boards are autoclaved

1809465

ATC 0034959



MEMORANDUM

1992 March 12

Page 3

marinite headers for casting 5xxx series ingot. There have been severe quality problems with the BNZ material in terms of outgassing (bubbling) of feedboards. This not only results in poor ingot quality but is a serious safety issue during casting. Feedboard breakage during or after machining has also been a major problem. The qualification of the Pabco Metaltemp material for feedboards (for 5xxx and 3xxx series alloys) and headers (for 3xxx series alloys) should help eliminate these quality issues as well as having a favorable cost impact since material is currently available in Warrick's inventory.

Characterization Techniques

DENSITY: The density was calculated from dimensional and weight measurements on 1"x1"x6" bars machined from the boards. The bars were dried at 230°F for a minimum of four hours prior to measurement.

HARDNESS: The material hardness was measured using a Durometer conforming to the ASTM 2240-75 test method. The hardness was measured on both the die side and the screen side of the pressed boards.

MOLTEN METAL RESISTANCE: The molten metal resistance was evaluated by both metal immersion testing and direct examination of used header surfaces. In the immersion testing, 3"x3"x1" specimens machined from the boards were dried at 230°F and submerged 1.5 inches in a 5182 aluminum alloy at 1400°F for 3-10 minutes. The changes in specimen thickness and weight were measured immediately after removal from the molten metal and after the specimens had cooled for 15 minutes.

COMPRESSIVE (COLD CRUSH) STRENGTH: The compression strength was measured by loading 1"x1"x6" specimens perpendicular to the pressing direction. The testing was performed at Westmoreland Testing Laboratories.

LUBRICANT ABSORPTION: The material's propensity for absorption of the lubricant was determined by hot oil immersion tests. In the hot oil immersion test, 2"x2"x1" specimens were immersed in 400°F oil (the same composition used during casting). The weight change was measured after 0.5, 1, and 3 hour exposure times.

MODULUS OF RUPTURE: The modulus of rupture was measured on 1"x1"x6" specimens. The testing was performed at Westmoreland Testing Laboratories.

MICROSTRUCTURAL ANALYSIS: Scanning electron microscopy (SEM) was used to examine the microstructural features. Elemental dispersive X-ray analysis (EDAX) was further used to characterize the materials microstructural and chemical make-up.

ATC 0034961

discussions, Willie Lansdale has stated that the more recent Pabco boards do not appear to contain these white spots.

A memorandum from R. A. Marra (1991 May 03) discussed the microstructural and chemical analysis of Pabco Metaltemp header surface used to cast 5082 alloy at Warrick Operations. The analysis showed that magnesium and to a lesser extent aluminum attacks the calcium silicate bond phase at the header surface. It has also been shown the additions of minerals containing FeO (such as red mud and vermiculite) are beneficial in preventing this attack. Therefore, it is recommended that the metaltemp boards be used as header material only for the casting of low magnesium containing alloys and the modified composition containing red mud and vermiculite be used for the higher magnesium containing alloys.

Conclusions and Recommendations

The Pabco Metaltemp board materials have physical and chemical characteristics similar to the header-grade molten metal marinite (with the exception of containing no asbestos). In comparison with the BNZ Metalform material, the Pabco material's cold crush strength and modulus of rupture are significantly higher. The higher density Pabco boards also showed much less oil absorption than the BNZ material.

Pabco's material should be considered for use as feedboards for HDC casting of 3xxx and 5xxx series alloys and for headers for casting the lower magnesium containing alloys. The modified mold clamp design should be used to optimize performance. The higher density boards (50 pcf) should perform better than the lower density materials because of their higher strength (more easily machined and handling), higher crushing strength (less likely to fail in the mold package), and lower propensity for oil absorption. The 600°F heat treatment appears adequate for these applications. In fact, the higher heat treatment (1100°F) may result in increased porosity and oil absorption and a decrease in the material strength.

The material should be monitored for density, strength, hardness, moisture and carbonate content (TGA/Mass Spec), and hot oil absorption on a regular basis. The microstructure should also be examined to ensure that large unreacted CaO is not present in the board and especially along the machined header surface. It appears Pabco has done a much better job than BNZ in controlling their lime supply and screening the raw material to remove any large particulates.

Table II
Pabco Meteltemp Board Characteristics

Material		HG/MMM	BNZ Metalform	104-6-2	104-8-2	112-16-1	112-16-1 AA HT 1100°F	112-16-1 XG Dry 230°F HT 1100°F
Test	Units							
Density (pcf)	pcf	45.8±1.4	48.1	51.4±0.3	52.6±0.3	49.8±1.4	48.7±0.5	51.9±0.2
Hardness	Durometer	69.6±3.2	64	71.4±0.9	61.0±1.3	70.7±0.7	68.9±0.7	71.8±0.9
Molten Metal	% weight gain			1.49±0.04	NM	0.3	0.33	NM
Immersion	% dimensional change			0.4±0.2	NM	0.23±0.21	0.23±0.21	NM
Hot Oil	% weight gain							
Immersion	0.5 hr	20.3	34.7	25.5±2.6	24.9±0.9	23	34.6	NM
	1 hr	31.3	41.6	30.4±4.1	30.4±1.5	26.2	37.5	NM
	3 hr	44.8	57.5	35.9±1.1	37.0±3.8	32.9	48.9	NM
Compressive Strength	psi	1960±160	1450	2210±340	2560±250	2100±160	1810±60	1920±140
Modulus of Rupture	psi	1520±160	670	1080±460	1280±260	950±140	970±210	990±130

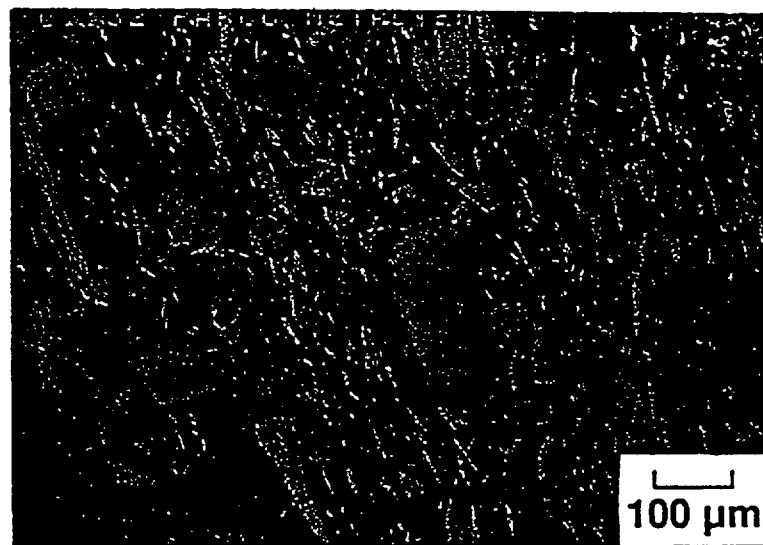
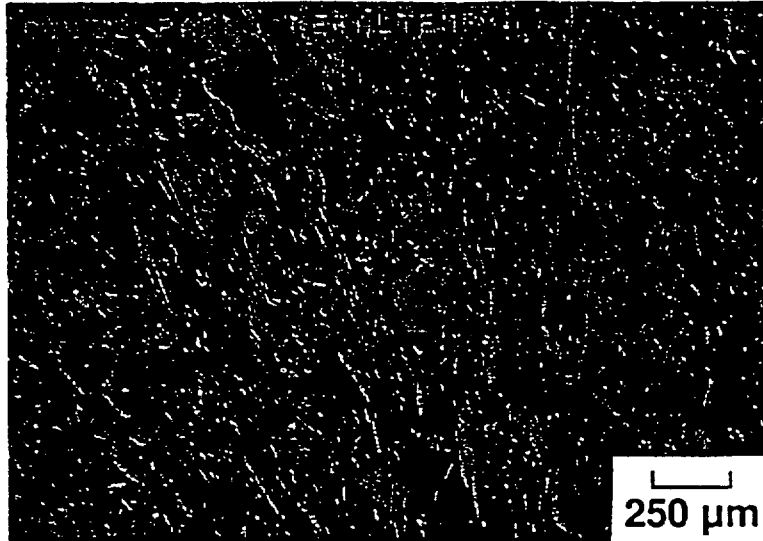
Table II
Pabco Meteltemp Board Characteristics

Material		121-19-1	121-19-1 AA HT 1100°F	121-19-1 XK Dry 230°F HT 1100°F	122-15-1	122-17-1	123-16-1	123-16-1 AA HT 1100°F
Test	Units							
Density (pcf)	pcf	48.8±1.8	48.6±0.5	48.7±1.6	52.1±0.5	51.0±0.5	39.9±1.0	37.9±0.4
Hardness	Durometer	70.7±0.7	67.0±0.9	67.7±0.1	71.2±0.8	68.7±1.0	53.9±2.2	47.8±1.1
Molten Metal	% weight gain	1.7	0.4	NM	1.9±0.3	1.8±0.2	1.5±0.0	0.31
Immersion	% dimensional change	0.47±0.15	0.13±0.12	NM	0.47±0.15	0.35±0.16	0.03±0.05	0.10±0.10
Hot Oil	% weight gain							
Immersion	0.5 hr	31.2	37.8	NM	28.3±0.1	34.3±3.2	66.8±6.1	79.5
	1 hr	31.7	46.3	NM	35.1±0.2	38.8±3.8	78.1±3.2	98.7
	3 hr	42.7	53.3	NM	35.2±2.4	36.3±0.7	93.2±6.7	97.1
Compressive Strength	psi	1910±230	1680±130	1590±200	1910±250	1600±160	1010±110	830±90
Modulus of Rupture	psi	1030±150	1330±130	720±40	1220±270	970±120	630±100	750±100

Table II
Pabco Meteltemp Board Characteristics

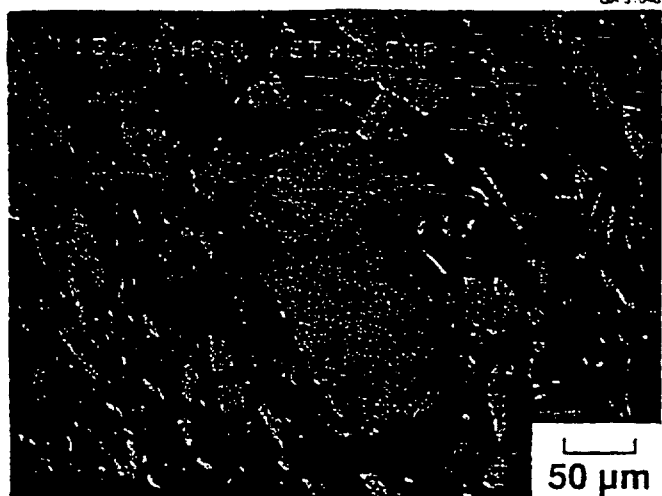
Material		123-16-1 XQ1 Dry 230°F HT 1100°F	124-8-2	124-8-2 AA HT 1100°F	124-8-2 XQ4 Dry 230°F HT 1100°F	124-14-1	124-18-1
Test	Units						
Density (pcf)	pcf	41.2±0.1	44.3±0.5	43.4±0.2	43.8±0.1	44.6±0.5	39.5±0.9
Hardness	Durometer	54.8±1.4	59.6±0.9	56.7±0.4	62.0±0.6	61.3±2.0	62.4±1.2
Molten Metal	% weight gain	NM	1.7±0.0	0.57	NM	1.3±0.1	1.3±0.1
Immersion	% dimensional change	NM	0.17±0.08	0	NM	0.32±0.08	0.32±0.19
Hot Oil	% weight gain						
Immersion	0.5 hr	NM	53.3	59.8	NM	58.2	70
	1 hr	NM	54.4	66	NM	79.5	83.4
	3 hr	NM	78.2	72.5	NM	84.1	85.7
Compressive Strength	psi	960±130	1250±120	910±40	1150±50	1241±10	950±100
Modulus of Rupture	psi	560±80	570±170	400±90	670±50	790±270	440±120

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Microstructure of a Typical Pabco Metaltemp Board
Figure 1

ATC 0034968



**Pabco Metaltemp Board Showing Unreacted
CaO Particle. (Top Photo: SEM Image,
Middle: Ca Map, Bottom: Si Map)**

Figure 2

TID 18656

ALUMINUM COMPANY OF AMERICA
ALCOA LABORATORIES



ELIMINATE ASBESTOS IN ALL FURNACE APPLICATIONS

R. G. LaBAR

March 13, 1974

"CONFIDENTIAL"

INGOT CASTING DIVISION
REPORT NO. 11-74-12B103

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A handwritten signature in cursive script, appearing to read 'R. G. LaBar', is written over a horizontal line.

R. G. LaBar
Group Leader

Noted by

A handwritten signature in cursive script, appearing to read 'G. K. Turnbull', is written over a horizontal line.

G. K. Turnbull
Section Head

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ALUMINUM COMPANY OF AMERICA
ALCOA LABORATORIES



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G. K. Turnbull
Section Head

ATC 0034971

SYNOPSIS

This report summarizes the program to replace asbestos containing products in applications other than HDC and FDC headers. A broad number of candidates (Table I) and test data (Tables II-VI) were narrowed to a specific group of recommended materials. Appendix A contains the recommendations along with updated procedures for installation and use. Whereas the project is closed out, assistance and advice will be provided as required. Further, candidate materials for insulation, trough linings and packing, basin and FDC pot linings, etc. will be evaluated in Project 06W05311, Refractory Evaluation.

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INTRODUCTION

Overwhelming evidence in recent years links human exposure to asbestos dust with several forms of cancer. One result of this is the U. S. Asbestos Standard* which greatly restricts the exposure limit (no. particles/volume air/ unit time) effective July 7, 1972 with a further reduction effective July 1, 1976. The restrictions are so severe, that the dust handling equipment, monitoring equipment and additional personnel required to monitor concentrations will make the use of asbestos containing materials impractical as well as uneconomical.

The major thrust towards solving the problem is the elimination of asbestos containing products in all applications. However, the unique properties of asbestos and many asbestos containing products are not readily matched by any one substitute for all applications. In fact, many different materials had to be qualified and specified for use in various applications; hence, the purpose of the project and this report. Preliminary to this report was a recommendation issued by D. R. Barch and the author in April 1973. The appendix of this report is an update of the original recommendations based on laboratory and in-service results.

CANDIDATE MATERIALS

The number of replacement materials is great, as is the variety of trade names and manufacturer's claims for them. To minimize the confusion, Table I lists the materials according to the following categories:

1. Castables - supplied in bags or drums to be mixed with water and poured, tamped or troweled in place.
2. Moldables - supplied in bags or drums to be used as is (if premixed) or mixed with water and tamped, troweled and/or molded by hand.
3. Board - in most cases to be cut, installed and used as is. However, some products require coatings to improve erosion and corrosion resistance when used in metal contact.
4. Miscellaneous - includes packing materials, gasketing, cements, etc.
5. Preformed Special Shapes - includes drop-in trough liners and other special shapes (FDC pots, etc.).

As a list of major candidates, Table I is neither a list of recommendations, nor an all inclusive compilation of alternatives. Recommendations are detailed later in Appendix A. Materials intentionally excluded from Table I include inadequate candidates plus other materials which were evaluated and for one

*Federal Register, Vol. 37, No. 110, pp. 11320-11322 (6/7/72).

reason or another failed our laboratory tests, but will appear elsewhere in this report for comparisons.

EVALUATION AND TEST PROCEDURE

The methods for testing asbestos replacement materials vary with the intended use.

The standard cup and immersion tests were used to evaluate those materials which could be prepared in the required sample configurations and which would be used in metal contact in transfer systems.

Because the severity of metal corrosion at 1500 and 1400 F is significantly greater than that at 1350 F and lower, many materials (for applications downstream from the holder) were tested in contact with metal at 1350 F.

In several cases, materials were evaluated in service and found to be suitable prior to any laboratory tests.

Laboratory, in-service evaluations were conducted on nearly all materials in various forms to determine those performance characteristics not attainable in standard test procedures, e.g. thermal stress cracking, erosion resistance, handling characteristics, etc.

RESULTS

Table II contains the results of standard tests on a number of castables. Of particular interest is the rather high silicon pickup from most of the fused-silica castables and yet their actual performance, as noted in Table V, is quite satisfactory. Conversely, the low silicon pickup from Alcoa C-1 is somewhat counteracted by its tendency to crack from differential thermal stresses. Tables III and IV contain the results of tests run on moldables and board products. The comments under thermal stability refer to degradation due to any decomposition of the material during the test or subsequently to its exposure in air and normal levels of humidity which can cause rehydration of some cementitious phases, i.e. dicalcium silicate, calcium sulfate, etc.

The conflicting results between cup tests and in-service evaluations can be attributed to lower metal temperatures and, in most cases - less corrosive alloys than 7075.

The low temperature tests produce better correlation with in-service evaluations. Further, the use of coatings, e.g. Lumnite, Glasrock Sealer, J-M Ceracote, Zirconite, whiting, etc. on moldables and board products upgrades their ability to resist erosion and wetting (metal adgency) so that their in-service performance is significantly different from that predicted from cup and immersion tests.

Results of plant trials at Warrick using Thermo 340 (R-340) and a developmental mix designated R-616 (contains equal amounts of fused-silica and plaster with somewhat less calcium aluminate, CA-25) were less than satisfactory. The materials were not sufficiently durable to survive beyond 2 1/2 mos. (Thermo 340) and 5 mos. (R-616) respectively. These results correlate with our observations in the laboratory. We have found that with materials containing considerable amounts of plaster (CaSO_4) partial rehydration occurs during cyclical use. This phenomenon produces swelling and dusting which weakens the material. The A-641 product was developed with this in mind so that it only contains 1% plaster which is added to accelerate the hydraulic set of the CA-25 cement so that higher productivity of cast pieces is possible (1% plaster addition halves the time required to develop handling strength).

One unique feature of the R-340 material is that it expands during curing more than it shrinks during firing. We have utilized this property to successfully line pump piping and FDC pot shells. The linings are tight, quite durable and easy to install as the R-340 mix is fine-grained and thus flows easily into small annular spaces.

As mentioned above, we have developed a new product called A-641. This material, in a preformed and fired shape (board or otherwise) can be cut to the required size with standard metal cutting tools and bolted in place. However, because the material is brittle, a back-up cushion of ceramic fiber blanket or ceramic fiber paper is recommended. A companion product now under evaluation is A-646. This is a slight modification incorporating a corrosion inhibiting zinc borosilicate frit which all but eliminates wetting and metal attack (see data in Table IV).

Another new product development is A-647. The concept of this material was derived from J. P. Austin's Mandoseal (See Table III) which contains Portland Cement (unstable in cyclical use). The objective was a moldable mix using expanded vermiculite blended with various other materials to obtain a product which could be wet mixed more easily than mineral wool containing moldables, would have low drying and firing shrinkage and could be used as a substitute for asbestos shorts/Lumnite.

The need for a suitable moldable in many applications is critical. One of the major problems with their use is cracking due to shrinkage. In order to quantify shrinkage versus temperature and product, a series of tests was conducted. Samples of each material were formed into bars 1 in. x 1 in. x 10 in. and then measured after curing, air drying, oven drying to 230 F, firing to 600 F and then to 1350 F. The data are shown in Figure 1 and Table VI.

Of particular interest is the extremely low firing shrinkage of Consolidated's Moldable, Panelex Super (still being evaluated in the laboratory), Variform and A-647. The JM-375 L and asbestos/shorts Lumnite seem to increase their rate of shrinkage

with temperature which is attributed to irreversible phase changes in their fibrous components. The Panelex Super is a new product containing some mica (probably vermiculite) which would tend to counteract shrinkage of the mineral wool constituent. As noted in Table VI, the total shrinkage of the Consolidated Moldable is 0.0%. However, as seen in Figure 1, shrinkage occurred during air and oven drying to 230 F. Reports from the field indicate that this product does not shrink at all. This is understandable in view of the fact that in our test, the bars were allowed to air and oven dry lying on one side. Thus the bottom did not dry at the same rate as the top surface, causing the bars to warp (compared to the other materials, this product dries slowly).

Finally, a comment is warranted about moisture content. As reported for JM-375 L, its shrinkage was only 1.4%. However, the mix was prepared using 50% water. When 75% water was used, the shrinkage nearly tripled. This type of sensitivity led us to embark on the development of A-647 and simultaneously begin to search for other products such as Plisulate and Panelex Super.

The prices in Table VI are not precisely accurate but were based on recent data to provide a comparative base. The figures for A-647 and A-641 were estimated from available data on raw materials and trial manufacturing (costs to us by outside vendors for preparing experimental materials). The range of prices for fused-silica castables is the difference between the least expensive (Glasrock 840) and the most expensive (Silfrax ARC).

RECOMMENDATIONS

In keeping with our overall effort, previous recommendations have been amended to reflect the results of our work at the Laboratories and to include the results of various and successful plant trials. These recommendations appear in Appendix A.

While the reader may at first be somewhat confused by the diversity of materials, it is recognized that local availability and delivery will at times necessitate the selection and use of alternate products within a specific category.

Recommendations on the use and source of supply for A-641 and A-647 will be forthcoming as more extensive in-service trials are completed and/or when agreement is reached with a vendor(s) for their manufacture.

CONCLUSIONS

1. Molten Metal Marinite and asbestos insulation can be replaced by asbestos free materials in troughs, basins and for back-up insulation.
2. Asbestos shorts/Lumnite and asbestos rope can be replaced by

asbestos free materials for troughs, pot linings and miscellaneous packing.

3. The recommended substitute materials are described herein. They are numerous and, in many cases, suited for a limited number of applications dependent on thermal conductivity, erosion resistance, corrosion resistance, bulk density (as this pertains to inclusions and metal quality), workability and cost.
4. In many instances, asbestos free substitutes for Molten Metal Marinite are less expensive, more refractory but in no instance as uniquely versatile or flexible.
5. More care in handling and use will be required when using other materials where Molten Metal Marinite has been the standard.

TABLE I

MAJOR CANDIDATE MATERIALS BY CATEGORY

<u>Category</u>	<u>Class</u>	<u>Brand</u>	<u>Manufacturer</u>
Castables:			
1.	Fused-silica/calcium aluminate -	Glasrock 840 Glasrock 820 Insilrock Silfrax ARC Thermosil 120 Thermosil TG Vis Sil	Glasrock Co., Inc. " " Carborundum Co., Inc. Thermo Materials, Inc. " Sil Base Co.
2.	Insulating aluminous/calcium aluminate	Kast-O-Lite 30	A. P. Green Co.
3.	Dense aluminous/calcium aluminates	Alcoa C-1 SJP Cast	Alcoa, Bauxite, Ark. Wahl Refractories Co.
4.	Eucryptite/calcium aluminate	LO-X (Duramics)	Pryor - Giggey Co.
Moldables:			
1.	Mineral Wool/calcium aluminate	JM-375L Panelex Super P-430	Johns-Manville Co., Inc. Quigley Co., Inc.
2.	Ceramic fiber/calcium aluminate	Variform A and B Consolidated Moldable	Carborundum Co., Inc. Consolidated Ceramics
3.	Ceramic fiber/colloidal silica		
4.	Vermiculite/talc/calcium aluminate	A-647	Alcoa Laboratories
5.	Phosphate bonded plastics	Solar 80	J. P. Austin Associates

<u>Category</u>	<u>Class</u>	<u>Brand</u>	<u>Manufacturer</u>
Board: (See Preformed also)			
1.	Fiberglass/calcium silicate	Thermo 12(Superex Kaylo 10 1600) Superex SG Carey Hi Temp	Johns-Manville Owens Corning Fiberglas Johns-Manville Carey Insulation Co.
2.	Ceramic fiber (vacuum formed)	Thermotect A or B Hot Board M - Board	CE Refractories, Inc. Carborundum Babcock & Wilcox
3.	Mineral wool	Various	Many
4.	Fused silica	Glasrock Foam	Glasrock, Inc.
5.	Fused silica/calcium aluminate	A-641	Alcoa Laboratories and Sil Base Co.
6.	Fused silica/plaster/colloidal silica	Thermo 340	Thermo Materials
Miscellaneous:			
1.	Packing (Ceramic fiber bulk, rope and blanket)	Fiberfrax Kaowool Cerafelt	Carborundum Babcock & Wilcox Johns-Manville
2.	Gasketing	Fiberfrax Paper Fiberseal	Carborundum Fibrous Glass Products
Preformed Trough Liners:			
1.	Ceramic fiber	Thermotect A M - Board Kalmin 5000	CE Refractories, Inc. Babcock & Wilcox Foseco, Inc.

<u>Category</u>	<u>Class</u>	<u>Brand</u>	<u>Manufacturer</u>
2.	Fused silica	Masrock	Glasrock Co., Inc.
3.	Fused silica/calcium aluminat	A-641	Sil Base Co.
4.	Fused silica/plaster/colloidal silica	Thermo 340	Thermo Materials

RGL/ae

Note: Materials recommended for plant use are listed in Appendix A.

TABLE II

LABORATORY TESTS ON CASTABLES

Brand	72 Hour Cup Test/70/5 Alloy			
	Temp.	Pickup		Penetration
		Si	Fe	
Glasrock 840	1500	17.9	.09	Severe
Glasrock 820	"	19.9	.07	"
Duramics 10-X	"	3.8	.03	"
V1 Sil	"	17.9	.07	"
Thermosil 120	"	19.9	.07	"
Thermosil TG	"	11.9	.07	"
Silfrax ARC	"	.12	.00	None
Kast-O-Lite 30	"	2.31	.06	Slight
Alcoa C-1	"	.03	.04	Negligible
SJP Cast	(Same as Alcoa C-1)			

TABLE III

LABORATORY TESTS ON MOLDABLES

Brand	72 Hour Cup Test/7075 Alloy				"B" Immersion			Thermal Stability
	Temp.	Si	Pickup Fe	Penetration	Temp.	Cracks	Penetration	
JM-375 L	1350°F	.01	.02	None	1400°F	None	None	Excellent
JM-375					"	Many	"	Poor
Mandoseal (Vermiculite/ Portland Cement)	1500°F	.00	.02	None				Poor
Variform A	1350°F	.04	.03	None				
A-647	1350°F	.05	.04	Negligible				
Solar 80	1500°F	2.45	.23	Severe				Excellent
Consolidated Moldable	1350°F	14.0	.09	Severe				
LDS Moldable (Carborundum)					1400°F		Severe	N.A.

TABLE IV
LABORATORY TESTS ON BOARD PRODUCTS

<u>Brand</u>	<u>72 Hour Cup Test/7075 Alloy</u>			<u>"B" Immersion</u>				<u>Thermal Stability</u>
	<u>Temp.</u>	<u>Pickup</u>		<u>Penetration</u>	<u>Temp.</u>	<u>Cracks</u>	<u>Penetration</u>	
		<u>Si</u>	<u>Fe</u>					
Thermo 12	--	--	--	--	1400°F	None	None	Poor
Kaylo 10	1350°F	.01	.03	None	--	--	--	Fair
Thermotect A	--	--	--	--	1400°F	--	Severe	--
M - Board	--	--	--	--	1400°F	--	Severe	--
A-641	1350°F	2.69	.02	Slight	--	--	--	Excellent
A-646*	1350°F	.19	.02	None	--	--	--	V. Good
Thermo 340	1500°F	.41	.06	Negligible	--	--	--	Fair

*A-646 is a modification of A-641 with a frit additive.

TABLE V

IN SERVICE EVALUATION RESULTS

<u>Brand</u>	<u>Erosion Resistance</u>	<u>Cracking Resistance</u>	<u>Metal Adherence</u>	<u>Durability</u>
M.M. Marininite (for comparison)	Excellent	Excellent	None	Excellent
Glasrock 840	Good	V. Good	Slight	Good
820	"	"	"	"
Duramics LO-X	"	"	"	"
V1 Sil	"	"	"	"
Thermosil 120	"	"	"	"
TG	"	"	"	"
Silfrax ARC	"	"	None	"
Kast-O-Lite 30	V. Good	Fair	Negligible	V. Good
w/Luminite Wash				
Alcoa C-1	Excellent	Fair	Negligible	Excellent
JM-375L	Poor	Fair	Slight	Fair
Variform	Good	Good	None	Good
A-647	Poor	Good	Slight	Fair
Solar 80	Excellent	Excellent	None	Excellent
Consolidated M.	Good	Good	Slight	Good
Thermo 12	Poor	Excellent	None	Poor
Thermotect A	Good	Excellent	Negligible	Good
w/Glasrock Sealer				
A-641	V. Good	Excellent	Slight	V. Good
Thermo 340	Good	Fair	None	Fair

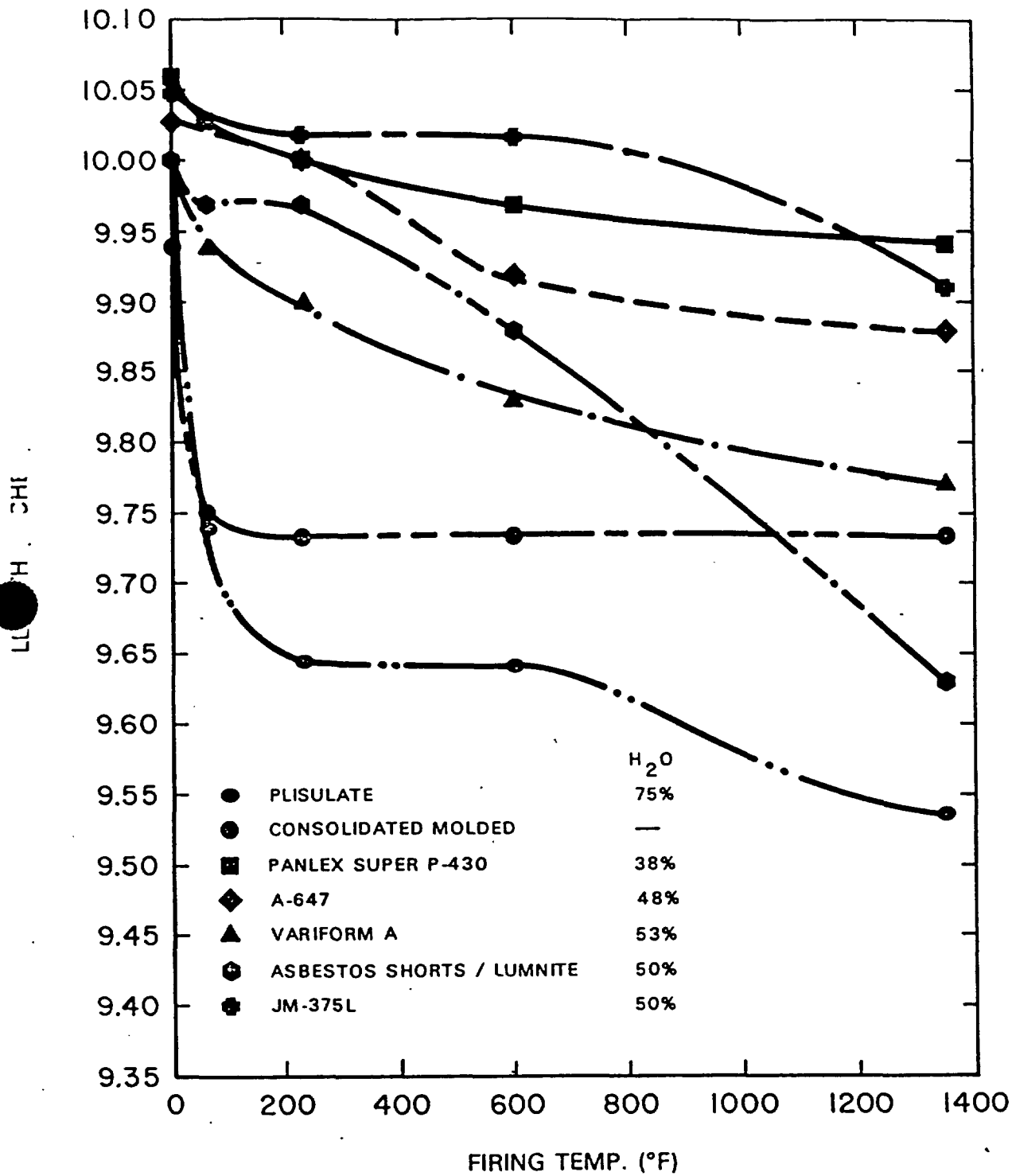
TABLE VI

PHYSICAL AND THERMAL PROPERTIES

Brand	Fused-Silica Castables	Solar		Kast-O- SJP		JM-375L	Variform		Consolidated Moldable	Thermotect		A-641	Thermo	
		80	Lite	30	Cast		A-647	A		340	M. M.			
Bulk Density - (lb./cu. ft.) (g/cc)	110-111 1.76	175 2.80	85 1.36	150 2.40		40-50 .64-.80	45-55 .72-.88	.63* 1.01	40 .64		28 .45	90* 1.44	95 1.52	33-40 .53-.64
Strength -														
Cold MOR (psi) (kg/cm ²)	750 52.7	(1800) 133	300 21	900 63		-- --	130 9.1	-- --	-- --		-- --	280* 19.7	750 52.7	600-700 46
Cold Crush (psi) (kg/cm ²)	5000 352	(7500) 527	1000 70	-- --		-- --	-- --	-- --	-- --		-- --	830* 58.4	-- --	-- --
Shrinkage - Rm - 1350°F	0.6	0.1	0.1	0.2		1.4*	2.6*	1.5*	0.0*		0.5	0.7*	1.0*	1.85%*
Conductivity - Btu/hr./ft. ² /°F/in.	5-6	(9.0)	2.69	(15)		.84	0.8	--	0.5		0.5	3.1*	3.5*	1.0*
Thermal Exp. - { in./in./°F } { in./in./°C }	.45-.65 .82-1.19	-- --	-- --	-- --		-- --	-- --	-- --	-- --		-- --	(.5) --	-- --	1.3x10 ⁻⁶ 2.4x10 ⁻⁶
Price - \$/ft. ³	18-50	31	16	20		3	43	(4)	54		35	(18)	60	22

* ARL Data

() Figures in parentheses are estimated.



NEAR FIRING SHRINKAGE INSULATING MOLDABLES

FIGURE 1

APPENDIX A

Recommendations for Preparation and Installation
(Supercedes Memo by D. R. Barch and R. G. LaBar
of April 27, 1973).

A. Melter to Holder

Castables: Alcoa C-1, SJP Cast, Kastolite 30, fused-silica castables (See Table I in text).

1. Recommended minimum wall thickness is 2 inches.
2. Follow manufacturer's recommended mixing instructions for a pourable mix using minimum water.
3. Coat forms with oil, grease or polyethylene sheet (Stapled to forms).
4. Tack weld expanded metal mesh lathing to inside of trough 1/2 - 1 inch from steel work.
5. Post-casting procedure:
 - a. Cover casting with plastic sheet for 24 hours.
 - b. Dry casting in air for 24 hours.
 - c. Castables should be heated slowly to 600 F prior to use, preferably overnight.

Caution: Avoid flame impingement and rapid heatup.

6. The recommended coatings, except for Silfrax ARC which requires none, are Lumnite, Plistix 900 or 900F.

Moldables: Solar 80 Plastic

1. Recommended minimum wall thickness is 1 1/2 inches.
2. Follow manufacturer's recommended procedure for installation paying particular attention to leaving a roughened surface - do not smooth or overwork the surface!
3. One half to one inch of mineral wool or fiber glass board back-up insulation is recommended. It can be glued to the steel lining with sodium silicate.
4. Post-molding procedure:
 - a. While some air drying is permissible slow heating of this material is recommended after installation is complete.

Apply heat uniformly across the entire section.
 - b. Heat slowly to 230 F and hold for 1/2 day per inch thickness.
 - c. Raise temperature to 600 F and hold for 1/2 day per inch thickness prior to use.

Caution: Avoid flame impingement during initial heatup and use.

5. Coatings are not required.

Brick: Coral P or Chas. Taylor 12054

- a. Use R & I Super 3000 mortar.
- b. Standard practice for refractory brick installation.

B. Holder to Molten Metal Treatment Process:

Castables: Fused-silicas or Kast-O-Lite 30.

1. Follow recommendations in Section A for castables.

Moldables: JM-375L, Variform B, Consolidated Moldable.

1. Use 50 lb. water to 100 lb. JM-375L for power mixing. Maximum addition should not exceed 75 lb. water to 100 lb. JM-375L!
2. The same restrictions stated in 1 above apply to variform.
3. Thorough mixing will permit the use of minimum water and produce more uniform, workable materials.
4. Consolidated Moldable is ready to use as received.
5. Tack weld expanded metal mesh lathing to inside of trough 1/2 in. from steel work for long trough sections, unless the trough is equipped with an overhanging lip.
6. May be hand molded, tamped and/or troweled in place.
7. Do not overwork the surface!
8. Post-molding procedure:
 - a. Cover with plastic sheet for 24 hours.
 - b. Air dry for at least 24 hours, (longer for Consolidated Moldable).
 - c. Heat each section (between joints) uniformly and slowly to 230 F overnight.
 - d. Continue heating to at least 600 F prior to use.

Caution: Avoid flame impingement, non-uniform heating, rapid heatup and scraping with sharp edged tools.

9. The recommended coatings are Lumnite, Plistix 900 and 900 F, J-M Cerakote, whiting (apply whiting by rubbing it into the surface after step 8 b. above and brush away all the excess).

Board and Preformed Shapes: Vacuum Formed

Ceramic Fiber (see Table I in text), Glasrock Foam (50 pcf).

1. These materials may be installed using the same procedure as with Molten Metal Marinite or follow manufacturer's instructions (especially in the case of Glasrock Foam).
2. Recommended coatings are Lumnite, Glasrock Sealer (with Glasrock Foam), J-M Cerakote, Plistix 900 and 900 F, whitening (as per instructions under moldables above), or coating supplied with Kalmin (Foseco).

Caution: Avoid flame impingement and scraping with sharp edged tools.

C. Downstream from Molten Metal Treatment Process

Moldables: Same as B above.

Board: Same as B above except do not use Lumnite or Plistix coatings.

D. Packing and Miscellaneous

Ceramic fiber bulk, blanket and rope may be used in place of asbestos rope for packing, etc.

Thermo 12, Kaylo 10, or Ceramic fiber board products may be used for insulation in place of Thermobestos, Superex, Molten Metal Marinite, etc.

General In Service Care and Cautions

1. Avoid excessive preheating. If preheating is necessary, apply uniform heat, avoiding hot spots and direct flame impingement.
2. Avoid mechanical abuse.
 - a. Install protective caps over linings near tap spouts.
 - b. Lift skulls by hand, minimize scraping and/or gouging.
3. Extra care is required when installing dams in drop-in liners to avoid breakage. Recommend use of ceramic fiber paper (Fiberfrax, Cerapaper, etc.) gasketing around dams and joints.
4. A program of scheduled inspection and maintenance should be implemented.

It is recognized that individual plant differences exist in trough usage and design, but it has been concluded that these differences can be taken care of if made known and discussed.

The ingot plant metallurgists are requested to inform M. L. Redhair of contacts with various vendors and receipt of sample materials. In addition, brief feedback on plant experiences, either pro or con, with the recommended materials are requested.

New materials and/or revised procedures for installation and use will be added to the list as they develop.

FROM G. K. TURNBULL
INGOT CASTING DIVISION
ALCOA TECHNICAL CENTER - B

TO MR. J. E. HATCH
INGOT & POWDER PRODUCTS DIVISION
PITTSBURGH OFFICE - 23

"CONFIDENTIAL"

August 8, 1974

RE: ELIMINATE ASBESTOS IN ALL FURNACE APPLICATIONS
Ingot Casting Division Report No. 11-74-12B103

Asbestos has become thoroughly engrained in ingot casting practices through generations of use. We've learned where and how to use it, and have even adapted our practices to take maximum advantage of its characteristics. Yet we must curtail such use and do so over a fairly short transition period.

We could hardly expect to have a shelf item that is the ideal substitute: superior to asbestos in every way. Yet in the series of engineering tradeoffs inherent in such substitution we are fortunate to have a series of alternatives adequate to undertake nearly every aspect of substitution. Some preexisted and have been evaluated by plants and Alcoa Laboratories. Others were recently developed at the Laboratories. Major candidates are compared in the attached report by Dr. R. G. LaBar. Preferred candidates have been singled out into the Appendix section and paired up with detailed installation instructions.

Preferred technique for use of the report is to use Appendicized materials and limit them to the applications and procedures described. If local conditions, refractory availability or other considerations override this approach, some insight into the shortcomings of other candidates are described in the body of the report. It is strongly recommended, however, that use of the not-recommended materials be coordinated through Dr. R. G. LaBar or Mr. D. R. Barch to provide maximum opportunity for their success and to extend the extremely important "In Service Evaluation Results" of Table V.

A separate report will be issued covering parallel developments on asbestos elimination in headers.

Mr. Sartschev plans to expand narrow initial distribution by submitting to you a list of plant recipients.

Keith Turnbull
G. K. TURNBULL

GKT:sh

Attachment

cc: R. E. Spear/TID

T. R. Gauthier/J. H. Dunn - Pgh
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ALCOA

1993 December 14
Report No. 06-93-85

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**HDC HEADER AND FEEDBOARD MATERIAL
DEVELOPMENT AND PRODUCTION QUALIFICATION**

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IT SHOULD BE NOTED THAT THE MATERIAL FORMULATIONS
DESCRIBED IN THIS REPORT ARE CONSIDERED HIGHLY
PROPRIETARY AND SHOULD NOT BE DISCUSSED OR DISCLOSED
WITH ANY NON-ALCOANS WITHOUT THE CONSENT OF WARRICK
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EXECUTIVE SUMMARY

Calcium silicate board materials are used as headers and feedboards at Warrick Operations for the Horizontal Direct Chill (HDC) casting of aluminum ingots. Header Grade Molten Metal Marinite (HGMMM), an asbestos-reinforced board material, has historically been used in this application. Due to the health hazards associated with this asbestos containing material, replacement materials for both the headers and feedboards needed to be identified for Warrick Operations to meet its ingot production demands. This report describes the activities which led to the development and production qualification of Pabco PMT and PMTHG calcium silicate materials for feedboards and headers. During the program, an improved understanding of the effects of the header material and mold package design on the casting performance was developed. This understanding was critical to the success of the program. An excellent working relationship was established between ATC, Warrick (MPE-Ingot and Production) and Pabco (materials vendor) personnel. The development of standard operating procedures by Warrick mold preparation and ingot casting personnel greatly aided the evaluation of potential replacement materials. The use of designed experiments and statistical analysis provided the basis for downselecting the potential material formulations. Statistical methods were further utilized to optimize the material formulation, mold package design, and casting process variables. Proposed activities for further material refinements and HDC process optimization which should result in improvements in casting life, ingot recovery, and ingot quality are discussed.

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I. INTRODUCTION

Calcium silicate board materials are used as headers and feedboards at Warrick Operations for the Horizontal Direct Chill (HDC) casting of aluminum ingots. The HDC process was originally designed to use Header Grade Molten Metal Marinite (HGMMM) for the header and feedboard. HGMMM is an asbestos fiber reinforced calcium silicate board material. Due to the health hazards associated with this asbestos containing material, replacement materials for both HDC headers and feedboards are required for Warrick Operations to meet its ingot production demands. An acceptable header replacement should have the characteristics shown in Table I. The feedboard should have similar characteristics, although the resistance in molten metal attack is less important. The moisture and carbonate content of the feedboard is extremely important since a large area is exposed to molten metal contact.

Table 1
Desired Characteristics for HDC Header Materials

Characteristic	Importance
Molten Metal Resistance (Low Wettability)	- Prevent metal / header reactions which degrade the header surface and result in ingot defects.
Low Thermal Conductivity	- Prevent molten metal solidification at the header surface.
High Compressive Strength	- Prevent crushing of header in the mold package.
Low Moisture / Carbonate Content	- Prevent risk of explosion when contacted with molten metal and avoid outgassing which degrades ingot quality.
High Abrasion Resistance	- Prevent degradation of header surface during casting.
Low Porosity / Permeability	- Minimize lubricant absorption.
Adequate Strength (MOR)	- Allow machining and handling of header.
Low Thermal Expansion (Good Thermal Stability)	- Minimize change in header / oil ring overhang and stress build-up in mold package.

Pabco Metaltemp (PMT) and BNZ Materials MetALform calcium silicate boards have been evaluated for feedboard and header replacement. These materials are boards produced by pressing slurries of lime (CaO), silica (SiO_2 ; typically added as diatomaceous earth and a colloidal material), wollastonite (CaSiO_3), and alkali resistant glass fibers. The pressed boards are autoclaved to convert the lime and silica into a calcium silicate compound. The autoclaved boards are further heat treated to remove physically and chemically bound water.

Recent work on identifying a header replacement has shown that chemical reactions between the metal and header material are important in determining the header life, i.e., the life that the header can be used before ingot defects and excessive scrap results in casting termination. It is believed that the magnesium and aluminum in the molten metal attack the calcium silicate bond phase which results in defects on the header surface. The molten metal can solidify in these areas. The solidified material continues to grow until it is trapped in the solidification front and pulled out by the moving ingot. This process results in ingot strawberries, tears, or surface cracks which increase the ingot scrap and eventually lead to termination of the casting run.

This report details the header and feedboard program activities and accomplishments since the first quarter of 1991. The format of the report describes the progress (where we are in the program) and the chronological steps that were taken to develop and implement the replacement materials. Details on the understanding of the mold package functionality are also discussed. The success in finding alternative HDC mold package materials and improving the mold package design can be largely attributed to the emphasis that had been placed on understanding the mold package and the header / molten metal interactions during casting. Finally, planned and proposed activities for future improvements are presented.

II. PROBLEM STATEMENT

In 1990 October, the following project objective was established:

New products for marinite replacement must meet the following criteria: 1) The average mold life must equal or exceed the average life with header grade molten metal marinite for all alloys; and 2) The casting recovery must be greater than or equal to 88% due to header related problems.

In 1991 October, this objective was modified to reflect the changes in ingot production schedules to reduce "flowtime." With the emphasis of reducing the material "flowtime" through the plant, achieving maximum mold life was no longer the primary goal. The new objective was:

Develop a replacement for the header grade molten metal marinite header to be used in 5XXX alloy casts which meets production goals. The header life objective is 28 hr. The casting recovery must be greater than or equal to 88% due to header related problems.

To further reflect the desire to link header performance with production goals and flowtime reduction, the problem statement was recently changed to:

Develop a replacement for the header grade molten metal marinite header (HGMMM) to be used in 5XXX alloy casts which meets production goals. The header life should be sufficient to ensure that less than 10% of all casting terminations are not caused by defects related to the mold package. The header life objective is 28 hr. The casting recovery must be greater than or equal to 88% due to header related problems.

III. SUMMARY OF ACCOMPLISHMENTS

The following data summarizes the accomplishments of the HDC Header Materials Development Team in developing and implementing a replacement for HGMMM.

- The average HDC mold life on 5XXX series alloy castings has increased from a 1991 BNZ MetALform header average of 12.4 hr to a 1993 (January-June) average of 24.0 hr with Pabco PMTHG2.5 headers - a 94% increase.
- Average HDC recovery on 5XXX series alloy castings has increased from 83.9% in 1991 using MetALform headers to 88.0% in 1993 (January-June) using PMTHG2.5 headers.
- The percentage of molds shut down for nonheader related reasons (3XXX and 5XXX series alloys) has increased from 49% in 1992 to 64.2% in 1993 correlating with the 100% conversion to Pabco materials on all 3XXX and 5XXX series alloy casts. In addition to a higher quality header and feedboard material, flowtime considerations would drive this percentage.
- A complete conversion from the BNZ MetALform material was accomplished in 1993 January. During 1991 and 1992, a considerable amount of resources (dollars, time, utilization, recovery, etc.) were expended while utilizing BNZ MetALform materials for HDC headers and feedboards.
- Average HDC mold life on 3XXX series alloy castings has increased from a 1991 average of 41.7 hr to a 1993 average of 52.0 hr. Recovery has also increased from 86.3% in 1991 to 92.0% in 1993.
- A direct comparison of 3XXX series alloy mold life during 1992 shows that the Pabco PMT material has an average life 30% higher than the BNZ MetALform material - PMT average of 56.8 hr versus MetALform average of 43.8 hr. Also, the 3XXX series alloy cast with PMT headers had a higher recovery than those with MetALform headers - 92.5% versus 91.9%.

IV. PROGRAM RESULTS - SIGNIFICANT EVENTS

This section describes the significant events that led to the implementation of Pabco PMT and PMTHGX.X materials for feedboards and headers in the mold packages for Warrick's HDC casting. The events are documented in chronological order. Only the highlights of these activities are provided; references which present more detailed descriptions are provided.

1991 February-March: R. A. Marra published (1991-03-07) an ATC data letter "Header / Metal Surface Reactions - Magnesium Reduction" which postulated the important role of header surface oxide component reduction on the header life and ingot quality during HDC casting of 5XXX series aluminum alloys. It was determined that magnesium, a highly reducing element, will reduce both calcium silicate and iron oxide components. If iron oxide is present, it should be preferentially reduced, thereby preventing (or, at least, delaying) the detrimental reduction of the calcium silicate bond phase. HGMMM contains a large amount of amosite-type asbestos fibers ($\text{MgFe}_6[(\text{OH})\text{Si}_4\text{O}_{11}]_2$) which have a high content of iron oxide. This may contribute to the success in using this material as a header for HDC casting. Based on these results, it was decided to evaluate calcium silicate boards with additives to control the board chemistry and reduction reactions.

1991 April: Based on their laboratory capabilities and willingness to develop new material compositions, Pabco was selected as the preferred company to work with on developing modified calcium silicate boards with iron-containing additives. During a 1991-03-21 visit to Pabco [Ref: Warrick Memorandum: D. C. Sikora to HDC Marinite Replacement and Process Improvement Team, "Progress Report - 1991-03-16 to 1991-03-22"], Alcoa discussed the desire for developing these modified materials. It was determined that boards containing vermiculite, red mud, iron oxide (FeO), and combinations of these materials would be evaluated for potential use as headers for 5XXX series alloy casting.

1991 May-July: Pabco performed lab trials to determine that levels of additives which could be processed into a calcium silicate board product. Coupons of these materials were evaluated for molten metal resistance at ATC. Based on these trials, a materials screening DOX was developed to evaluate the potential substitutes for HGMMM and BNZ MetALform. The materials consisted of:

- Pabco modified PMT with 13% vermiculite;
- Pabco modified PMT with 2% iron oxide (FeO);

- Pabco modified PMT with 10% red mud;
- Pabco modified PMT with 5% vermiculite + 5% red mud;
- Pabco modified PMT with 10% vermiculite + 2% iron oxide (FeO);
- BNZ MetALform as a control; and
- Header grade molten metal marinite (HGMMM) as a control.

The screening experiment was designed to generate header material specimens for analytical examination that have had similar molten metal contact in a high magnesium alloy casting process (800 ingot inches or 3 hr cast time of alloy 5082 on HDC strand 1A). Each material was tested in three different casts. The test was designed such that the machining, mold shop, and casting variables were blocked. [Ref: Warrick Memorandum: M. J. Dunlay to G. R. Swanberg, "Screening of Marinite Replacement (Header) Materials," 1991-07-12]. Materials testing was performed on all materials before and after casting at several mold positions. The material testing included approximately 25 elemental, structural, and physical analytical techniques performed at ATC, Warrick, Pabco, and an outside laboratory [Refs: Warrick Memoranda: M. J. Dunlay to G. R. Swanberg, "Update on Testing of Board Materials used in Header Screening Evaluation," 1991-06-20, M. C. Lukens to M. J. Dunlay, "Testing of Header Materials," 1991-07-23 and M. C. Lukens to M. J. Dunlay, "Testing of Header Materials-Revised," 1991-11-13].

1991 November: The material testing and statistical analysis of the 1991 July materials screening tests were completed. The following material criterion measures were found to be significant:

- Cold crushing strength;
- Modulus of rupture;
- Thermal expansion (perpendicular to the pressing direction);
- Leco carbon analysis at mold positions 2, 5, and at positions relative to the oil ring;
- Oil absorption on unused materials and correlation of oil absorption and Leco carbon analyses; and
- SEM with EDAX analysis of magnesium, aluminum, iron, calcium, and silicon on used header surface.

Based on these results, the calcium silicate board material with 5% vermiculite and 5% red mud additions was selected to be used for confirmation evaluations.

1991 November-1992 January: Experiments were conducted to qualify PMT feedboards and PMT headers for 3XXX series alloy casting. During these tests, problems were observed with header crushing at the area in contact with the oil ring. Separate testing using pressure loading paper and strain bolts confirmed the need for redesign of the mold clamp design [Ref: Warrick Memorandum: W. Lansdale to M. J. Dunlay, "Feedboard - Header Clamp Evaluations," 1991-11-26]. A new clamp was designed which provided a more uniform distribution of the compressive forces. Experiments with the old clamp and new clamp showed that 80% of the explained variability (55%) was a two way interaction between the clamp and material, [Refs: Warrick Memoranda: W. Lansdale to M. J. Dunlay, "Feedboard / Header Material and Header Clamp Evaluation Results," 1992-01-09 and W. Lansdale to J. A. Bloomer and T. D. Plassmeier, "Feedboard/Header Material and Header Clamp Confirmation DOX," 1992-01-13]. Based on these findings, the clamp design and feedboard type were incorporated in the confirmation trial of the Pabco 5% vermiculite + 5% red mud (PVRM) material on 43" 5082 castings [Ref: Warrick Memorandum: M. J. Dunlay to G. R. Swanberg and P. D. Thomas, "Upcoming Marinite Replacement Materials Experiments," 1992-01-17].

1992 January: The PVRM confirmation trial was completed during the week of 1991-01-20. This trial evaluated seven possible combinations of header, feedboard, and clamp design. A total of 11 casts of 43" 5082 alloys were completed on HDC Complex No. 5. The data obtained strongly supported the conclusion that an alternative mold assembly package utilizing headers machined from Pabco calcium silicate boards with 5% vermiculite and 5% red mud additions, Pabco Metaltemp feedboards and the new clamp design results in superior mold life as compared to the mold package comprised of BNZ MetALform headers, BNZ MetALform feedboards, and the old clamp design. [Ref: Warrick Memorandum: M. J. Dunlay, W. Lansdale, M. C. Lukens, B. W. Siebe, and R. A. Marra to G. R. Swanberg and P. D. Thomas, "Marinite Replacement Material Screen (1992-01-20) Update," 1992-01-29]. Casts using the alternative mold package resulted in two to three times the life of the typical mold assembly averages. Other findings of the trial included:

- A mold assembly combination of PVRM header, MetALform (MF) feedboards, and new clamp design may also provide a competitive mold package for casting of high magnesium alloys. The potential of using both PMT and MF feedboards would offer the long term advantage in supplier / inventory flexibility.
- No mold assembly combination using MF headers resulted in an improvement over the historical MF header mold life of 12 hr.

- The thermal dimension changes (material thermal expansion) have a significant impact on ingot quality and therefore on mold life. Ingots cast with the PVRM header showed a more prominent (improved) lap pattern as casting time progressed.
- More than 24 of the ingots cast with the PVRM header were successfully hot rolled.

1992 March: The 1992 January trial highlighted the importance of not cracking or crushing the feedboard during clamping. Based on these results, new clamps were ordered for all mold sizes.

1992 January-June: During the first half of 1992, Pabco encountered difficulties in producing boards with the 5% vermiculite and 5% red mud additions. The problems appeared to be related to difficulties in dewatering and drying the boards as the red mud level was increased. Alcoa provided assistance to Pabco in characterizing the red mud to determine the cause of the production problems. [Ref: ATC Letter Report 06-92-54: R. A. Marra to Memorandum, "Pabco Red Mud Characterization," 1992-07-10.] It was found that the red mud raw material (J. R. Goslee Company, Powder Division, R-20 Product) contained large fraction of fine particles. This may effect the pore structure in the board and contribute to the difficulties in dewatering and drying the material during production. Also, the presence of silica, calcite (CaCO_3), and the de-silication product (sodium aluminosilicate) may have an effect on the slurry gelation. Permeability measurements (ASTM Test C-577-87) of boards produced with varying red mud levels showed a decrease in the permeability with increasing red mud content. This supports the postulation that the red mud is effecting the pore structure.

1992 May: Pabco and Alcoa agreed to manufacture boards with red mud levels increasing in 0.5% increments; Warrick would evaluate the casting performance of these materials. During the week of 1992-05-26, Pabco boards containing 5% vermiculite and 0.5% red mud were evaluated for casting performance of 43" 5082 ingots. [Ref: Warrick Memorandum: M. J. Dunlay, W. Lansdale, M. C. Lukens, B. W. Siebe, and R. A. Marra, "Marinite Replacement Materials Evaluation with Pabco Vermiculite (5%) and Red Mud (0.5%) Header Board," 1992-06-04.] The effect of board heat treatment temperature (600°F and 950°F) was also studied during this trial. The results strongly supported the conclusion that a PVRM header with 0.5% red mud additions will provide superior mold life versus that offered by the MetALform materials. This screening trial did not generate casting data which would indicate a concern with the lower level of red mud (0.5%) versus the 1992 January trial with 5.0% red mud levels. However, subsequent materials testing provided data to suggest that higher red mud levels would provide improved performance. This is discussed in more detail in the Section V. There was not a significant difference in casting performance at the different heat treatment temperature. Previous

characterization of PMT materials [Ref: ATC Letter Report No. 06-92-28, R. A. Marra to Memorandum, "Characterization of Pabco Metaltemp," 1992-03-12] showed that higher heat treatment temperatures resulted in increased porosity and oil absorption and decreased strength. Therefore, it was decided that a standard heat treatment of 600°F would be used to produce all production boards.

1992 June: M. J. Dunlay, W. Lansdale, M. C. Lukens, and R. A. Marra visited Pabco in Fruita, Colorado, to conduct a technical review of the marinite replacement project status and observe the manufacturing of additional vermiculite - red mud boards [Ref: Warrick Memorandum: M. J. Dunlay, W. Lansdale, M. C. Lukens, and R. A. Marra, "Pabco Visit of 1992-06-08 to 06-10," 1992-07-15]. The following items were discussed:

- Management / business items;
- Vermiculite and red med formulation issues;
- Vermiculite and red med process issues;
- Pabco fabrication processes; and
- Pabco laboratory and testing capabilities.

The visit was very informative and provided an opportunity to begin a joint effort to resolve PVRM fabrication issues. Management issues that Pabco considers important were identified. The close working relationship that was established between Alcoa and Pabco was a key element to the success of this program.

1992 August: Pabco PMT boards and boards with vermiculite and red mud additions continued to show significant potential for use feedboards and headers. Material specifications [Ref: Warrick Memorandum: M. J. Dunlay to D. C. Sikora, "Pabco Material Specifications," 1992-08-13] were developed to provide consistent quality of production materials.

1992 September: PMT qualification trials (10 boards) were completed and PMT feedboards (used with the new clamp design) were qualified for casting of all alloys and all sizes. [Ref: Warrick Memorandum: W. Lansdale to M. J. Dunlay, "Qualification of Pabco's Metaltemp Material - Feedboard Applications," 1992-06-04.] The PMT header was also qualified for all sizes of 3XXX (low magnesium) alloy castings.

1992 November: Secrecy and purchasing agreements were signed between Pabco and Alcoa for the production and use of boards containing red mud and vermiculite additions. These agreements included 10 year confidentiality and exclusive use clauses. As a measure to protect the confidentiality of this material formulation, a new material designation was adopted. This designation, PMTHGX.X, represents Header Grade Pabco Metaltemp material with XX percent level red mud content. **IT SHOULD BE NOTED THAT THIS FORMULATION IS CONSIDERED HIGHLY PROPRIETARY AND SHOULD NOT BE DISCUSSED OR DISCLOSED WITH ANY NON-ALCOANS WITHOUT THE CONSENT OF WARRICK PERSONNEL.**

1992 June-1993 January: Production quantities of PVRM with 0.5%, 1.0%, 1.5%, 2%, and 2.5% red mud content (5% vermiculite in all materials) were successfully used in the casting of 5XXX series (high magnesium) alloys. Based on the casting results, materials testing, and Pabco's ability consistently to produce board with 2.5% red mud additions, trials to qualify the PVRM material containing 5% vermiculite and 2.5% red mud were completed. In 1993 January, this material was qualified for 5XXX series castings on all ingot sizes.

1993 January to Present: Pabco PMT and PMTHGX.X materials have been used almost exclusively (>90% of casts) for casting of 3XXX and 5XXX series alloys, respectively. As summarized in Section III (Summary of Accomplishments) substantial increases in casting life and ingot recovery have been realized. There have been several 5XXX casts which have exceeded 100 hr and several 3XXX casts which have exceeded 200 hr indicating that significant potential for process improvements exist with these mold packages. [Ref: Warrick Memorandum: M. J. Dunlay, W. Lansdale, M. C. Lukens, R. A. Marra, and B. C. Siebe to G. R. Swanberg and D. C. Ashley, "Pabco Header / Feedboard Update," 1993-07-26.]

V. MOLD PACKAGE UNDERSTANDING

During the course of this program, an improved understanding of the effects of the header material and mold package design on the casting performance has been developed. This understanding has been critical to the success of the program. This section briefly discusses the impact of header material characteristics and mold package design on casting performance.

A. Header Material Physical Characteristics

The physical characteristics of the header material have a large impact on the casting performance of the mold package. This discussion will concentrate on analyses conducted on the Pabco vermiculite + red mud material (PMTHGX.X) and the comparison with Header Grade Molten Metal Marinite, BNZ MetALform, and Pabco Metaltemp. Correlations between the material characteristics and casting performance for other materials were examined in the 1991 July materials screening trial [Ref: Warrick Memorandum: M. J. Dunlay to G. R. Swanberg, "Update on Testing of Board Materials used in Header Screening Evaluation," 1991-06-20 and Warrick Memorandum: M. C. Lukens to M. J. Dunlay, "Testing of Header Materials," 1991-07-23].

PMTHG materials with 5% vermiculite and red mud levels of 0.5% to 5% were characterized with respect to cold crush strength, modulus of rupture, oil absorption (% weight gain in coupons submerged in oil for 3 hr) and thermal expansion. The results are shown in Figures 1, 2, 3 and 4, respectively. Similar analyses on Header Grade Molten Metal Marinite (HGM), BNZ MetALform (MF), and Pabco Metaltemp (MT) are shown for comparison. The following conclusions can be made:

- Red mud additions of 2.0% and higher result in increased cold crushing strength [Ref: Warrick Memorandum: M. C. Lukens to M. J. Dunlay, W. Lansdale, R. A. Marra, and B. W. Siebe, "Cold Crush/M.O.R. Update," 1993-04-22]. The strength of these materials is significantly higher than the BNZ MetALform strength and at least as high as that measured for the marinite material.
- A similar trend was observed in the material's modulus of rupture (MOR) [Ref: Warrick Memorandum: M. C. Lukens to M. J. Dunlay, W. Lansdale, R. A. Marra, and B. W. Siebe, "Cold Crush/M.O.R. Update," 1993-04-22]. Red mud additions of 2.0% and higher resulted in significantly higher strength than PMTHG materials with lower

levels of red mud and the BNZ MetALform material. Molten metal marinite had the highest modulus of rupture although the strength was not significantly higher (at the 0.1 level) than the PMT material or PMTHG materials with red mud contents of 2.0% and higher. The asbestos fibers in the HGMMM material tend to strengthen the material.

- Oil absorption has been shown to have a significant effect on header performance. All of the Pabco materials (PMT and PMTHGX.X) show significantly less oil absorption than the HGMMM and BNZ MetALform materials. PMTHG materials with intermediate level of red mud additions (1.0%, 1.5%, and 2.0%) showed the lowest oil absorption. [Ref: Warrick Memorandum: M. C. Lukens to M. J. Dunlay, W. Lansdale, R. A. Marra, and B. W. Siebe, "Oil Absorption Update," 1993-04-22].
- The header material's thermal expansion characteristics also play an important role in the mold package performance. This is discussed in more detail in Section V.4. Materials with negative expansion coefficient or low positive values of expansion coefficient will maintain the header overhang which results in a more prominent lap pattern. The PMT, MetALform, and PMTHG0.5 have a significantly higher coefficient of thermal expansion than the other materials. [Ref: Warrick Memorandum: M. C. Lukens to M. J. Dunlay, W. Lansdale, R. A. Marra, and B. W. Siebe, "Thermal Expansion Update," 1993-04-31].

It is clear that the performance of the header is related to these physical characteristics. Optimum performance can be obtained by developing a material which has high crushing strength and modulus of rupture, low oil absorption, and a negative or low positive coefficient of thermal expansion. The Pabco PMTHG2.5 material (now qualified and being used almost exclusively for HDC casting of 5XXX alloys) has a favorable combination of these characteristics. The importance of header's chemical composition and its impact on reactivity with the molten metal is discussed in the next section.

B. Molten Metal / Header Chemical Interactions

Chemical surface analysis of used headers along with thermodynamic considerations showed that both magnesium and aluminum (to a lesser extent) will reduce the oxide components in the header board [Ref: ATC Memorandum: R. A. Marra, "Header/Metal Surface Reactions - Magnesium Reduction," 1991-03-07]. Analysis of the marinite material showed the importance of iron oxide in controlling the magnesium reduction reactions. Thermodynamic calculations

showed that magnesium will reduce both calcium silicate and iron oxide. If iron oxide is present, it should be preferentially reduced.

SEM / EDAX analysis confirmed the reduction of the header surface by both magnesium and aluminum. The much higher content of Mg (especially in comparison with the alloy compositions) indicate that the reduction by Mg is much more severe. This is consistent with the observed difficulties in casting 5XXX series alloys. The EDAX analysis suggested that the role of iron oxide in the asbestos containing marinite boards was important in preventing the attack of the calcium silicate bond phase and thus the degradation of the mechanical and physical properties. Since the iron oxide components are discrete phases (can be thought of as fillers), their attack is much less detrimental to the integrity of the board than the attack of the calcium silicate matrix phase. The iron oxide component can be thought of as a sacrificial phase which prevents degradation of the matrix.

A similar analysis was performed on the Pabco PMT header material [Ref: ATC Memorandum: R. A. Marra to Memorandum, "Pabco Metaltemp: Header/Metal Surface Reactions," 1991-05-03]. The PMT material is comprised of a calcium silicate bond phase with wolastonite and alkali resistant glass fiber fillers. There is no detectable iron oxide content in the material. SEM / EDAX analysis of a used header (5182 alloy cast) showed that magnesium and, to a lesser extent, aluminum reduced the calcium silicate bond phase during casting. The wolastonite fibers were quite resistant to chemical attack. Based on these observations, it was recommended that iron oxide-containing additives such as vermiculite, red mud, and iron oxide be added to the PMT material in an attempt to prevent the attack of the bond phase and the deterioration of the material's physical and mechanical properties. Pabco agreed to produce boards with these additives which were evaluated in the 1991 July materials screening test (See Section IV). SEM / EDAX analysis of the headers used during this screening evaluation confirmed the importance of iron oxide in controlling the molten metal/header chemical reactions.

The analysis of a defect (strawberry) on a 5XXX alloy ingot showed that fragments of the header material were present below the ingot surface. This supports the hypothesis that chemical reactions between the metal and the header leads to the attachment (freezing) of metal on the header surface. The solidified metal gets trapped in the solidification front and is pulled out by the moving solidified ingot.

C. Oil Absorption

There is evidence that the amount of oil absorbed in the boards has a significant effect on the generation of ingot defects and thus the header life. The effect is not completely understood although various observations support its importance. The amount of oil absorption is much greater at the bottom and sides of the header than on the top. This was determined quantitatively during the detailed analysis of the header materials used in the 1991 July materials screening evaluation. It is much more common to observe defects on the sides and bottom of the ingots than on the top surface.

In 1991 September, a BNZ MetALform header was used for casting of 5182 alloy. The header life was 38 hr; this is much longer than the typical life (1991 average life of 12.4 hr) of the BNZ material for casting 5XXX alloys. [Ref: Warrick Memorandum: M. C. Lukens to R. A. Marra, "Analysis of BNZ Header # 1109005-1," 1991-10-03]. Visual inspection of the header suggested that oil absorption was extremely low. This was confirmed by Leco carbon analysis of the used header. SEM / EDAX analysis of the used header showed that there was substantial chemical interactions between the molten metal and header in the region near the oil ring (surface cation composition: Mg-8.5 wt%, Al-5.6 wt%, Si-30 wt%, Ca-56 wt%) whereas there was no chemical attack in the region where there was no oil absorption (surface cation composition: Si-30 wt%, Ca-70 wt%). It appears that the oil absorption and the products of the pyrolysis effect the calcium silicate's wetting characteristics thus allowing the molten metal to react with the header.

In 1975, R. LaBar [Ref: ATC Memorandum, R. G. LaBar, "Johns-Manville/Alcoa Meeting, Warrick Operations - 1975-06-17," 1975-07-22] postulated that the oil and oil vapor is intruded into the pore structure of the marinite board and is pyrolyzed to produce carbon, hydrocarbons, hydrogen, carbon dioxide, water vapor, and possibly some fatty acids. These pyrolyzed products can react with the components (iron oxide impurities, free lime, lime-rich silicates, and silica-rich phases) in the header material. The net result of these reactions is a weakening or destruction of the bond phase and an increase in thermal conductivity due to carbon deposition. He suggested that the increased difficulty in casting higher magnesium may not be related to corrosion by the alloy but rather to the additional lubricant required to cast these alloys.

It is obvious that the oil absorption plays an important role in the header degradation. Further studies are needed to optimize the lubrication rates and determine the impact on casting performance.

D. Header Failure Mechanisms

Various header failure mechanisms have been proposed (Ref: R. LaBar, "Johns-Manville / Alcoa Meeting, Warrick Operations - 1975-06-17," 1975-07-22 and ATC Memorandum: M. G. Chu / G. J. Hildeman to R. Bachowski, D. A. Granger, J. E. Jacoby, R. A. Marra, R. J. Milauskas, and R. A. Ramser, "Proposed Mechanisms for HDC Header Failure and Coarse Dendritic Structure Formation", 1991-05-22). As discussed above LaBar emphasized the important role of oil absorption and pyrolysis. The work by Marra, see Section V.1.b, showed the importance of magnesium reduction of the calcium silicate bond phase.

A report by M. Kulak [Ref: ATC Report 52-85-09, M. Kulak, "The Prediction of Temperatures and Stresses in Molten Metal Marinite Header During HDC Casting at Warrick," 1985] described the development of finite element models to predict the temperature and stresses in the molten metal marinite material during casting. The results agreed well with experimental temperatures and strains measured during an actual production run. It was concluded that: "the failure of the MMM header cannot be explained solely on the stresses predicted by this analysis. This is consistent with the life expectancy of the header, which may vary anywhere from hours to days. The life of the header is dependent on maintaining the integrity at the corner overhang region. Failure in this region is related to both the thermomechanical stresses and the material degradation with time because of oil vapor penetration and chemical attack at the hot face. Any clamping damage in this critical overhang region will accelerate failure."

The proposed failure mechanisms can be classified into three primary categories: physical / mechanical failure, chemical attack, and failure resulting from thermal profile changes.

Physical failure is believed to occur when excessive oil soaks into the header and weakens the header to an extent that it cannot withstand the thermal stress caused by the nonuniform thermal field. Fragments of the header break away from the header overhang region. Molten aluminum freezes and sticks on the back side of the oil ring which results in tears and strawberries on the bottom and side surfaces of the ingot.

Chemical failure results from chemical reactions of the magnesium and, to a lesser extent, aluminum with the calcium silicate bond phase in the header. This reaction gradually corrodes the header behind the oil ring. Molten aluminum freezes and sticks on the back surface of the oil ring or corroded header which results in tears and strawberries in the cast ingot.

Thermal failure results from excessive oil absorption into the header. The oil pyrolyzes leaving a carbonaceous deposit in the header which increases the thermal conductivity. Molten aluminum freezes and sticks on the header due to a lower surface temperature. This results in tears and strawberries on the cast ingot.

It is likely that all of the proposed mechanisms play some role in the header failure. A hypothesis for the failure mechanism was proposed in 1991 November (Ref: ATC Memorandum: M. G. Chu to G. R. Swanberg, "Possible Mechanism for HDC Header Failure," 1991-11-04). This hypothesis was developed based on the results of analyses of the content and distribution of carbon (oil and pyrolysis products) in the used headers, chemical composition at the header surface, and analysis of the subsurface structure and chemical composition of the ingot defects. It was proposed that as excessive oil soaks into the header, the oil pyrolyzes and changes the molten metal wetting characteristics of the calcium silicate material. The header is wetted by the molten metal and chemical reactions between the metal and header components occur. The header surface is corroded and the material is structurally weakened. Defects (microcracks, voids, surface roughening) form on the header near the header / oil ring overhang. The metal begins to freeze and stick in the defective regions. The solidified metal gets trapped in the solidification front and is pulled out by the moving solidified ingot. This results in the commonly observed strawberries, tears and surface cracks. It is possible that fragments of the header are also pulled out by the moving ingot. This results in larger surface defects and ultimately to the failure of the header.

E. Header Thermal Expansion: Effect on Mold Package Clamping and Header Overhang

The thermal expansion of the header material during casting can result in crushing the header in the mold package clamping region. This problem was more prevalent when dissimilar materials are used for the header and feedboard or when stronger materials such as the PMT board are used. A new clamp was designed which distributes the clamping forces more evenly on the header surface. This clamp also results in a closer alignment of the center points of loading between the clamp / feedboard contact surfaces and the oil ring / header contact surfaces. This clamp design resulted in the ability to utilize the stronger Pabco PMT and PMTHG materials by eliminating crack initiation sites and the crushing at the header / oil ring interface.

The thermal expansion of the board material in the direction perpendicular to the pressing direction (during board production) is important since it effects the header / oil ring overhang. A low positive or negative thermal expansion coefficient results in the retention of the overhang as casting progresses. This behavior was observed with marinite and PVRM (PMTHG) headers. It is possible that the slight negative thermal expansion of these materials results in an increase in overhang due to material shrinkage and expansion of the metal components in the mold package. It was noted that the BNZ MetALform material which had a high positive expansion coefficient had the lowest level of lap pattern and the PVRM (PMTHG) compared most favorably with marinite which had the most prominent lap pattern. The lap pattern has been shown to correlate with increased mold life and recovery.

VI. FUTURE IMPROVEMENTS (CURRENT, PLANNED, AND PROPOSED ACTIVITIES)

A. Casting Process Optimization

PMTHG2.5 has been qualified as a marinite replacement material for HDC casting of 5XXX series alloys. Further improvement in mold life and ingot recovery may be obtained through optimization of the casting process parameters. As discussed in Section V, it is believed that the lubrication rate plays an important role in the casting process. The casting speed and water mold spray are also believed to have a significant impact on the casting performance. A DOX has been designed to evaluate the impact of these casting variable on the PMTHG2.5 mold life and recovery. The results of this trial will be used to optimize the HDC process parameters. The following matrix shows the proposed experimental design:

	<u>Low (-)</u>	<u>Midpoint</u>	<u>High (+)</u>
Oil Flow (ml/min)	1	2.5	4
H ₂ O Mold Spray (g/min)	300	350	400
Speed (in./min)	4.75	5.0	5.25

The experiment will be designed to block the following variables: mold package, casting strand, temperature, bottom sprays, alloy type (43" 5082), filtration, skimming, metal head, start-up procedures, and people. The measurements will include casting life, recovery, and rollability.

B. Materials Development

1. Red Mud Level

While the PMTHG2.5 material is providing acceptable casting performance, it is not known what the optimum level of red mud should be. Pabco has fabricated the following tests boards:

- Pabco Metaltemp with 5% vermiculite and 2.5% red mud;
- Pabco Metaltemp with 5% vermiculite and 5.0% red mud; and
- Pabco Metaltemp with 5% vermiculite and 8.0% red mud.

ATC has conducted material evaluation tests (density, crushing strength, TGA, thermal expansion, and oil absorption). The results showed that these materials had similar cold crushing strengths and densities. There was a slight reduction in oil absorption with increasing red mud content. Thermal analyses showed the materials had similar weight losses and small negative expansions from RT to 700°C. HDC casting trials will be performed on materials which show the promise (based on material evaluation and comparison with existing materials database) for improved casting performance.

2. Calcium Silicate Board: Xonotlite Structure

The primary phases in the Pabco PMT and PMTHG boards is the tobermorite and wolastonite forms of calcium silicate. The wolastonite is added to increase the hardness and strength of the board; it is a very stable form of calcium silicate. Tobermorite forms the board's matrix phase. This phase is a less stable form of calcium silicate. The xonotlite form of calcium silicate is a more stable phase and is expected to be more resistant to attack by the high magnesium alloys. This material may also have expansion characteristics that are closer to the molten metal marine material. Pabco has agreed to produce boards with the xonotlite phase with the additives (5% vermiculite and 2.5% red mud) in the present PMTHG2.5 material. The initial production run was unacceptable due to a high amount of white spots (unreacted lime). Material characterization and casting performance trials on these boards will be conducted.

3. Red Mud / Vermiculite Ratio

The vermiculite content has remained constant (5%) in all of the PMTHG boards that have been evaluated. The effects of varying the vermiculite content and the red mud / vermiculite ratio on the material characteristics and casting performance should be examined.

C. Feedboard Cracking

Feedboard cracking during and after machining has been an significant issue with the BNZ MetALform material. Although this has been less of a problem with the Pabco PMT material, some cracking was observed in this material during 1992 December and 1993 January, [Ref: Warrick Memorandum: M. J. Dunlay to Memorandum, "PMT Feedboard Cracking," 1993-01-15]. Tests were conducted at Warrick and ATC to determine the effects of water absorption (humidity changes) on the board's weight gain and dimensional stability. It was concluded that the PMT material required about a 7 day time period to stabilize to different

humidity / temperature conditions [Ref: Warrick Memorandum: W. Lansdale to M. J. Dunlay, "HDC Feedboard Cracking (Metaltemp) Update," 1993-02-12]. It was also found that the cracking could be reduced by decreasing the machining feed rate by 30%. Pabco will continue to investigate their manufacturing process in an effort to determine the root cause of the cracking problem. There was been extensive works at Apex to develop an in control and capable machining process. The specific activities include:

- Establishment of control charts on critical dimensions;
- Resolving issues with the machine manufacturer on laser calibration of arms, board tuning to appropriate frequencies, establishment of circle-diamond-square test, etc.;
- Program changes and adjustments of machine settings to improve "out-of-control" corner conditions;
- A 43" mold template has been supplied to Apex by Alcoa which will be used as a standard for the establishment of a tracking system;
- Testing to determine if improved machining accuracies can be obtained by using a "coordinate" based program instead of the "incremental" based program;
- Continuing to develop measurement capability; and
- Establishing SOP's for all header, feedboards, and key sizes.

D. Header Overhang

The header-oil ring overhang plays an important role in the casting process. It has been proposed to further examine the effects of the overhang dimension on the casting performance.

E. Mold Shop Consistency

Programs recently initiated in the mold shop have had a positive impact on the consistency of mold packages delivered to the casting complex. These include tracking mold inside diameters, input actual data versus average data, and performing audits of the mold buildup process.

VII. CONCLUSIONS

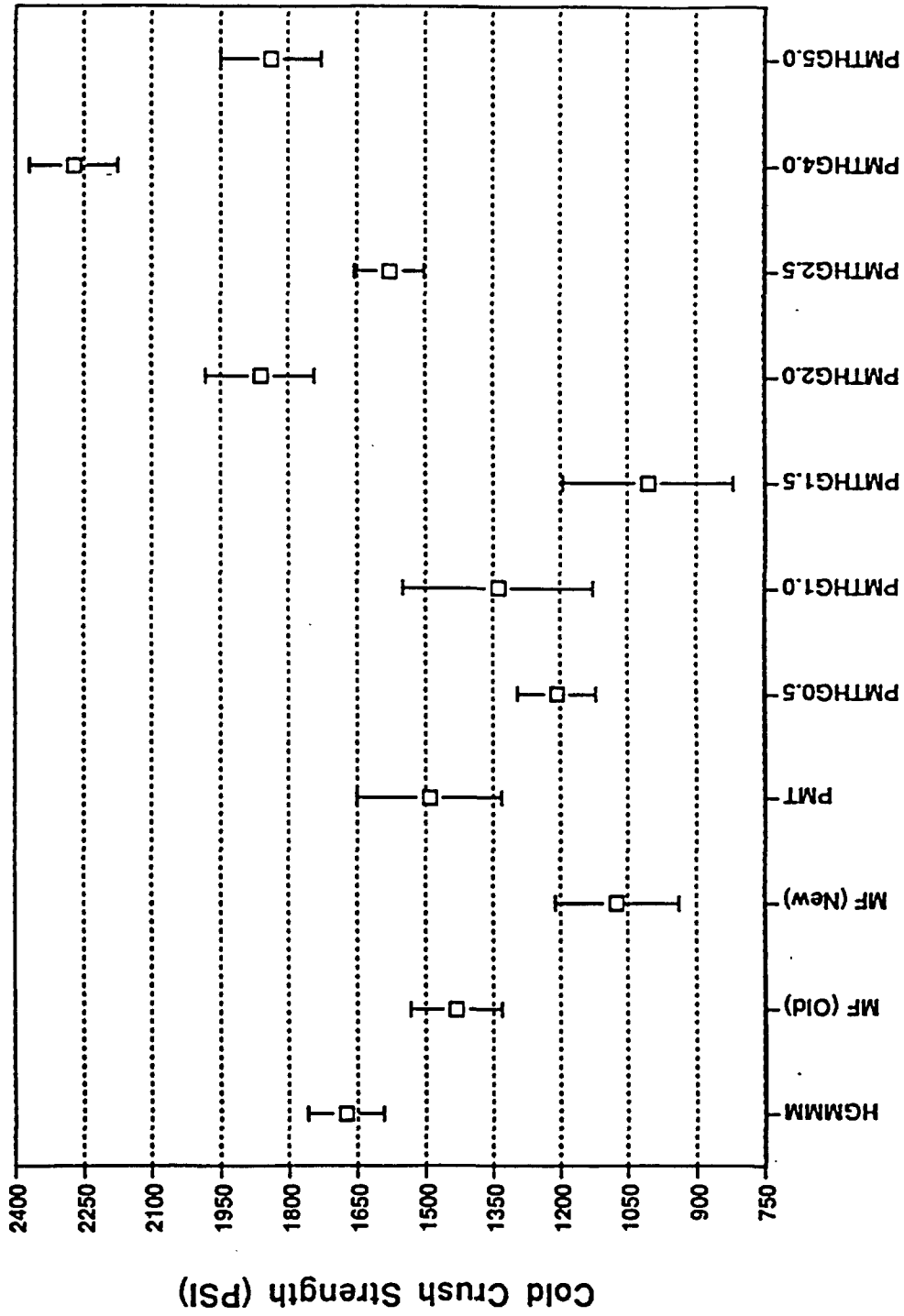
A replacement material for header grade molten metal marinite has been developed, evaluated, and qualified for the HDC casting of 5XXX alloys. The key elements and activities which contributed to the success of the program were:

- Warrick / ATC Teaming: An excellent cooperative team was established with ATC and Warrick personnel.
- Well Defined Project Objective: A technical objective which was consistent with Warrick production requirements was established to provide a measure of project success.
- Analysis of Header Materials: The use of analytical techniques to determine the header degradation mechanisms and role of the header material chemistry on casting performance led to the development of potential material formulations for the replacement material.
- Close Vendor Working Relationship: An excellent working relationship was established with Pabco. The program would not have been successful without the commitment of Pabco to assist in the development of the replacement material.
- Materials Screening: With the cooperation of Warrick's ingot production, a materials screening test was performed on the potential replacement materials. Through the combined efforts of Warrick and ATC personnel, the analysis of the materials screening test led to the selection of the PMTHG material.
- Process Modification / Optimization: The development of standard operating procedures by Warrick personnel greatly aided the evaluation of the potential replacement materials. The understanding of the mold package functionality which led to process modifications such as a redesigned clamp was critical to the success of the program.
- Experimental Design and Statistical Analysis: The use of designed experiments and statistical analysis provided the basis for downselecting the potential material formulations. Statistical methods were further utilized to optimize the material formulation, mold package design, and casting process variables. The systematic approach used to evaluate and qualify the replacement materials provided confidence that the materials could be successfully implemented in production.

While the PMTHG material has been qualified as a header for the casting of 5XXX alloys, material refinements and HDC process optimization may lead to further improvements in casting life, ingot recovery, and ingot quality. Planned and proposed activities include:

- Optimization of the red mud content;
- Optimization of the red mud / vermiculite ratio;
- Evaluation of boards with xonotlite as the matrix phase;
- Optimization of HDC process variables (lubrication rate, mold spray, casting speed);
- Evaluation of the effect of header overhang on casting performance; and
- Resolution of the feedboard cracking problems.

Cold Crushing Strength
95 % Confidence for Mean



Material

Figure 1 Header material cold crushing strength.

Modulus of Rupture
95% Confidence for Mean

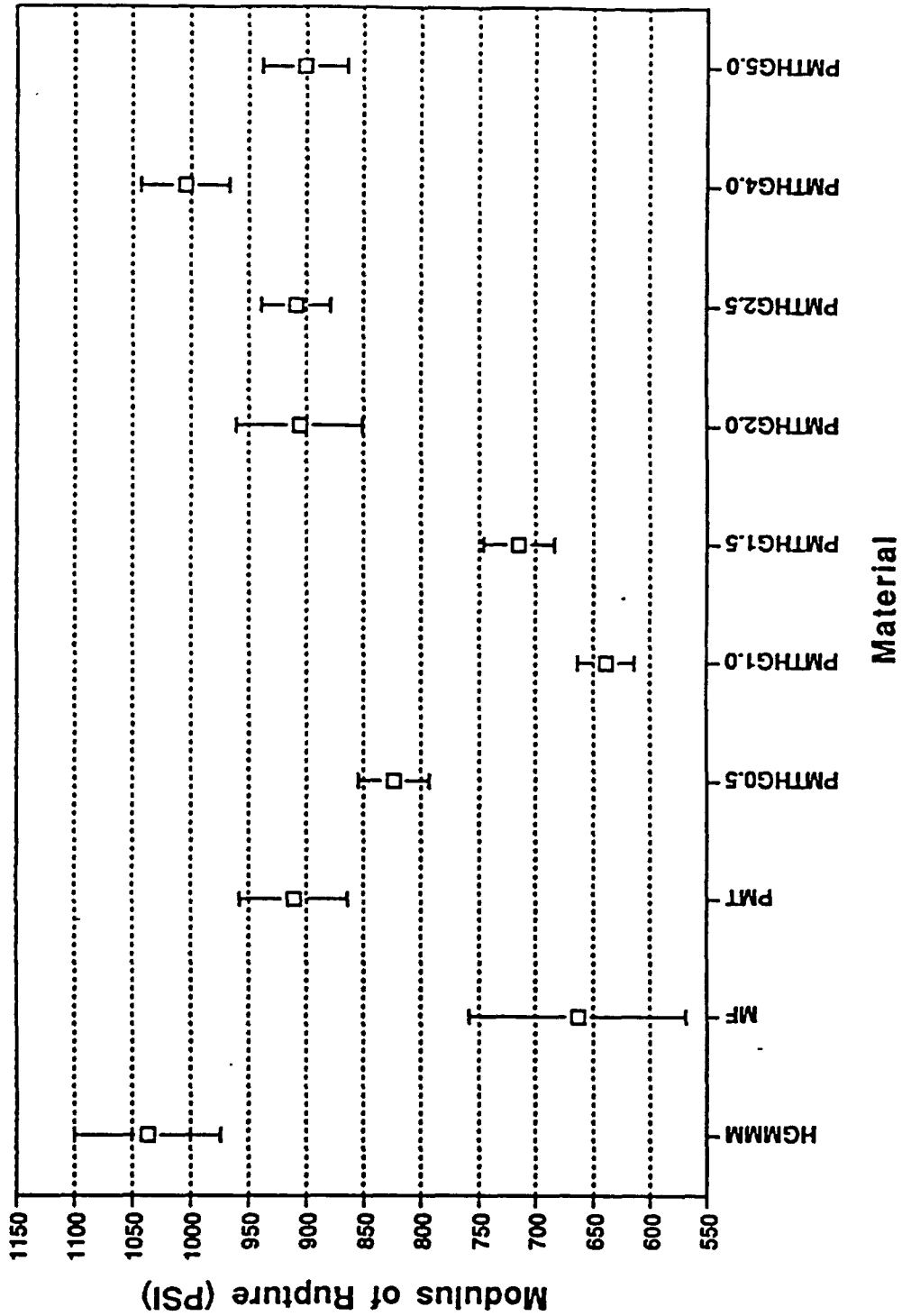


Figure 2 Header material modulus of rupture.

Oil Absorption (3 hours)
95% Confidence for Mean

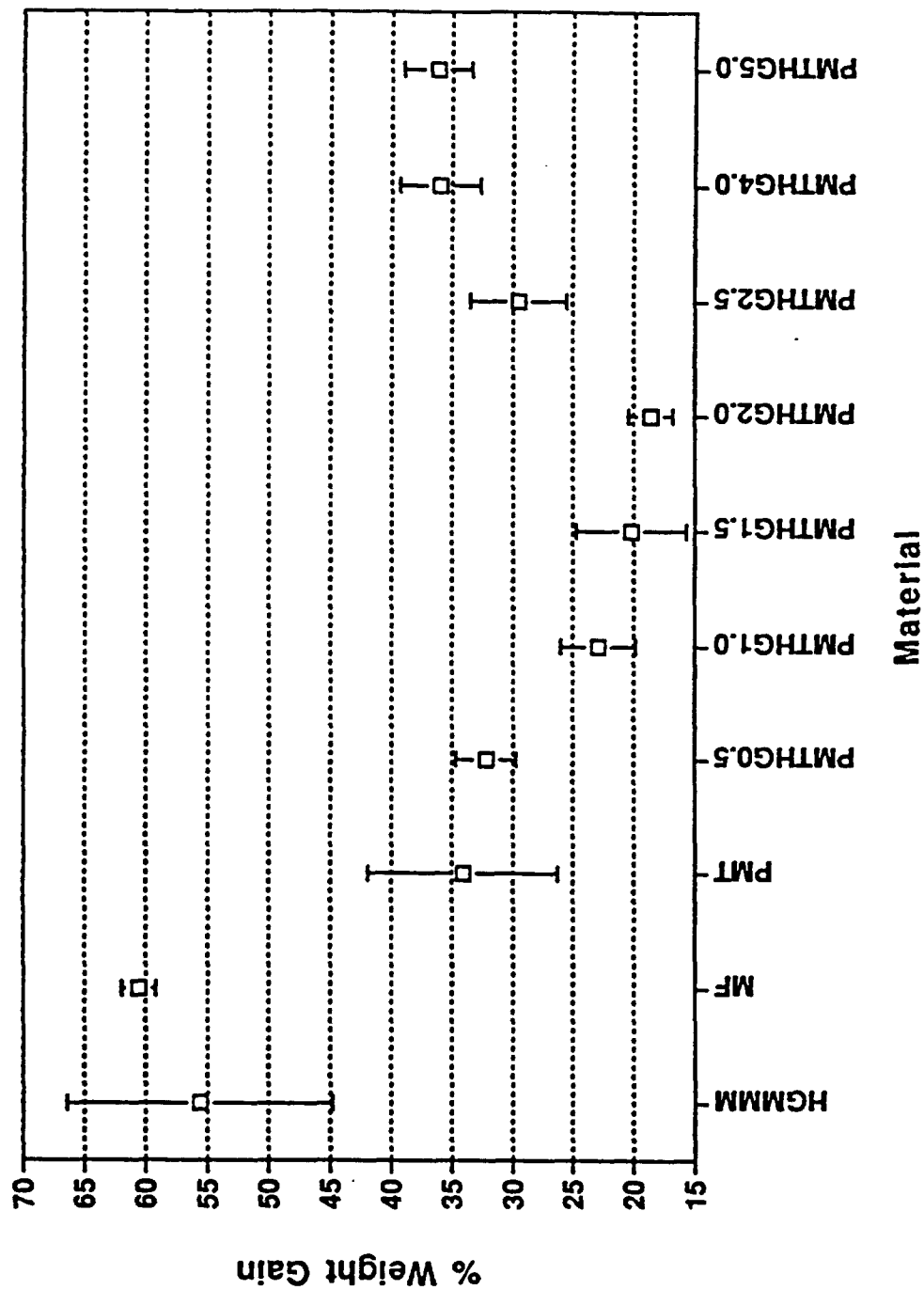


Figure 3 Header material oil absorption.

Thermal Expansion to 700 °C
95% Confidence for Mean

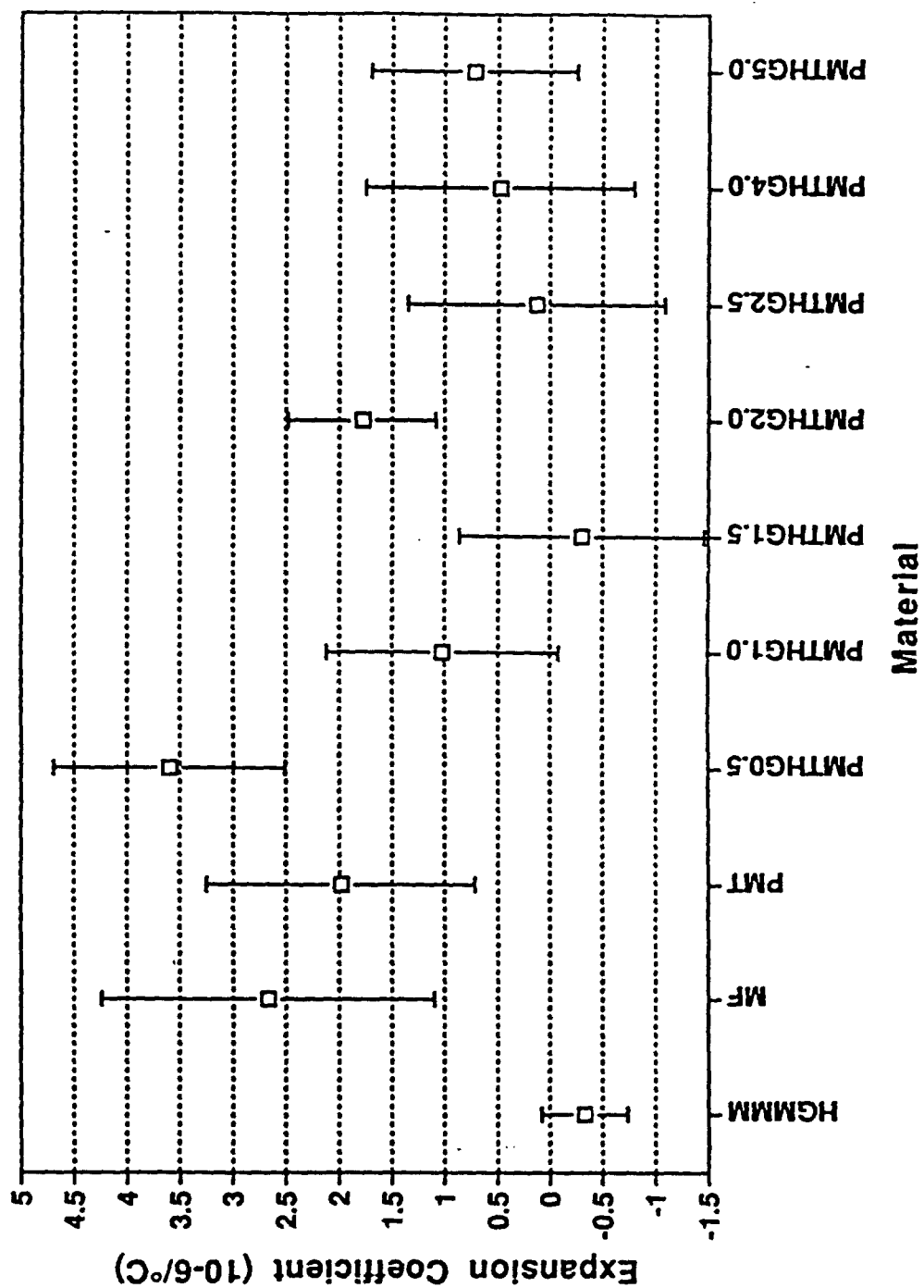


Figure 4 Header material thermal expansion.

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EVALUATION OF CANDIDATE MATERIALS
FOR HDC INGOT CASTING HEADER APPLICATION
AT WARRICK OPERATIONS

By

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1985 August 27

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Executive Abstract

This report summarizes a characterization program intended to improve understanding and develop a data base for selection of new header materials for the HDC casting process. These efforts have been guided towards determining key properties of currently used asbestos containing Molten Metal Marinite, and in conjunction with using finite element analysis techniques to define regions and magnitudes of stress in the header, provide rationales for selection of substitute materials. These inputs were shared on a routine basis with Warrick Operations personnel to insure that testing and design study results were of immediate value and then trial results at Warrick used as a feedback to guide further testing and analysis.

To date, key properties and design parameters have been successfully identified and two new calcium silicate bonded candidate materials recommended for field trials. Future ATC technical service activities will include limited testing or test recommendations, post mortem analysis and coordination with HDC operations personnel, suppliers, Purchasing and Engineering. Active participation in the HDC header task force activities will continue until substitute materials are successfully introduced into the HDC processes.

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1.0 INTRODUCTION

During 1984, a decline in the quality and threatened availability of asbestos containing Molten Metal Marinite (trade name for Manville Corporation's calcium silicate board) used for HDC headers prompted a renewed ATC program^{*1-6} to evaluate new materials for that application. The primary goals of this program were to initially perform materials characterization and design studies to understand how Molten Metal Marinite functions successfully, then to characterize alternate materials and conduct design and field studies to fit them into the HDC process. Hopefully this will lead to successfully finding more than one non-asbestos substitute to minimize sole-source dependency on one header material supply. Additional benefits of this program are the potential applications of these new materials for headers in HDC and FDC casting processes at other plants and for improved headers used in FDC casting aluminum- lithium alloys. If acceptable for the more rigorous header requirements, candidate materials could also qualify for use in molten metal containment applications such as troughs, floats, basin linings, and dams.

This report describes the materials testing program that has been underway during 1984-1985 and its relationship to the design studies performed on the instrumented header/assembly at Warrick.^{*7,8} The understanding of how header grade Molten Metal Marinite performs in this application can then be applied to more effectively substitute alternate materials. Many discussions with Warrick personnel and field observations conducted along with ATC studies have resulted in a checklist of important parameters that must be considered in relation to specific header requirements.⁹ These parameters are:

* See references.

1. Available in reproducible large board sheets which are free of warpage, cracks, voids and delaminations. The internal structure of the material must also be homogeneous without excessive concentration of reinforcing materials or soft zones (white spots).
2. Good machinability to form a smooth, defect-free, as-machined surface having acceptable chip and crack resistance.
3. Adequate strength for handling and installation.
4. Adequate strain capability to deflect when mating with the mold. Through-thickness compressive strength must be capable of withstanding clamping loads.
5. Minimum permeability to, and solubility in casting lubricants.
6. Acceptably low thermal conductivity and thermal diffusivity to minimize chill during start-up.
7. Acceptable thermal shock resistance. Acceptable resistance to cracking and chipping in the overhang region.
8. Relatively predictable and stable long-term dilation behavior (low permanent shrinkage in all directions) as a result of elevated temperature exposure. Low thermal expansion coefficient to minimize stresses.

9. No harmful outgassing when contacted by molten metal.
10. Adequate corrosion resistance and nonsticking in magnesium containing molten metal.

By combining efforts with Warrick personnel, all of these parameters have been addressed. The materials specification for MMM-HG,*⁹ given in Table I, addresses a few of the aforementioned parameters, but needs more elaboration from an engineering viewpoint. Therefore, it is also a major goal of this work to produce more complete guidelines for HDC header materials.

2.0 MATERIALS DESCRIPTION

The following materials have been included in our evaluation program.

<u>Supplier/Material</u>	<u>Description</u>
Manville/Molten Metal Marinite - Regular Grade (MMM-RG)	Asbestiform fibers (amosite) bonded with a hydrated calcium silicate phase (amorphous tobermorite).

(continued on next page)

* See references

List of header materials evaluated - continued -

Supplier/Material	Description
Manville/Molten Metal Marinite - Header Grade (MMM-HG)	Increased fiber content over MMM-RG bonded with the same hydrated calcium silicate chemistry. Heat treated at 1100°F (593°C).
Manville/Metal Mover (Old)	Alkali resistant glass and wollastonite (CaSiO_3) fibers bonded with a hydrated calcium silicate phase (amorphous tobermorite). Heat treated at 975°F (524°C).
Manville/Metal Mover (New) (A4.3-1 and A3.1-4)	Alkali resistant glass and wollastonite (CaSiO_3) fibers bonded with a hydrated calcium silicate phase (amorphous tobermorite). Heat treated at 975°F.
Manville/XM-4 and XM-6	Alkali resistant glass and wollastonite fibers bonded with a hydrated calcium silicate phase (amorphous xonotlite). Heat treated at 975°F.

(continued on next page)

List of header materials evaluated - continued -

<u>Supplier/Material</u>	<u>Description</u>
Nichias/Pyrotek/N-14	Wollastonite and E-glass fibers bonded with an amorphous hydrated calcium silicate binder (xonotlite). Not heat treated.
Nichias/Pyrotek/L-16, L-17, L-18, and KX	Wollastonite and carbon fibers bonded with an amorphous hydrated calcium silicate binder (xonotlite). Density is decreased from L-16 to L-17 to L-18 to KX. Not heat treated.
Pabco/32	Fiberglass bonded with an amorphous hydrated calcium silicate (xonotlite), bentonite and Portland cement. Autoclaved at 400°F (204°C). Not heat treated.
Alcoa Technical Products Division/ Permtech Sigma (R-680)	Calcium aluminate cement bonded fused silica castable with a nonwetting additive. Fired at ~1500°F (816°C).

The reinforcement phase of Molten Metal Marinite (MMM) consists of amosite fibers which are chain-silicates (amphiboles)*¹⁰ of the composition $(\text{Mg}, \text{Fe}^{+3})_7 \text{Si}_8 \text{O}_{22} (\text{OH})_2$ (more correctly, these fibers can be designated Cummingtonite which is the magnesium-rich composition). Amosite fibers are basically ash-grey in color, with a coating of iron oxides that give the fibers a reddish-brown color after the board is heat treated at 975°F. The hydrated calcium silicate binder phase is not composed of fully crystalline tobermorite*¹⁰ (plate crystals of the composition $5-6\text{CaO} \cdot 6\text{SiO}_2 \cdot 7 \cdot 5\text{H}_2\text{O}$) but an amorphous gel phase $(1.5-2.0\text{CaO} \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O})$ *^{11,12}. MMM is formed by filter-pressing an aqueous slurry of asbestos fibers, lime, and silica and then autoclaving at 100 psig/330°F (166°C) to form the pseudo tobermorite hydrated calcium silicate phase*¹³ which rigidizes the board. The asbestos fibers, if too short, make the resultant board excessively brittle. Long asbestos fibers improve board toughness but result in homogeneity problems (laminations) if fiber length becomes excessive. Asbestos fibers in excessively large, unbroken bundles (crudy fiber or pencils) are considered defects. The ability of asbestos bundles to be broken down into minute 1 μm fibers gives the soft, as-formed board acceptable durability and after autoclaving, develops a lightweight, tough, machinable material. Post-drying*¹⁴ or heat-treating at 1100°F (593°C) is performed to remove excess water. Because the amosite fibers dehydrate at 1200°-°1355°F (650°-735°C), they impart reasonable elevated temperature stability to the board over a wide range of temperatures. Most of the shrinkage noted in MMM board products at temperatures above 1400°F (760°C) is attributed to substantial dehydration of tobermorite gels. Xonotlite-type calcium silicate

* See references

binders tend to shrink less because of the lower amount of hydrated water present in the binder structure ($5\text{CaO} \cdot 5\text{SiO}_2 \cdot \text{H}_2\text{O}$). A number of reinforcement materials have been considered as substitutes for amosite; for example, E-glass, mica, cellulose (kraft), alumino-silicate ceramic, and wollastonite (calcium silicate) fibers. None of these materials either by themselves or combined, when mixed with hydrated calcium silicate binder would have the same combined green and fired strength, fired toughness, and homogeneity as MMM for header applications. The highly anisotropic properties of these board materials are related to the preferred orientation of the longitudinal axes of high-aspect ratio (length to diameter) fibers or fiber bundles parallel to the flat board surfaces. Therefore, special attention to a candidate material's anisotropy was given during the sample preparation, testing, design analysis and evaluation of data.

3.0 TEST DESCRIPTION

The various characterization and evaluation tests and rationales for using them were previously described.*^{4,5,6} An update of these tests is given below:

<u>Test Parameters</u>	<u>Rationales</u>
o Density, Density Gradient, and Durometer (Type D)	Correlate density and material homogeneity with firmness (Durometer). Density may also be correlated with heat transfer properties and strength.

(continued on next page)

Test Parameters	Rationales
o Modulus of Rupture, Strain to Failure and Modulus of Elasticity at Room and Elevated Temperatures (1000°F-538°C)	Determine strength levels at room temperature and at elevated temperature using a three-point bending method. Modulus of elasticity may be used to calculate maximum deflection allowable for clamping headers onto the mold package.
o Compressive Load Relaxation Test at Room Temperature, 1100°F (593°C) and 1400°F (760°C)	Determine the extent of stress relief for different materials obtained when the header is clamped (prestressed) on the mold and is heated during the process.
o Overhanging Ledge Compressive Load Test at Room Temperature	Investigate the chipping and cracking resistance of as-received and treated materials by simulating the stress-riser effect in the header's overhanging lip region. Treated specimens were subjected to oil soaking and coking at 1100° and

(continued on next page)

Test Parameters	Rationales
	1400°F. $MgCl_2$ was added to the coke to determine the effects of Mg or Cl_2 on header strength.
o Modified "B" Immersion in 5182 at 1400°F (760°C)	Determine wetting, corrosion resistance, dimensional stability, and crack resistance of candidate materials. This is employed as an initial screening test for unknown specimens.
o Dilation Measurements as a Function of Increasing Temperature	Determine the thermal stability of specimens as a function of anisotropy (measured parallel and normal to the board-plane).
o Hot Oil Tests and Coking Tests	Determine the extent of oil saturation and effects of oil and coking on the hardness and compressive strength of a material using the compressive ledge test previously described.

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- | | |
|--|--|
| o Differential Thermal Analysis (DTA) combined with Weight Loss Measurements | Determine the temperature of dehydration reactions and weight losses occurring as a function of increasing temperature. Relate this information to possible outgassing in service. |
| o Microscopic Examination of New and Post-Mortem of Used Header Materials | Use the scanning electron microscope (SEM) to identify morphological structure of new and used materials and relate properties and performance to changes in the microstructure. |

4.0 DISCUSSION OF RESULTS

4.1 Bulk Density and Strength

The overall HDC header grade board requirements are given in Table I. Adequate strength and strain to failure are needed for handling resistance, integrity in clamping to form a seal, and resistance to chipping at the overhang. See comparative values, Table II. The design of the header and mold assembly are shown in the cross-section view, Figure 1. During installation, the clamp rotates on its vertical appendage and pinches the header against the oil ring (Figure 1). On the other hand, excessive density accompanied by increasing strength is counterproductive if thermal conductivity increases occur. Elevated levels of open porosity (lower density) would increase the amount of oil absorption.

Generally, it has been observed that excessive chipping occurs for header grade material at the overhang if the ratio of amosite fiber to C-S binder becomes too low or the length of fiber bundles becomes too short. This could be attributed to loss of toughness which would be coexistent with a prevalence of C-S binder. The morphology of the amosite fibers/C-S binder indicates a relatively high fiber/binder ratio. A high strain-to-failure is also desirable when clamping the header to conform to a mold which may have warped during or after service. For example, a flexural deformation of the order of 1/8 to 3/16 inch/foot of length is obtainable with header grade MMM before cracks are initiated during installation.

Strength increases were noted at 1000°F (538°C) for all the C-S bonded materials except the L-16, L-17, L-18, and KX materials. This may be associated with the oxidation of carbon fibers. The KX and Pabco/32 materials were dropped from further consideration because of relatively low strength and crushing resistance. The Permtech Sigma exhibited a significant increase in strength but decreased maximum deflection at 1000°F. This would indicate a greater sensitivity to cracking if distortions occurred in the mold or temperature gradients become too severe during service. Past results of Permtech Sigma field trials in HDC casters at Warrick and at the Arnold Research facility have indicated that chances of immediate success using the board-type materials would be greater at this time. Further HDC trials using Permtech Sigma have been deferred at Warrick but are currently being pursued at Alcoa's Tennessee operations using a redesigned clamping system.

4.2 Bulk Density, Thermal Properties, and Oil Absorption

Generally, heat transfer properties of the various materials are related to composition, bulk density, porosity, and pore size and morphology. Values for thermal conductivity and diffusivity for various candidate header materials are given in Table III.*^{13,15} Materials having high thermal conductivity and diffusivity are more likely to chill molten metal.

Comparing those values at 600° (316°C) and 1000°F (538°C), thermal conductivity usually increases with temperature for the C-S bonded materials. If pores of the header were saturated with casting oil (AA Standard), which is mostly composed of castor oil having a relatively high density (60.1 lbs/ft³) and heat capacity (0.434 Btu/lb-°F) the density and thermal conductivity of the header would markedly increase and substantially increase the possibility of chilling molten metal.

The relative absorption of lubricating oil*¹⁶ by the various candidates is shown in Figure 2. The lowest weight pick-up for a header material saturated with warm AA standard lubricant was exhibited by MMM-HG. The ratings for the various materials according to lowest to highest degree of pick-up are: MMM-HG, MM, SFX, XM, N-14, L-18 and L-16. Because a definite relationship exists between the extent of oil pick-up of the header material and casting performance, a number of coatings and impregnants were evaluated in prior work*¹⁷ for purposes of decreasing corrosion, oil pick-up and moisture pick-up. Silicone resin coatings and impregnants were found to be the most

* See references.

promising in decreasing oil absorption and Al_2O_3 , TiO_2 and BN pigments were added to the silicones and applied to the header for improved molten metal resistance. However, during casting trials, exterior coatings were not effective in decreasing oil absorption or corrosion. The most promising materials easily impregnated the header material and provided an "in-depth" oil repulsion. Recent studies at ATC involved the impregnation of header materials with Dow Corning 805 which effectively decreased oil absorption. However, this effect declines when the header is heated up to molten metal temperatures (1200-1300°F). Some protection may be provided in lower temperature zones through the cross-section of the header shown in the computer plot, Figure 3. At temperatures above 800°F in air, silicones tend to break down to form silica and smoking may become a problem area.

For most candidate materials, no reproducible change in maximum compressive load resulted from oil saturation, although the compressive stress (psi) as a function of strain (inch/inch) plots (Figures 4 and 5) show a pronounced change in slope. This could be attributed to an initial collapse of the pore structure at the onset of loading followed by continued deflection to final failure. Only the compressive strength of L-16 seemed to be affected by oil saturation. The data given in the next table lists strength values determined for the two conditions:

001000

<u>Material</u>	<u>As-Received</u>		<u>Oil-Soaked</u>	
	<u>Initial Stress*</u>	<u>Crushing Stress*</u>	<u>Initial Stress</u>	<u>Crushing Stress</u>
MMM-HG	1300	2475	1400	3150
MM(A4.3-1)	1200	2130	1250	2100
XM-4	1000	1475	1200	1600
N-14	1425	2363	1400	2050
L-16	750	3325	400	2100
L-18	950	2400	1100	1850

* Stress-psi

Coking the oil at 1400°F resulted in significant decreases in strength for header grade, MM, and XM materials. The L-16 and N-14 materials retained a major portion of original strength. The compressive loading was performed on specimens with a 0.040 inch overhang to simulate the header/oil ring design. The as-received and oil soaked MMM-HG, N-14, L-16, and L-18 materials all withstood loading to failure (crushing) without chipping of this overhanging portion even when coked at 1000°F. The MM and XM chipped after 1000°F exposure. None of the materials had suitable chip resistance or strength when coked at 1400°F or retained compressive strength when exposed to the coke/MgCl₂ mixture at 1000° and 1400°F. This loss of bond strength was attributed to chlorine pick-up.

4.3 Thermal Dilation and Stability

Thermal dilation measurements^{18,19} were performed on candidate header materials to characterize their behavior at elevated temperatures. As

previously described, header grade MMM has composite-like properties because of the degree of preferred orientation of asbestos fiber bundles parallel to the board plane (upper and screen-side surfaces of the board). Therefore, two separate dilation measurements were made on specimens whose orientations were parallel and normal (perpendicular) to these surfaces. A typical plot of % linear change as a function of temperature for header grade MMM is shown in Figure 6. In-plane expansion occurs from 32°-1292°F (0°-700°C) followed by a dramatic shrinkage (2.0%) from 1292°-1472°F (700°-800°C). This marked shrinkage also occurs at a direction normal to the board plane and there is very little net expansion. This behavior seems to be related to most of the calcium silicate (C-S) bonded materials, which is shown in dilation plots (Figures 7 through 11). The tobermorite-type materials (MMM-HG and MM) exhibited greater shrinkage than most of the xonotlite-type materials (N-14, L-16, L-17, L-18, and KX). Anomalies are the non-C-S bonded Zircar and Permtech Sigma materials which are processed at temperatures above the molten metal use temperature (ZIRCAR) or composed of a calcium aluminate cement bonded aggregate (Sigma) respectively which exhibit a continued increase in thermal expansion. Typical values for average thermal expansion coefficients are given in Table IV and are useful for comparing the anisotropic nature of the candidate materials. For design studies, incremental values of dialation as a function of temperature must be considered if non-linear expansion behavior occurs.

During the casting process, the shrinkages exhibited by the C-S bonded materials will tend to offset (relieve) stresses caused by thermal expansion and clamping. However, excessive shrinkage normal to the board plane could completely break the desired seal between the oil ring and header directly causing defects in the as-cast ingot.

Understanding material characteristics such as dilation is important because bolt-torques and clamping movement required to maintain effective sealing through the use of Belleville washers may change with new materials. Dilation effects are modified considerably when the material is subjected to compressive stresses induced by clamping loads and relaxation occurs.

4.4 Differential Thermal Analysis (DTA)

Because of the tendency for C-S bonded board materials to pick up moisture in humid atmospheres and contain chemically bonded water associated with the calcium silicate and asbestos, DTA and weight loss versus temperature measurements were performed^{*20} to determine the temperatures where these reactions occur (see Table V). Most of the materials contained up to 3% mechanical water (adsorbed) and when heated at temperatures of 1292°-1472°F (700°-800°C), significant amounts of chemically bonded water continued to evolve. Reactions observed for L-16 and L-18 were attributed to the oxidation of carbon reinforcing fibers. Weight losses were detected for samples heated up to 2012°F (1100°C), which is well beyond the process temperatures encountered.

4.5 Load Relaxation Studies

To determine the extent of compressive load relaxation for various conditions, various specimens of candidate header materials were subjected to two different load levels (400 and 800 psi) at room temperature, 1100°F (593°C) and 1400°F (760°C). The results of these tests are summarized in Table VI.

At room temperature, all the candidates relaxed less than 10% even at higher loads (see Figure 12). Increasing test temperature to 1100°F (below the C-S dehydration temperature) the MMM exhibited lower levels of relaxation than the other candidates at 400 psi (see Figure 13). Increasing the load to 800 psi increased the relaxation for all candidates except L-18. At this point, it is suspect that some materials may exhibit crushing or initial failure and therefore relax less for the higher load (800 psi). Comparing test results for the 400 psi loads, tobermorite bonded materials relaxed substantially at 1400°F which is in the range of the dehydration temperature 1298°-1472°F (700°-800°C). This is further evidenced by an almost total relaxation of load for MMM-HG and MM (A4.3-1). It was surprising that the Manville xonotlite materials (XM) also relaxed almost completely at 1400°F (see Figure 14). This correlates with the dilation behavior and shows that excessive temperatures for these materials may also result in loss of seal between oil ring and header. The Nichias/Pyrotek materials did not relax as readily at 1400°F which is attributed to lower shrinkage and maintenance of load carrying ability at that temperature. These materials tend to relax more than MMM-HG at 1100°F, and the continued use of Belleville washers and 150 in-lb bolt torques is a reasonable requirement for initial installation trials. The relatively high relaxation at temperatures of 1400°F for MMM, suggests that the more stable xonotlite bonded Nichias candidates would offer better sealing for high temperature swings in the process, but may require slightly greater torques for sealing at nominal temperatures.

4.6 Molten Metal Immersion Studies

Bars of candidate header materials were immersed in molten 5182 alloy at 1400°F (760°C) for 3.5 and 24 hours*²¹ to determine the effects of intimate molten metal contact on their stability and corrosion resistance (see test results, Table VII). Of recent materials considered, L-18 had superior resistance to shrinkage after exposure to 1400°F. The XM board showed a marked improvement over MMM-HG. Except for the color change of the MMM-HG, none of the materials described in Table VII were wetted or reacted by the 5182 alloy. This test by itself was not sufficient cause to warrant acceptance or rejection of a material because of its excessive temperature over that commonly experienced in ingot casting (1220°-1360°F) (660°-738°C) (see Figure 15) and because of the isothermal exposure in lieu of hot-face exposure as actually experienced by the header. However, the sensitivity of MMM to elevated process temperature swings is shown by these tests. Overall, results of this testing closely agrees with dilation data formerly described because corrosion did not seem to be an obvious factor.

5.0 CORRELATION OF TEST RESULTS AND DESIGN ANALYSIS

Prior to conducting finite element analysis to determine the levels of temperature and stress in a Warrick HDC header*^{7,8,22}, a header and clamping mechanism was instrumented with thermocouples and strain gauges to measure temperatures, mechanical strains and thermal-mechanical strains during phases of: installation of the header on the mold, assembly of the mold and basin,

preheat, and start-up of casting and steady-state operations. Typical temperatures at a given location in the header is shown in Figure 16. To summarize this work, the following conclusions are related to results from materials tests on MMM:

1. Consistent with room temperature relaxation tests, very little clamping load relaxation occurred when the header was applied to the mold.
2. The tendency of the hot face of the MMM header is to contract when exposed to metal contact. This is attributed to the low or negative through-thickness expansion.
3. Without the compressive clamping loads, the thermal gradients in the header during start-up and operation induce zones of tension at the hot face (see Figures 17 and 18). This too is related to the negative through-thickness expansion.
4. Surface flaws on the hot face tend to open during start-up in the absence of clamping compressive loads. The negative through-thickness expansion influences this. Flaws are related to the laminar planes of fiber orientation.
5. Clamping loads induce compressive stresses in the header at the hot face and header/oil ring contact region. This compressive load opposes tension (from thermal effects) in the header. (These loads relax with excessive temperature.)

6. Nonuniform clamping loads introduce differences in clamping stresses and axial restraint around the header circumference. Excessive clamping loads in the critical overhang region (a stress-riser effect induces 3-4X average stresses) may cause localized damage. This may be in the form of cracks or crushing. Damage can be minimized by distributing clamping loads over a greater area in lieu of the point contact clamp design currently used. The use of the resilient Fiberfrax gasket between header and back-up board also decreases stresses.
7. Clamping loads are expected to decrease with time because of thermal expansion in the bolts and MMM header contraction in the clamping direction. Use of a material having positive through thickness expansion may result in higher stresses and cracking if torqued at present levels.
8. Extremely high thermocouple measured temperatures at the top corners and sides of the header indicate nonuniformity of cooling or combustion of the lubricant around the perimeter of the header and explains the localized damage to the experimental header during start-up. Higher thermal expansion materials would be more vulnerable to damage than lower expansion materials.
9. Fluctuation in metal temperature with time cycles stresses in the header material, accelerating material damage (through crack propagation) accumulated through clamping and start-up conditions.

10. Thermal expansion tests have shown severe dimensional contraction in the MMM at temperatures above 1300°F. Recorded metal temperatures fluctuating above 1360°F as previously shown could cause tensile cracks to occur in the hot face.
11. Combined effects of corrosion at the hot face, material shrinkage, and oil penetration and temperature effects in the header in the overhang region may cause material degradation and accelerate failure. This has been observed to be more of a problem area associated with magnesium containing aluminum alloys (5182). Chlorine pick-up also causes deterioration of material strength.

Using these findings to determine the selection of materials, the following comparisons can be made:

1. Tobermorite-type C-S bonded materials (MMM-HG and MM) tend to exhibit negative expansion coefficients in the through-thickness direction and therefore form tensile stresses on heat-up. Materials having positive expansion coefficients are in compression on heat-up. XM, L-16, L-17, L-18, Zircar, and Permtech Sigma all have positive through-thickness expansion coefficients. The marked shrinkage in MMM-HG and MM observed at 1292°-1472°F (700°-800°C) versus lesser shrinkage means more load relaxation and higher temperature stress relief than the other candidates. At lower mean temperature (1100°F), MMM exhibited the lowest relaxation of any material tested. The higher expansion coefficient materials such as Zircar could develop excessive stresses in the header application.

2. Excessive positive through-thickness expansion may result in chipping of the header for materials where shear or compressive failure results from excessive clamping loads. Cracks will not tend to open on the working face during heat-up.
3. The XM did not exhibit a marked through-thickness and in-plane shrinkage at 1292°-1472°F (700°-800°C) to the same degree as MMM-HG and MM. L-16, L-17, and L-18 showed a marked improvement in stability contributing to a decreased tendency for cracking and loss of seal between header and oil ring if process temperatures become excessive.
4. Corrosion of nonasbestos containing headers in magnesium containing alloys is improved for excessive temperatures. Oil soaking and coking does not seem to deteriorate mechanical properties of nonasbestos materials but does increase heat absorption and heat losses which could effect thermal properties and cause undesirable chilling of molten metal on the header in lieu of solidification at the oil ring. The early Nichias L-16 and L-17 materials both had exhibited excessive oil saturation. The L-18 material showed decreased oil saturation but was still at higher levels than the XM and MM materials which were on a par with MMM-HG. Pick up of chlorine was associated with a loss of strength for all the calcium silicate bonded materials.

6.0 EXAMINATION OF NEW AND USED HEADER MATERIALS

The composition of the various candidates were described in the initial section of this report. The purpose of this work was to examine fracture surfaces of new and used materials to determine the relative morphology before and after use and to improve understanding of the basic properties of the materials. Header materials are composites with the hydrated calcium silicate binder phase reinforced with one or more fibrous materials. A minimum amount of fibers are added to improve toughness and decrease chipping and cracking. If excessive amounts of fiber are added a condition could exist where too high a concentration of fiber of uniformly large diameter and length can create a material of high compressibility and lowered bending strength because of inefficient bonding and excessive void formation. The information in the Appendix provides detailed comments on scanning electron photomicrographs (Figures A-1 through A-17) of new and used candidate header materials.

The structure of MMM-HG is composed of chopped bundles of asbestos fibers (Figure A-1) that on close examination develops a material structure that is largely controlled by these bundles. It is apparent that some of the alternate materials tested are designed to duplicate the properties of asbestos fiber reinforced MMM-HG by adding a substitute fibrous material. However, the matrix (hydrated calcium silicate) controls the material structure in these alternate materials because comparable levels of long, large diameter substitute fibers which are used in lieu of asbestos would tend to increase compressibility of the board and also increase oil permeability. The more open network of crystalline Xonotlite that make up the bond of L-17 and L-18 could also contribute to excessive permeability. When MMM-HG is

installed or clamped on the mold, the fine bundles of asbestos fibers serve to block these open channels and therefore reduce oil permeability and tend to act as a more efficient "gasketing" material. This blocking tendency would increase as the header is compressed during clamping and the bundles are broken into minute fibers that more efficiently fill void spaces. Special attention is given to placing the "screen side" of MMM-HG against the oil ring because of the higher levels of finely divided fibers present in the surface layers of that surface are more suitable for sealing.

In all cases the used header materials are altered in the immediate zone that was exposed to molten metal. This zone seems to be denser and less of the original binder morphology is obvious. Reinforcement is almost completely depleted in the exposed surface of the carbon fiber containing L-17 material. This would result in the decreased hot strain-to-failure values observed for the Nichias materials (with the exception of N-14). Other than the densification observed, no obvious cracking, reaction or melting seems to have taken place in the used materials examined. Carbon pick-up related discoloration occurs, but no marked build-up of deleterious material was obvious. A continued post mortem examination of used header specimens should continue as part of this program.

7.0 DESCRIPTION OF PLANT TRIAL RESULTS

To date, Permtech Sigma, Metal Mover, N-12¹⁰, N-14^{11,12}, L-17¹³, and XM¹⁴ materials have been evaluated in plant trials. Table VIII reviews the results of these trials. Trials with Permtech Sigma, N-12, N-14 and L-17 were not successful.

Trial results to date indicate that the XM material has currently proven to show the most promise. The new L-18 material, with decreased oil absorption over L-16 and L-17, may also prove to be a viable candidate. (The L-17 trial was deemed unsuccessful because of excessive oil pick-up.) Although Metal Mover showed an acceptable trial performance, processing problems (white spots) prevented it from being reliably produced and from being commercially available. The same Manville Corporation process equipment is being used for making MMM-HG, MM and XM.

8.0 OVERALL SUMMARY AND RECOMMENDATIONS

The requirements for an acceptable HDC header material are fairly complex because of the number of material and process variables involved. The primary goals of this work were to: (1) understand how MMM-HG functions in the HDC process, (2) characterize candidate materials and develop a database, and (3) recommend substitute materials based on combined information from design analysis, material properties, material post mortem analysis and field experience. The desirable properties of MMM-HG are:

1. Available in suitably homogeneous (macro) board or shape form. If in board form, the material must be readily machinable into a header shape within required tolerances. Machined surfaces should be relatively

smooth to minimize friction (actual surface roughness should be characterized). The working surface is often lightly sanded with emery paper to obtain the required surface texture.

2. Nonwetted and limited corrosion by magnesium containing alloys up to temperatures of 1300°F (~700°C).
3. Suitably low density to minimize heat losses and heat transfer from molten metal (for HDC process conditions).
4. Low permeability, absorption, and reaction with lubricating oil.
5. Limited and reproducible compressibility and load relaxation perpendicular to the board plane at room temperature and at operating temperature (=1100°F).
6. Low thermal expansion in the in-plane directions to minimize cracking when constrained in the HDC mold. Near zero or negative thermal expansion perpendicular to that plane. Predictable and uniform dilation behavior throughout the process temperature range. Limited use beyond 1300°F (~700°C).
7. Acceptable thermal shock resistance (combined low coefficient of thermal expansion, low elastic modulus, and high strength).

8. Adequate strength and toughness, low Poisson's ratio to effectively maintain a stable overhang when compressed against the oil ring.
- Adequate resistance to chipping during service in the HDC process.

A summary comparing the suitability of various candidate materials for HDC headers is given below:

SUMMARY: Analysis of Various Aspects of Suitability for Various Candidate HDC Header Materials

Parameters	Materials					
	MMM-Hg	MM	XM	N-14	L-18	Permtech Sigma
Legend o = unacceptable ✓ = acceptable • = no comment or unknown						
1. Available in homogeneous board form. Must be machinable into or available in accurate header shape.	✓	✓	✓	✓	•	✓
2. Non wetted and limited corrosion by Mg-Al alloys up to 1300°F.	✓	✓	✓	✓	✓	✓
3. Suitably low density to minimize heat losses and heat transfer from molten metal (HDC process).	✓	✓	✓	✓	✓	o
4. Low permeability, absorption and reaction with lubrication oil (HDC process).	✓	✓	✓	o	•	✓

(continued on next page)

SUMMARY: Analysis of Various Aspects of Suitability for Various
Candidate HDC Header Materials

Parameters	Materials					
	MMM-Hg	MM	XM	N-14	L-18	Permatech Sigma
5. Limited and reproducible compressibility and load relaxation normal to the board plane.	✓	✓	✓	o (L-17 unacceptable)	• (incompressible)	•
6. Low in-plane thermal expansion to minimize cracking when constrained in the HDC mold. Near zero or negative expansion normal to that plane. Predictable and uniform dilation behavior up to 1300°F.	✓	✓	✓	✓	✓	o
7. Acceptable thermal shock resistance.	✓	✓	✓	✓	✓	o
8. Adequate strength and toughness, low poissons ratio to maintain a stable overhang during service in the HDC process.	✓	✓	✓	o	•	o
9. Heat treated to minimize offgassing during the casting process.	✓	✓	✓	✓	•	✓

Physical properties that more accurately describe these parameters are listed in Table IX. These properties should not be considered as specifications, but as guidelines for further materials selections. Material consistency is obviously an important issue once a given product has been selected. Detailed discussion of this aspect is beyond the scope of this report and should be given separate treatment.

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TABLE I. CURRENT HDC HEADER BOARD REQUIREMENTS*⁹

- I. Dimensional tolerances: Both lateral and longitudinal lengths must meet requirements.
- Taper - .010 inch/ft (MAX.)
 - Flatness - Bow - .0156 inch/ft (MAX.)
 - Twist - .0156 inch/ft (MAX.)
 - Thickness - $1.00 \pm .0312$ inch
- Effects of dimensional tolerances:
1. Material loss (cracking)
 2. Header overhang ($\pm .0625$ inch)
 3. Uniform continuous clamping pressure
- II. Physical Properties and Requirements:
- Density - 40 ± 2 lbs./ft³
 - Transverse Strength (modules of rupture) - 800 psi min
 - Thermal Conductivity (BTU - in/hr-ft²-°F) at Mean Temperature of 1000°F (538°C) - 0.85 avg.
 - Screw holding strength (withdrawal load for type A, No. 8 sheet metal screw in a No. 29 drilled hole) -
 - .50 inch penetration - 100 lb/screw min.
 - .875 inch penetration - 250 lb/screw min.
 - Dimensional Change after reheating at 1100°F
 - length, width and thickness - ± 0.4 % Max.
 - Durometer Hardness Units
 - die side - 55 min.
 - screen side - 55 - 70

(Table I continued on next page)

* Reference 9

TABLE I - Continued

III. Finish Standards:

•Sanded Surface Requirements

1. Roller Marks - .005 inch max.
2. Ridges, groves, scratches or gouges - depth and width
.0156 inch max.

•Foreign material - None

IV. Identification:

•Identification of the screen side is desired due to its higher density

V. Shelf Life:

•Moisture pick-up requirements currently being developed (hygroscopic)

TABLE II - Properties of Header Grade Candidates

Material	Density lbs/cu ft	Duro.	$\frac{\text{MOR (psi)}^1}{\text{R.T.}} @ 1000^\circ\text{F}$		$\frac{\text{MOE (psi)}^2}{\text{R.T.}} @ 1000^\circ\text{F}$		$\frac{\text{Maximum Deflection } (\Delta)^3}{\text{R.T.}} @ 1000^\circ\text{F}$	
MM-RG	41.7	60	663	1127	79	105	.035	.045
MM-HG	39.1	63	775	1057	98	104	.033	.042
MM	49.7	65	736	753	133	144	.023	.022
XM	46.2	58	594	714	108	87	.023	.034
N-14	54.4	63	895	1118	105	136	.036	.034
L-16	48.0	62	1343	721	155	129	.036	.023
L-17	46.0	62	1131	712	-	153	-	.019
L-18	43.7	70	1238	805	134	155	.038	.022
KX	31.2	48	609	71	-	-	-	-
Pabco 32	31.9	-	537	575	77	66	.029	.036
Permotech	125.0	N/A	1627	2744	306	700	.022	.016
Sigma								
Specification ⁴	40 +2	55-70	800	-	-	-	-	-

Notes: (1) $S = \frac{3PL}{2bh^3}$ = Three-point bending strength (psi), P = Applied load, L = Span (5 inches),
 b = breadth of specimen cross-section, h = height of the cross-section.

(2) $E = \frac{PL^3}{4bh^3\Delta}$ E = Bending modulus of elasticity (psi).

(3) $\Delta = \frac{PL^3}{4bh^3E} = 4.167 S/E$, $\Delta_{12}'' = 5.76 \Delta_5''$, Δ = Maximum deflection of a 5 inch beam (inches) or
 a 12 inch beam.

(4) References 9 and 15.

TABLE III - Thermal Properties of Various Header Candidates

<u>Material</u>	<u>Bulk Density</u> <u>lbs/cu ft</u>	<u>Thermal Conductivity¹</u> <u>Btu-in/ft²-°F-hr</u>		<u>Thermal</u> <u>Diffusivity²</u> <u>ft²/hr</u>
				<u>@ 1000°F</u>
		<u>@ 600°F</u>	<u>@ 1000°F</u>	
Ceramic Fiber Board ³	20-28	0.47	0.66	0.14- 0.10
Molten Metal Marinite ³	40	0.82	0.85	0.09
Metal Mover ³	50	0.76	0.84	0.07
XM ³	46	0.77	0.88	0.08
Marinite C ³	45	1.18	1.22	0.10
N-14 ⁴	52	1.33	1.38	0.11
Permotech Sigma ⁵	125	4.60	5.08	0.16

Notes: 1) Thermal conductivity - k
 2) Thermal diffusivity - $k/\rho C_p$
 ρ = density
 C_p = specific heat

Sources: 3) Manville Corporation - Reference 13
 4) Nichias Corporation - Reference 15
 5) Alcoa Technical Products Division - Reference 28

TABLE IV - Average Thermal Expansion Coefficient (70-1292°F) -in/in/°F x 10⁻⁶

	<u>Material</u>	<u>Parallel to Board Plane</u>	<u>Perpendicular to Board Plane</u>
Manville	MMM-HG	3.49	-0.95
"	MMM-RG	2.70	2.86
"	MM-old	2.78	0.71
"	MM-new	2.38	-3.57
"	XM-4	4.52	1.51
Nichias	N-14	3.81	-1.59
"	L-16	4.29	3.57
"	L-17	5.56	3.02
"	L-18	2.50	1.50
"	KX	2.22	-2.06
Pabco	32	2.70	-0.24
ZIRCAR	AL-45	6.03	5.63
Alcoa TPD	Permatech Sigma (R-680)	2.90	2.90

TABLE V - Observed Reactions and % Weight Change for
Header Materials

		Observed Reaction				
		Temp. - DTA				
		<u>Samples Heated to 1100°C*</u>				
Sample		Low Temp.	Hi-Temp.	Accumulative % Weight Loss ^{1,3}		
		Reaction 1	Reaction 2	110°C	800°C	1100°C
<u>Code</u>	<u>Description</u>	<u>Temp. °C</u>	<u>Temp. °C</u>			
R443	MMM-HG ²	100-**	775+	2.15	6.11 ²	7.05
R280	MMM-RG	None	775+	-	-	8.50
R418	N-14	100-	NONE	0.60	7.25	8.22
R386	L-16	100-	725-	0.27	6.47	6.57
R448	L-18	100-	725-	-	-	-
R370	MM(A4.3-1)	100-	800+	2.76	9.40	10.40
R372	XM-4	100-	800+	1.04	6.55	7.77

* (Temperature conversions - 100°C = 212°F, 725°C = 1341°F, 775°C = 1427°F, 800°C = 1472°F, 1100°C = 2012°F.)

** + exothermic, - endothermic

Notes:

1. All samples were held at each temperature for 1 hour. Weight losses are accumulative for each temperature. The same sample was used throughout the test.
2. MMM-HG fired at 800°C showed noticeable cracks, deformation, and shrinkage.
3. After these samples were fired at 1100°C, they were allowed to cool at room temperature for 28 hours.

TABLE VI - Load Relaxation for Candidate Header Materials

Material	Applied Load (psi)	Percent Load Relaxation @ Temperature - °F (1)		
		RT	1100	1400
MMM-HG	400	5.7	13.8	98.0 ₃
	800	7.0	33.0	82.0 ₃
MM-(A4.3-1)	400	4.9	28.0	100.0 ₉
	800	7.2	31.5	87.0 ₃
XM-4 ²	400	4.8	-	70.0
	800	5.8	21.0	81.0
XM-6 ²	400	4.8	28.1 ₃	100.0 ₉
	800	6.1	21.2 ₃	66.0 ₃
N-14	400	4.6	16.8	56.0 ₃
	800	6.6	19.8	39.0 ₃
L-17 ²	400	3.0	-	12.0
	800	4.7	16.0	26.0
L-18 ²	400	5.2	19.3 ₃	48.0 ₃
	800	5.6	13.0 ₃	24.0 ₃

Notes:

1. Temperature conversions - RT(70°F) = 21°C, 1100°F = 593°C, 1400°F = 760°C.
2. XM-4 and XM-6 are different batches of the same material. L-17 is the same chemistry as L-16 and L-18 but intermediate in density.
(L-16: 48.0 lbs/cu ft, L-17: 46.0 lbs/cu ft, L-18: 43.7 lbs/cu ft)
3. Relaxation decreased as a result of crushing that occurred when increasing applied compressive load from 400 to 800 psi.

TABLE VII - Immersion Test Results at 1400°F (760°C) in 5182 Alloy

Material	3.5 Hour Soak		24 Hour Soak	
	Shrinkage %	Comments	Shrinkage %	Comments
MM-HG W* T	1.1	Non-wetted, slight shrinkage, no discoloration.	9.8	Non-wetted, delamination cracks, turned brown to grey, significant shrinkage.
	1.5		12.0	
MM W A4.3-1 T	2.9	Non-wetted, moderate shrinkage, no discoloration.	5.0	Non-wetted, significant warpage and shrinkage, no discoloration.
	7.0		14.3	
XM-6 W T	0.8	Non-wetted, moderate shrinkage, no discoloration.	2.0	Non-wetted, moderate shrinkage, no discoloration.
	4.7		6.3	
L-18 W T	0.7	Non-wetted, slight shrinkage, no discoloration.	1.3	Non-wetted, slight shrinkage, white to grey above the metal line.
	1.4		1.9	

*Notes: T - Measurement made parallel to the board thickness.
W - Measurement made parallel to the board length or width.

TABLE VIII - HDC Header Trial Results - Warrick Operations

<u>Material</u>	<u>Date</u>	<u>Results</u>
Alcoa Permtech Sigma (R-680)	1977	Material sensitive to cracking as a result of stresses during installation and start-up. No successful start-up on Warrick's HDC caster resulting in ingot tearing.
Manville Metal Mover	1982	Successful HDC runs. Header material was inhomogeneous with the presence of white spots. Manville withdrew the product to correct processing problem areas.
Nichias/Pyrotek N-12	-	Excessive shrinkage and oil absorption. Ingot tearing attributed to header shrinkage.
Nichias/Pyrotek N-14	1984	Excessive absorption of mold lubricant. Cracking and chipping of the header. Excessive chilling caused tears and cracks in the ingot surface.
Nichias/Pyrotek L-17	1985	Termination of 5182 cast (26 1/4 hrs) resulting from excessive gaps forming between header and oil ring leading to ingot tears. Excessive oil absorption.
Manville XM	1985	Eight trials (10 1/2, 25 1/4, 43 3/4, 19 1/4, 45 1/4, 43 1/4 hrs - 5182; 75 hrs - 3004; 114 hrs 5182/5042, and 77 hrs - 5182/5352). No casting stoppages attributed to unusual header performance.

TABLE IX - Properties for Header Grade Marinite

Bulk Density	- 40 $\pm$ 2 lbs/cu ft
Durometer	- 55-70 (screen side)
MOR/MOE	- 700 psi/100,000 psi at R.T. to 1000°F
Compressive Strength	- 1000 psi (initial crushing normal to the-board plane)
Compressive Load Relaxation	- 5-10% at R.T., 10-20% at 1100°F at 400 psi load (normal to board plane)
Poisson's Ratio	- 0.10
Thermal Conductivity	- 0.85 Btu-in/ft ² -°F-hr at 1000°F mean temperature (normal to board plane)
Thermal Diffusivity	- 0.09 ft ² /hr at 1000°F mean temperature
Average Thermal Expansion Data (70°-1292°F)	- α = 3.49×10^{-6} in/in/°F (parallel to board plane) = -0.95×10^{-6} in/in/°F (normal to board plane)
Oil Absorption (AA Castor Oil)	- 10-15 weight % (hot oil)
Permeability	- No data

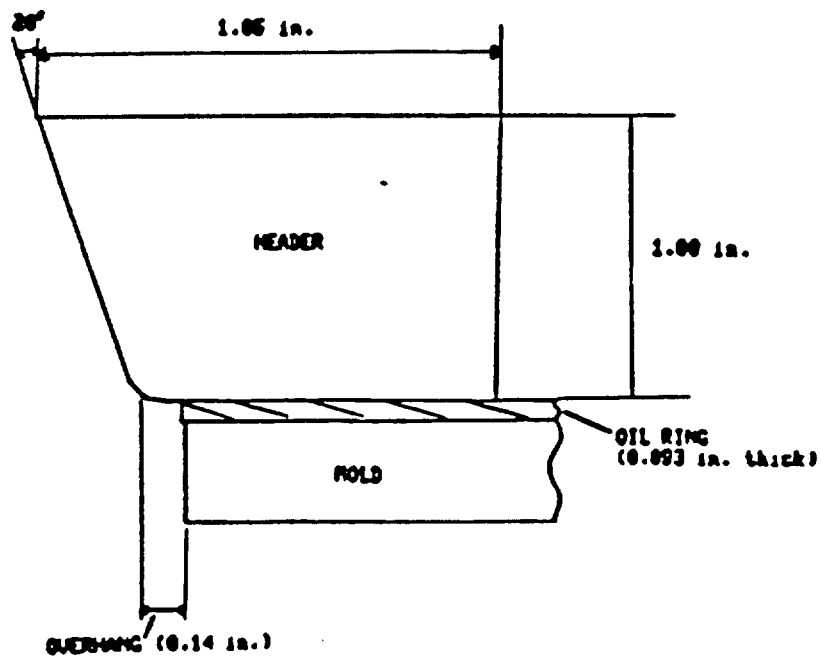
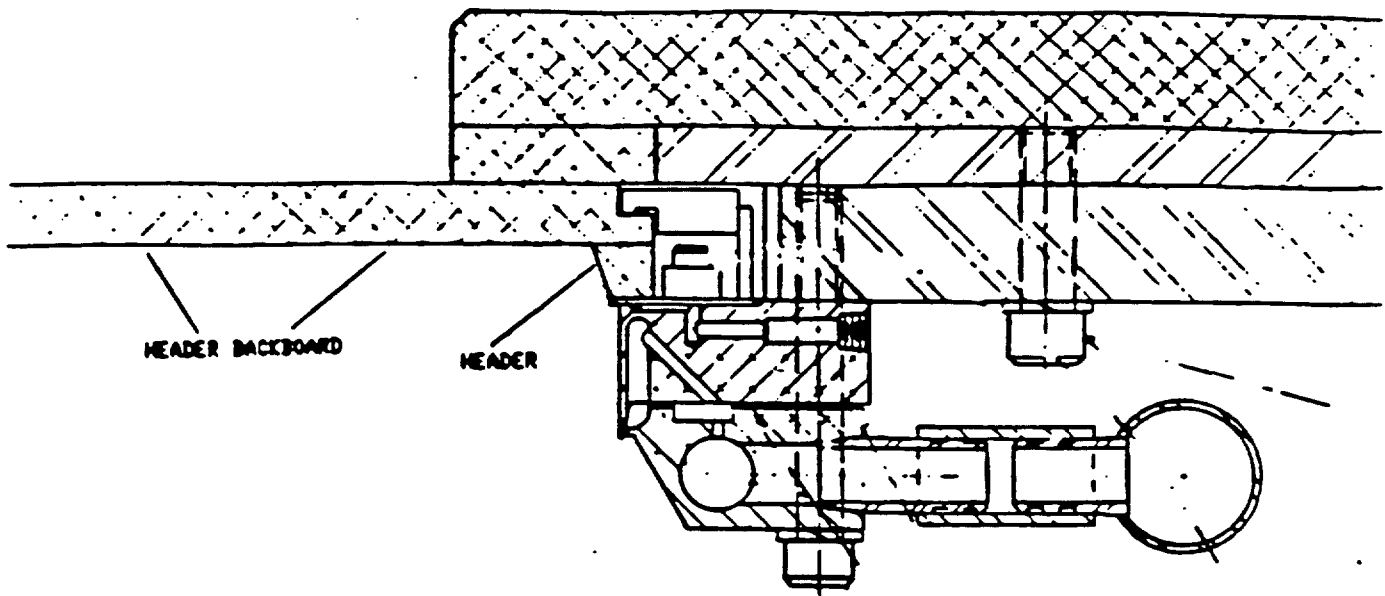
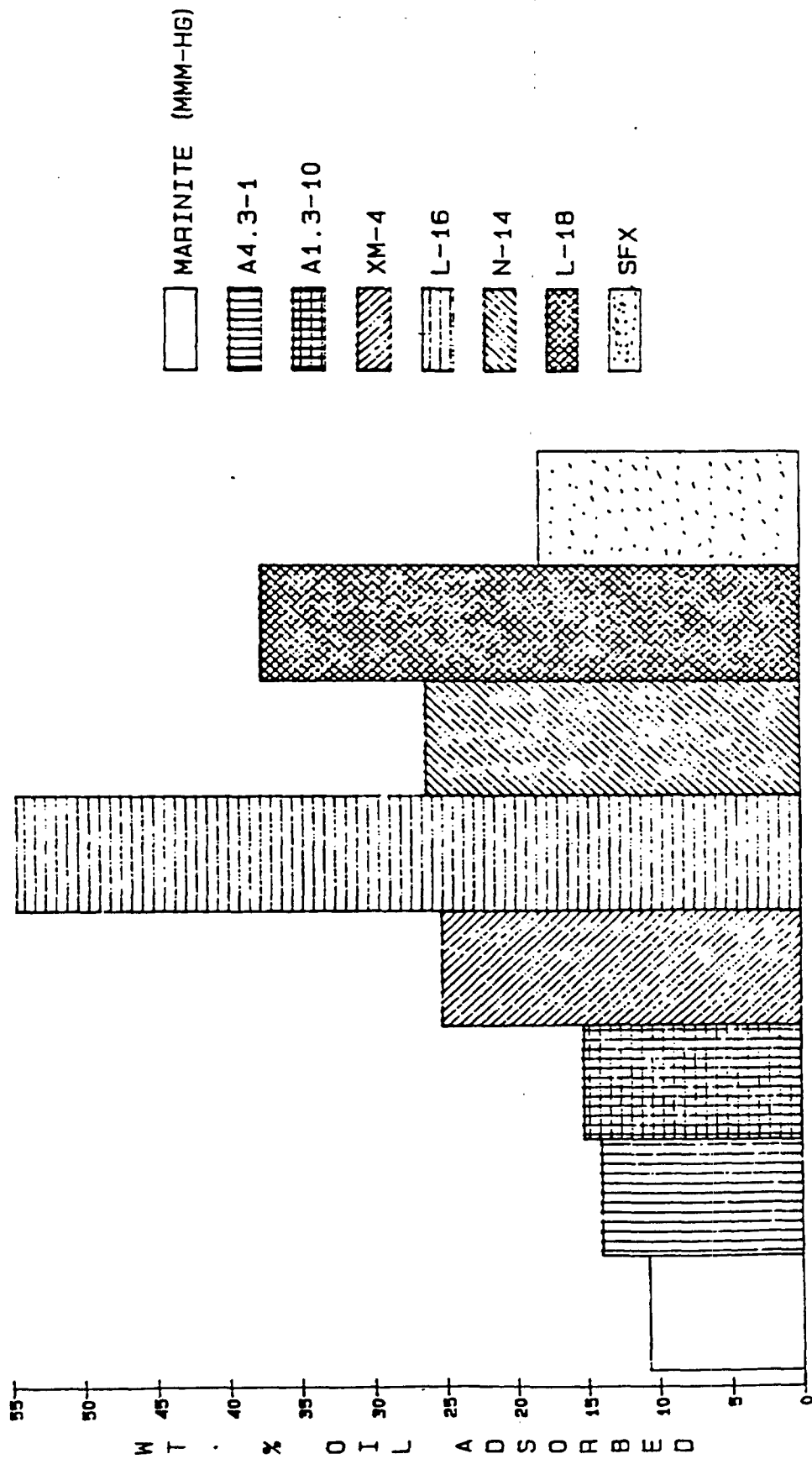


FIGURE 1 - DETAIL OF CLAMPING ARRANGEMENT

OIL "A" ADSORBED BY MATERIALS



MATERIALS TESTED

FIGURE 2. Amount of AA Standard Lubricating Oil Pick-up for Various HDC Header Candidates.

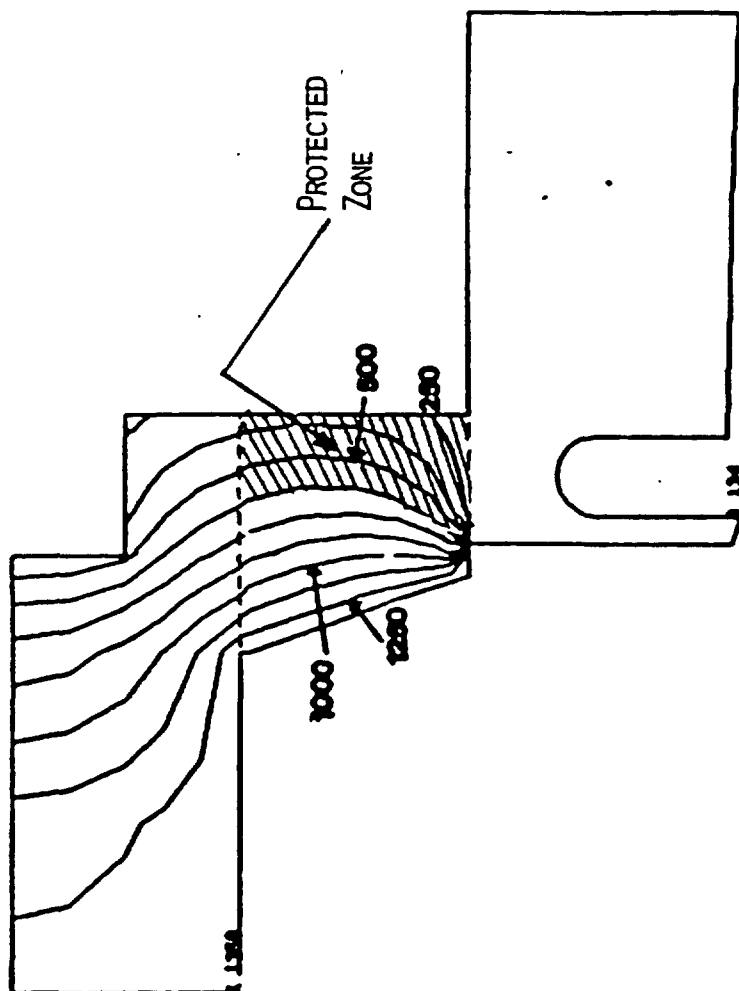


FIGURE 3 - STEADY STATE TEMPERATURE DISTRIBUTION
IN HEADER (METAL CONTACT TO OIL RING)

COMPRESSIVE STRESS VERSUS STRAIN IN AS-REC. MATL.

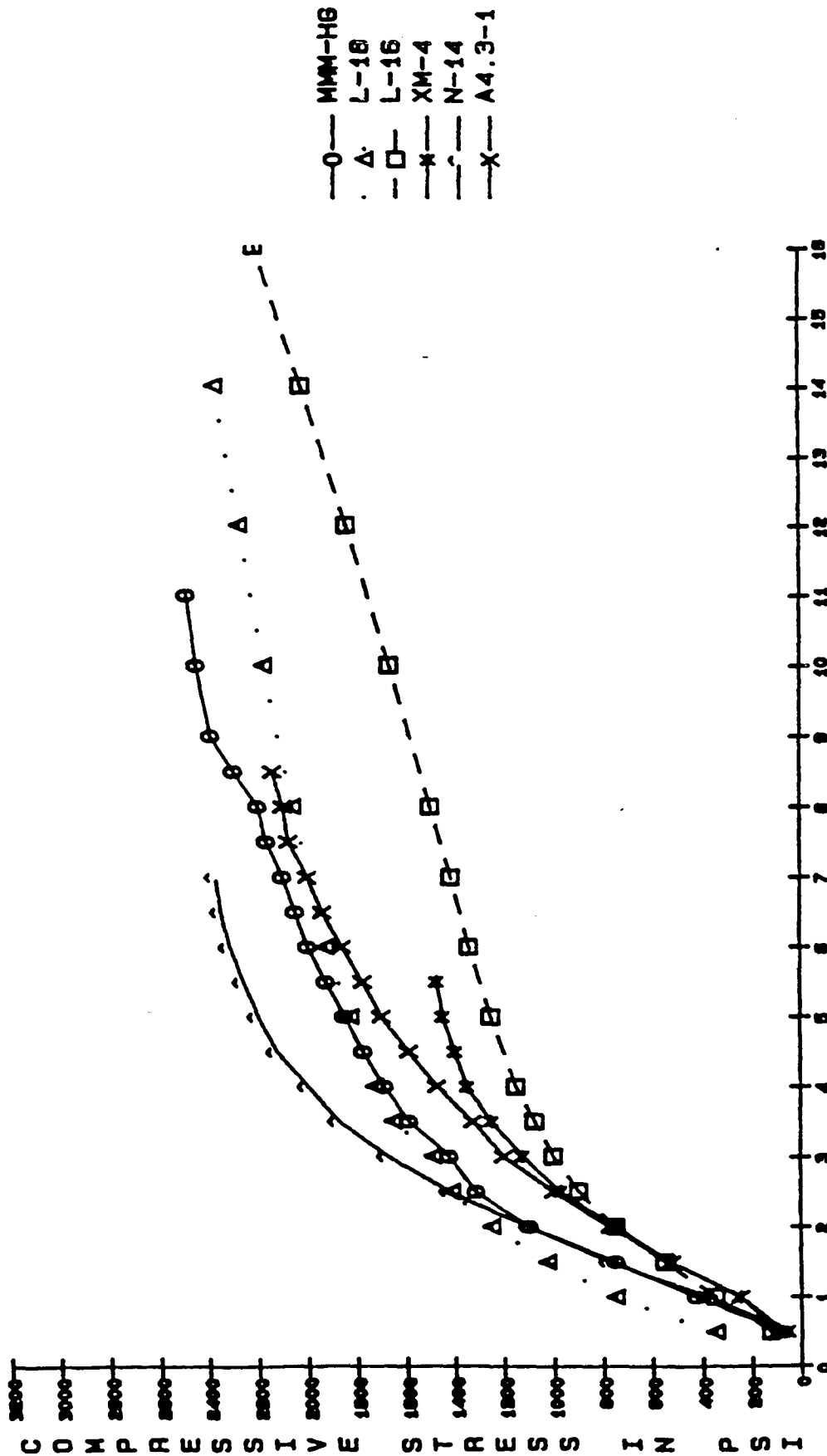


FIGURE 4. Compressive Stress Versus Strain-As-Received Materials.

COMPRESSIVE STRESS VERSUS STRAIN IN OIL SOAKED MATL.

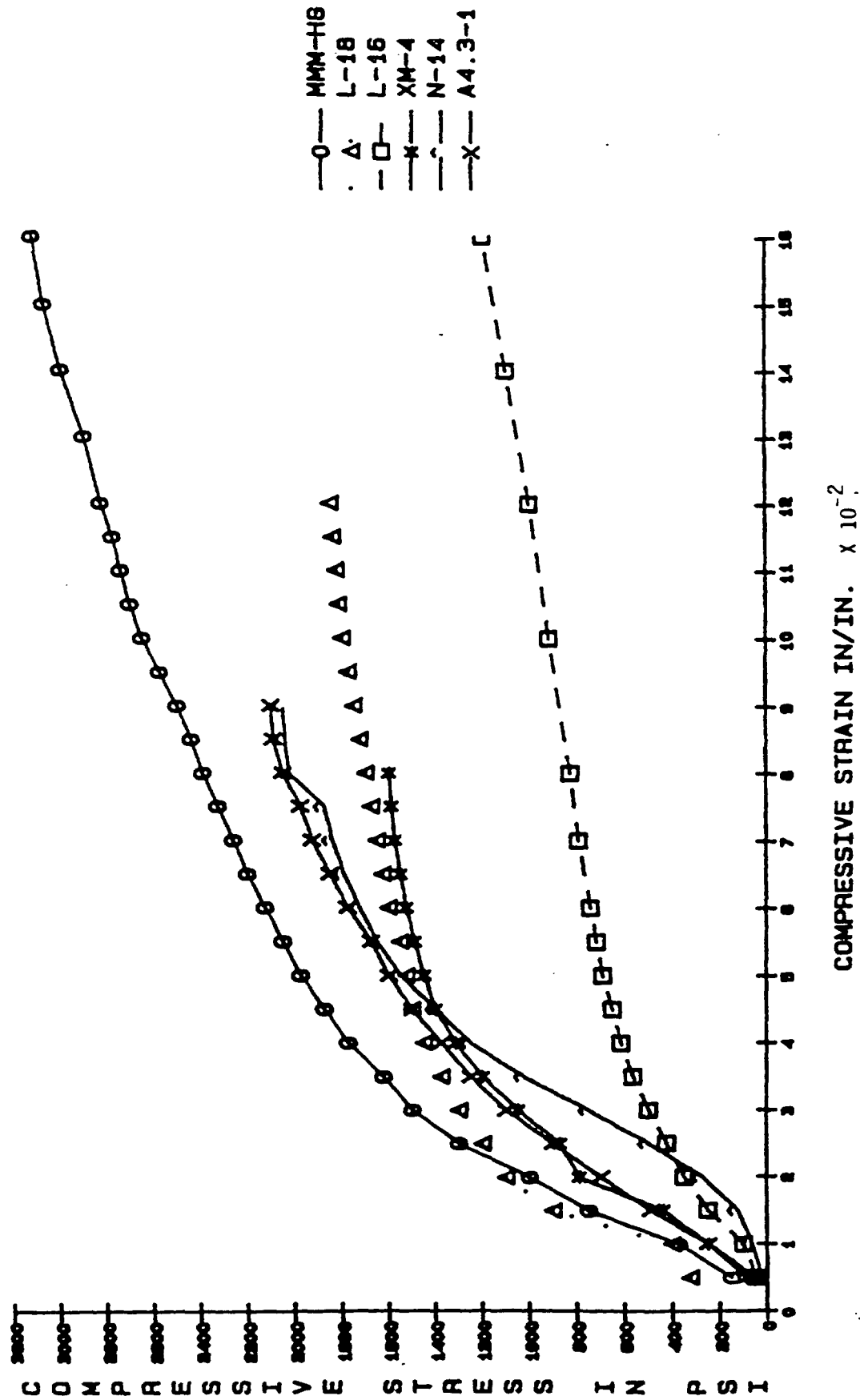


FIGURE 5. Compressive Stress Versus Strain-Oil-Soaked.

R279 MANSVILLE MOLTEN METAL MARINITE - HEADER GRADE

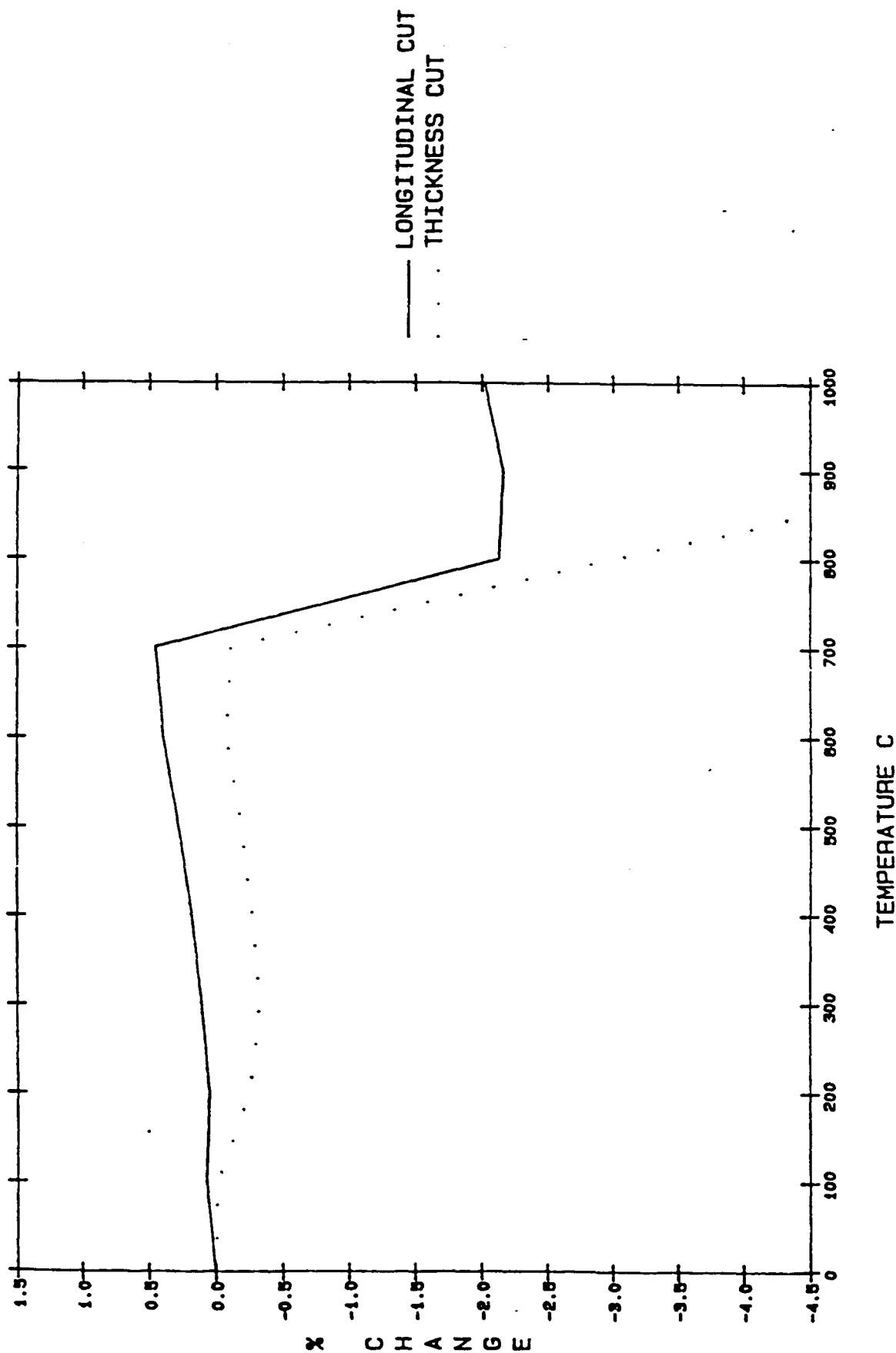


FIGURE 6. Thermal Change as a Function of Temperature
MM-HG: Parallel and Normal to the Board Plane.

COMPARISON OF LONGITUDINAL CUT HEADER MATERIALS

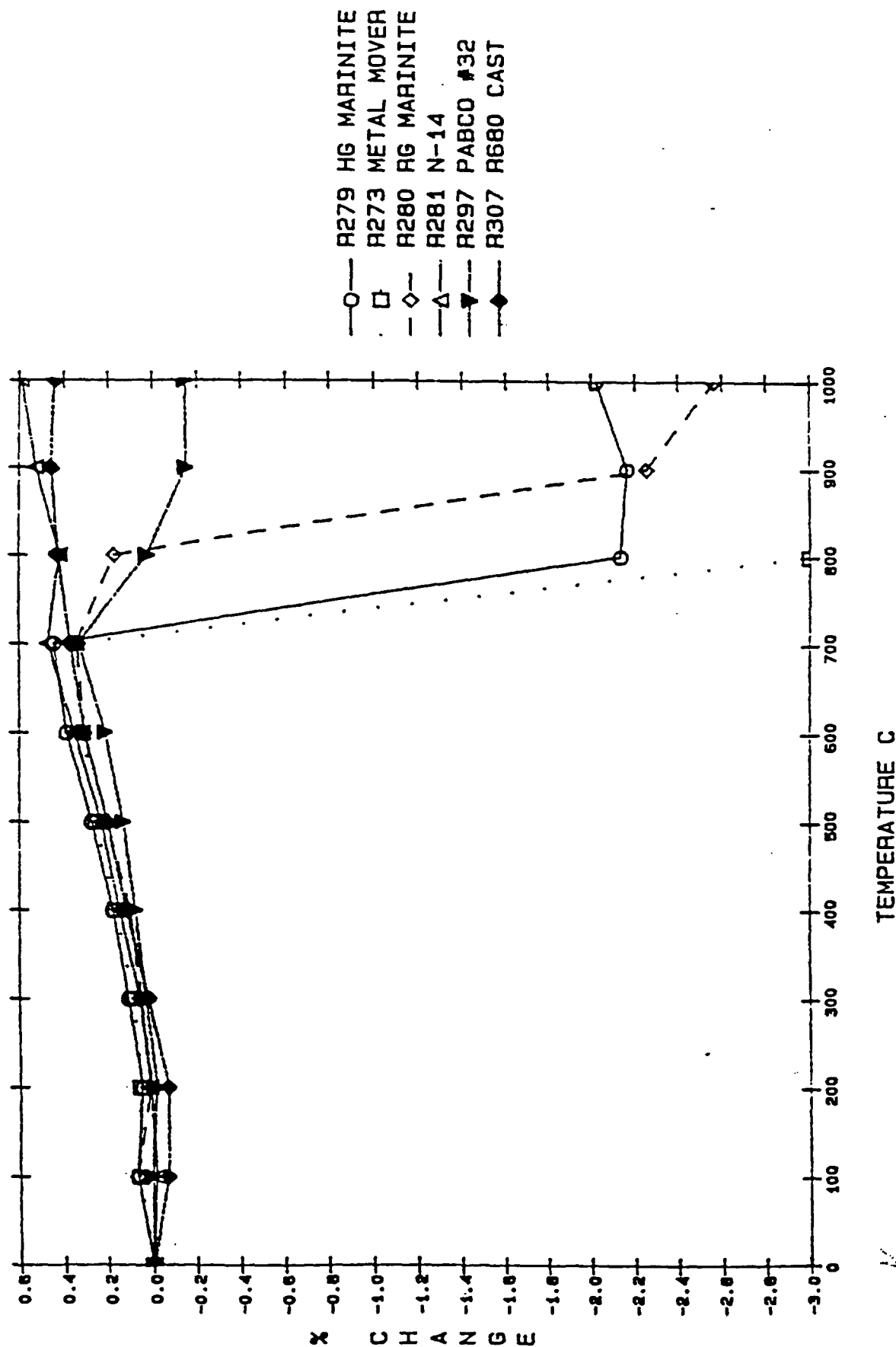
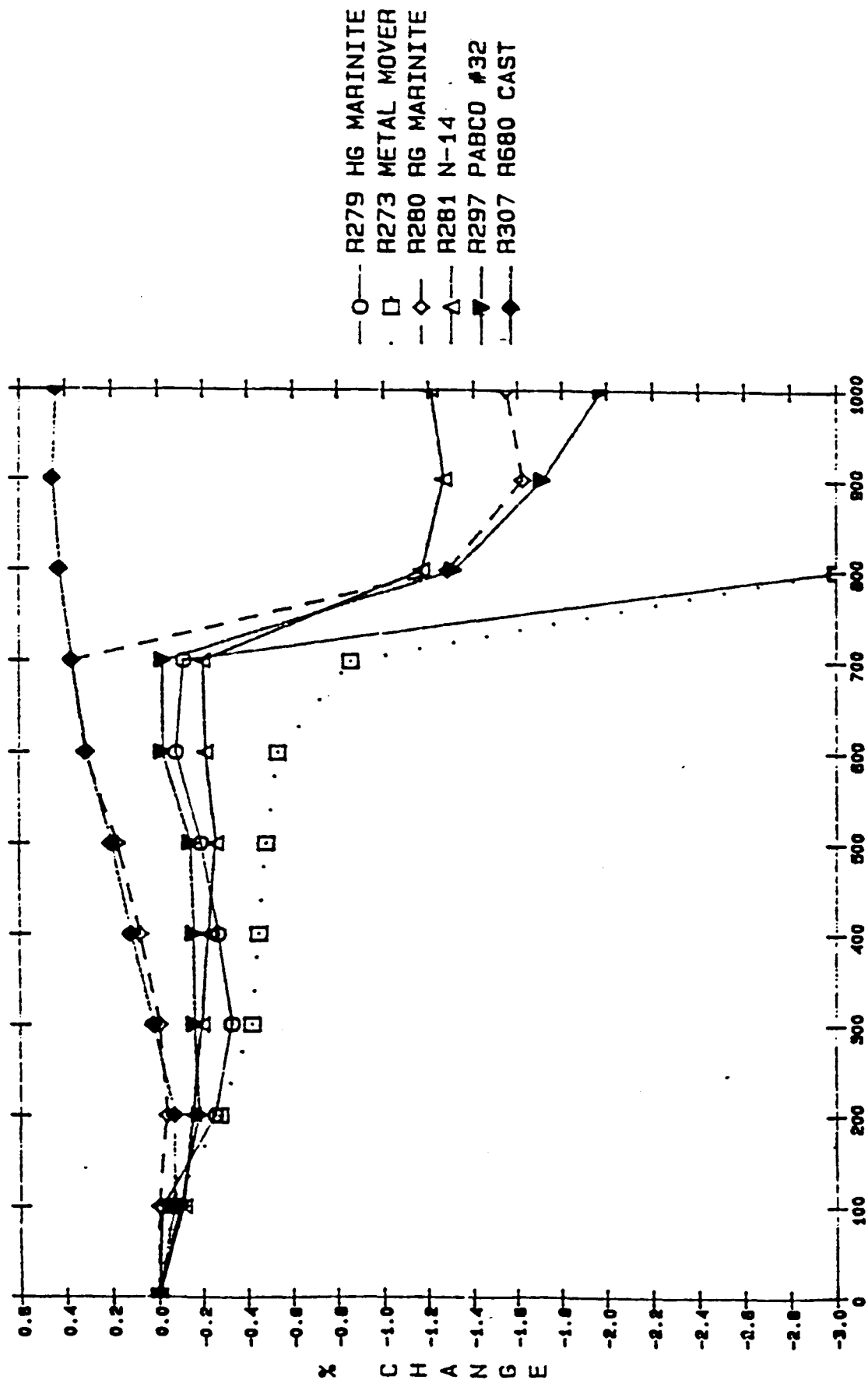


FIGURE 7. Comparison of Thermal Change Behavior for Various Header Materials - Parallel to the Board Plane.

COMPARISON OF THRU THICKNESS CUT HEADER MATERIALS



TEMPERATURE C

FIGURE 8. Comparison of Thermal Change Behavior for Various Header Materials - Normal to the Board Plane.

COMPARISON OF LONGITUDINAL CUT HEADER MATERIALS

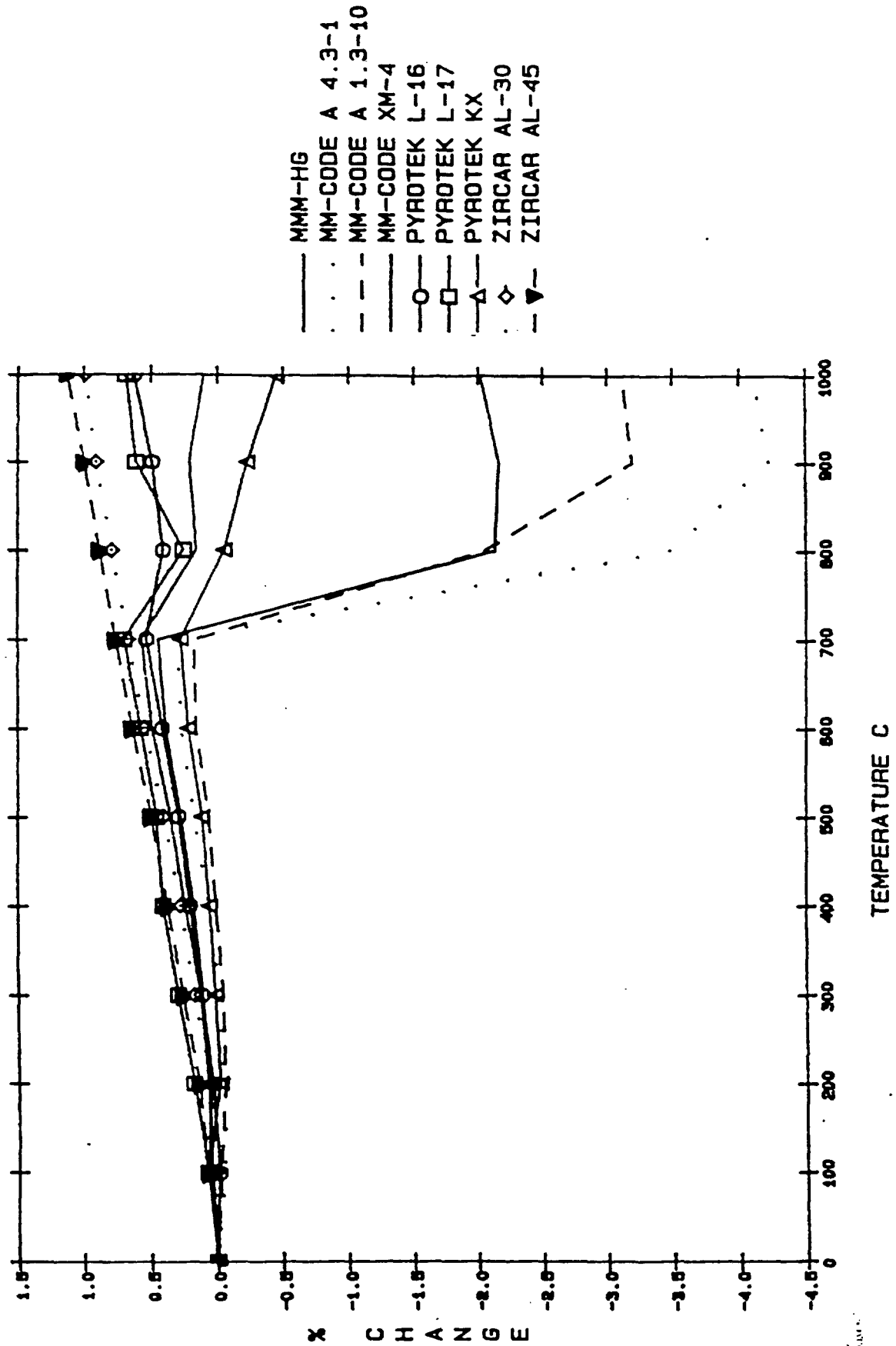


FIGURE 9. Comparison of Thermal Change Behavior for Various Header Materials - Parallel to the Board Plane.

COMPARISON OF THRU THICKNESS CUT HEADER MATERIALS

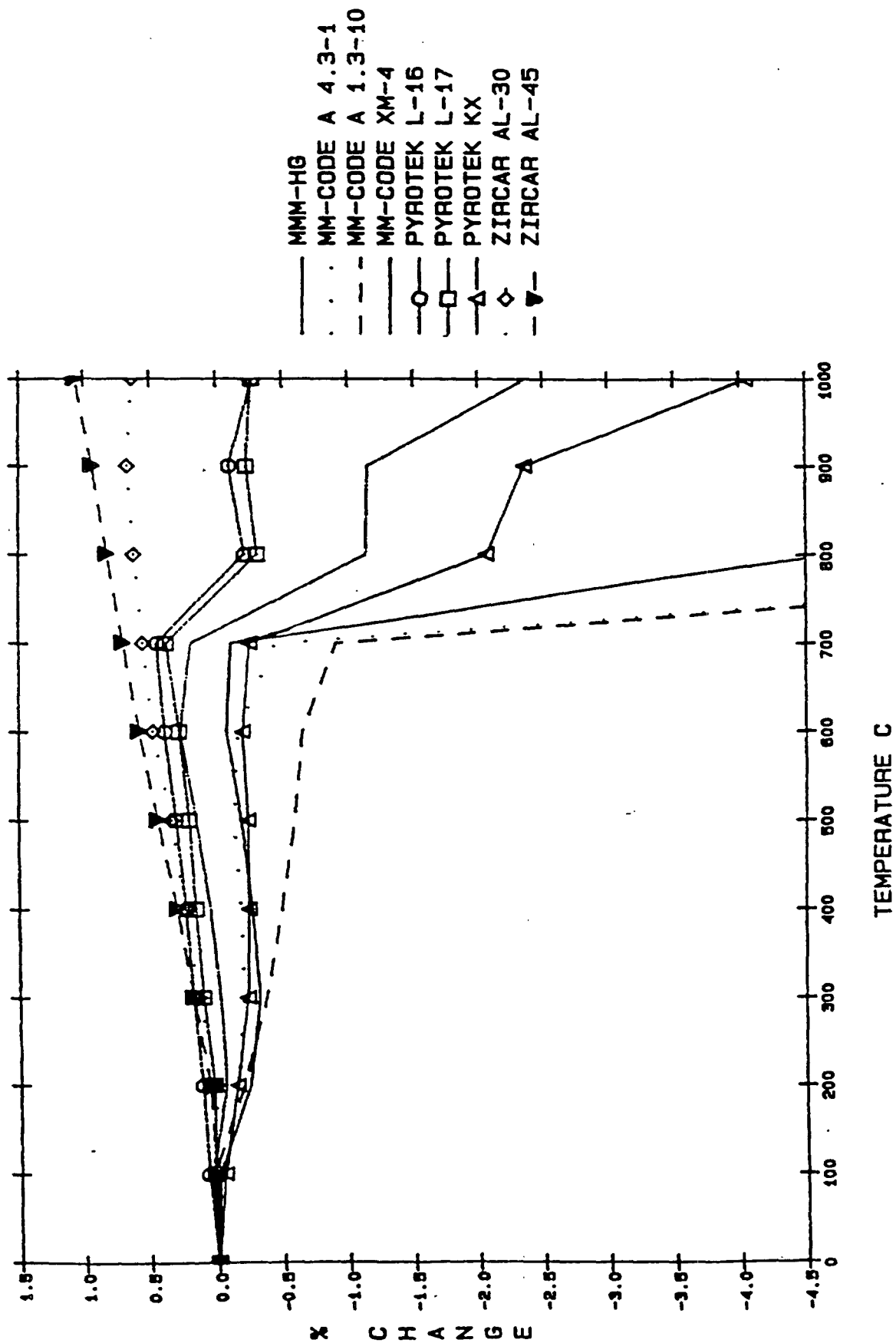


FIGURE 10. Comparison of Thermal Change Behavior for Various Header Materials - Normal to the Board Plane.

R44B PYROTEK L-18

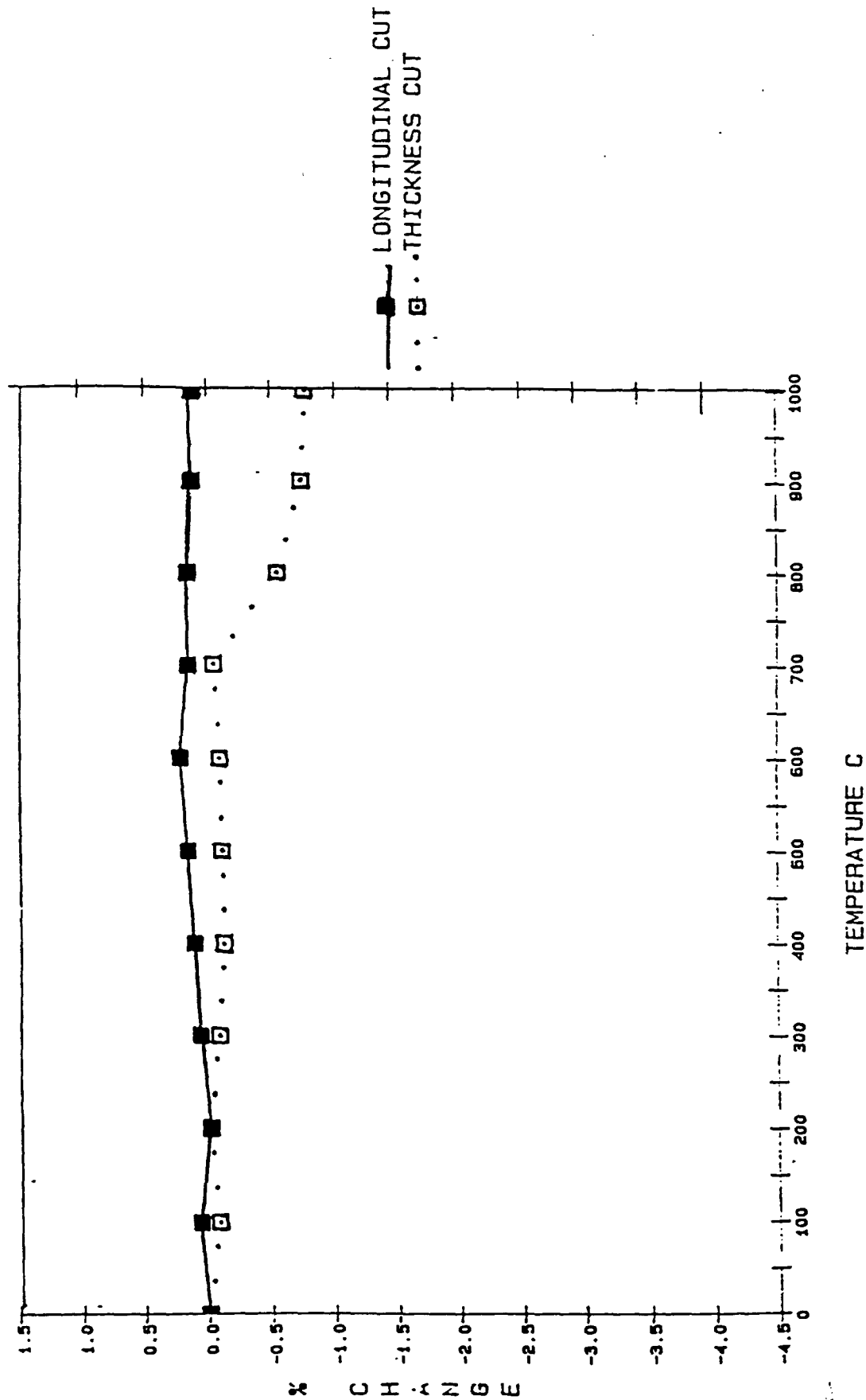


FIGURE 11. Thermal Change Behavior for L-18 Material - Parallel and Normal to the Board Plane.

RELAXATION UNDER LOAD 74F 400PSI

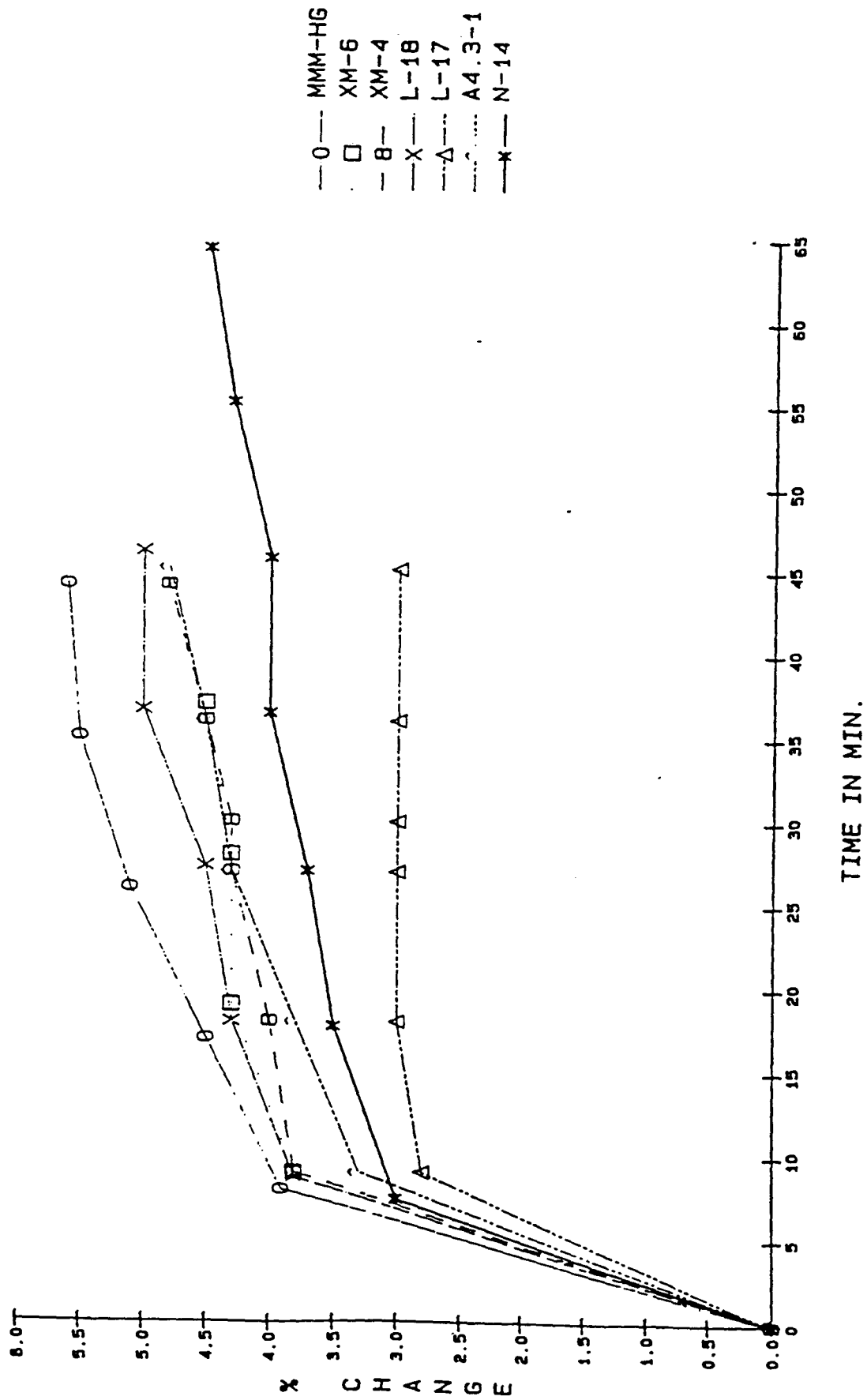


FIGURE 12

RELAXATION UNDER LOAD 1100F 400PSI

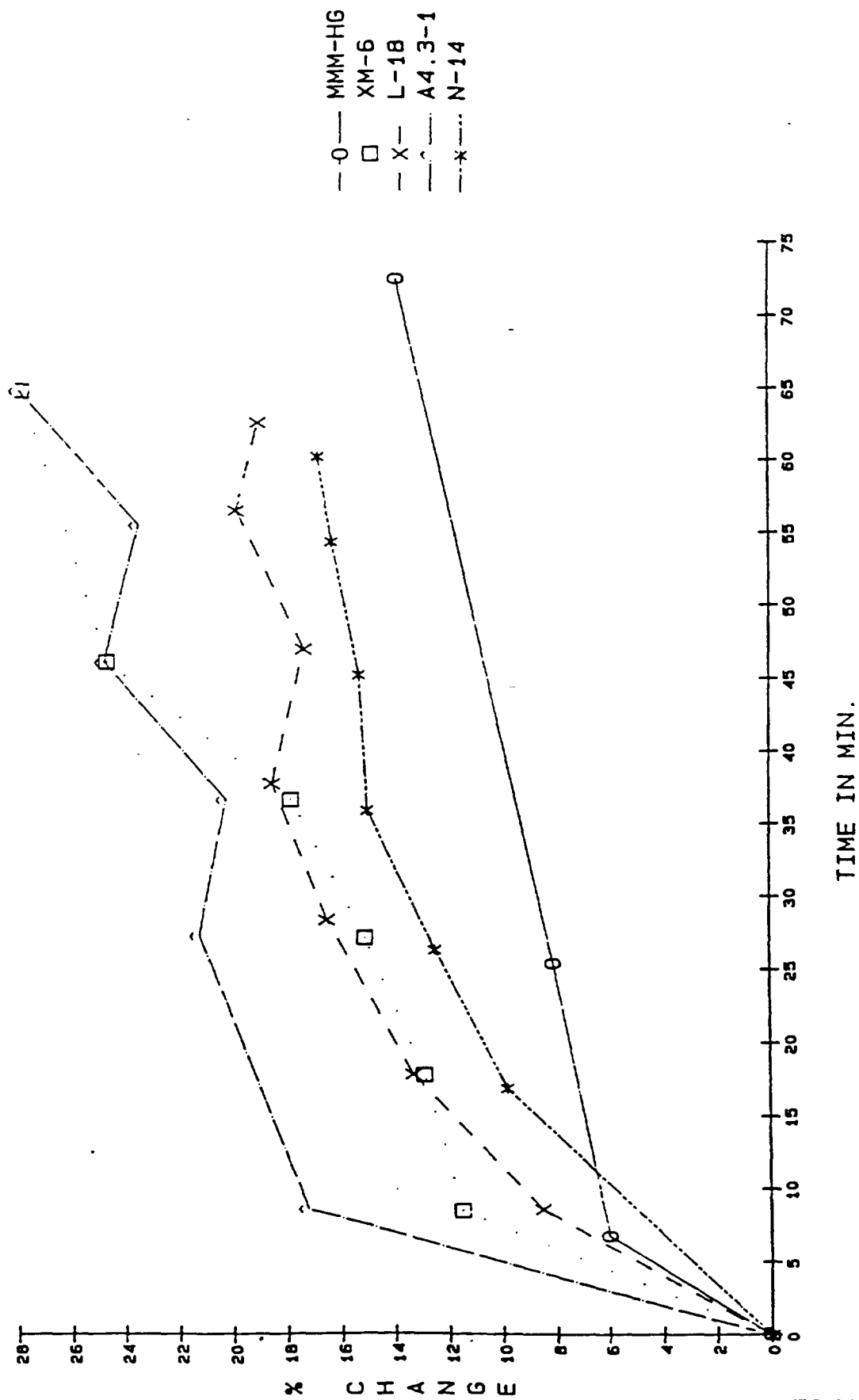


FIGURE 13

RELAXATION UNDER LOAD 1400F 400PSI

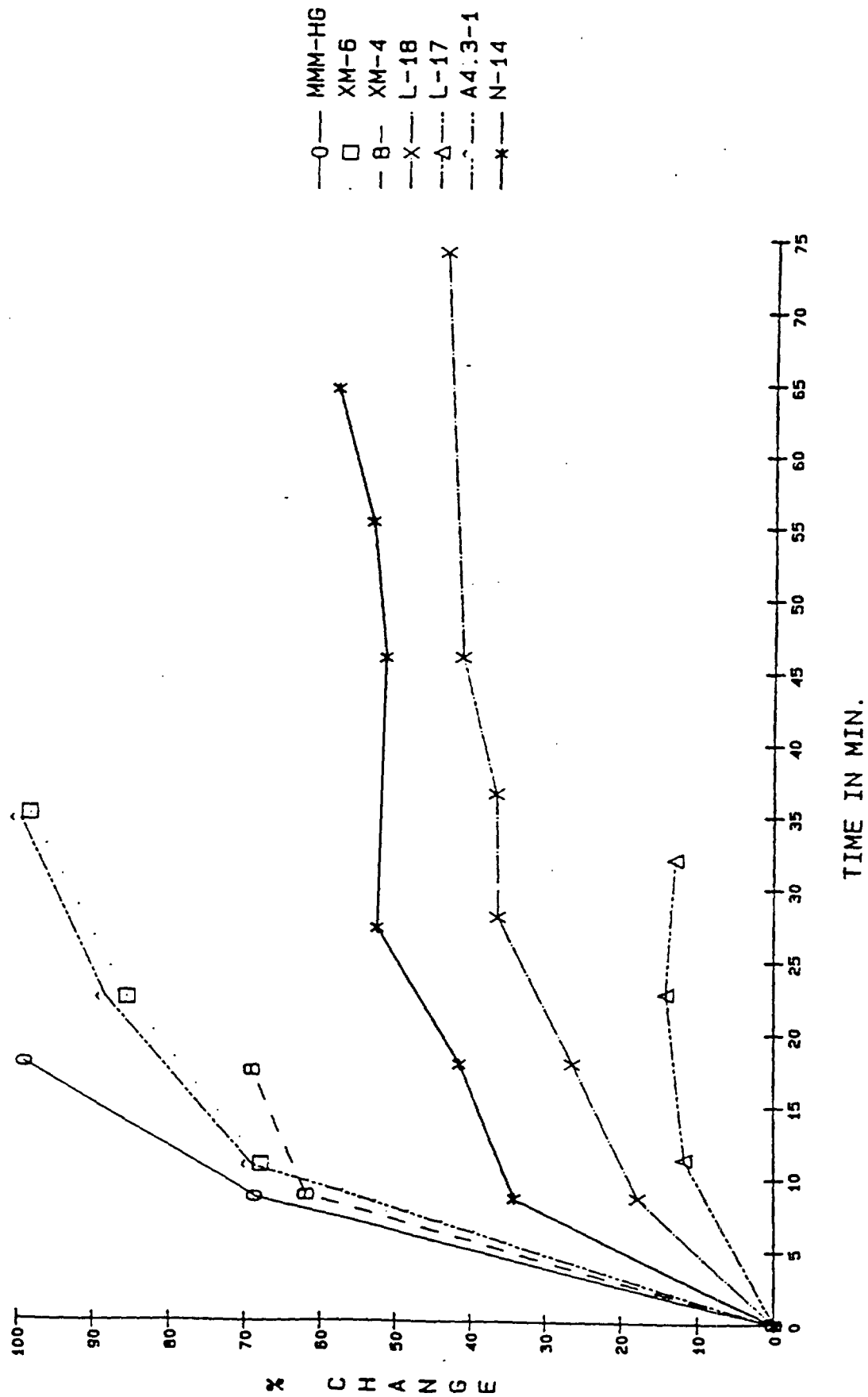


FIGURE 14

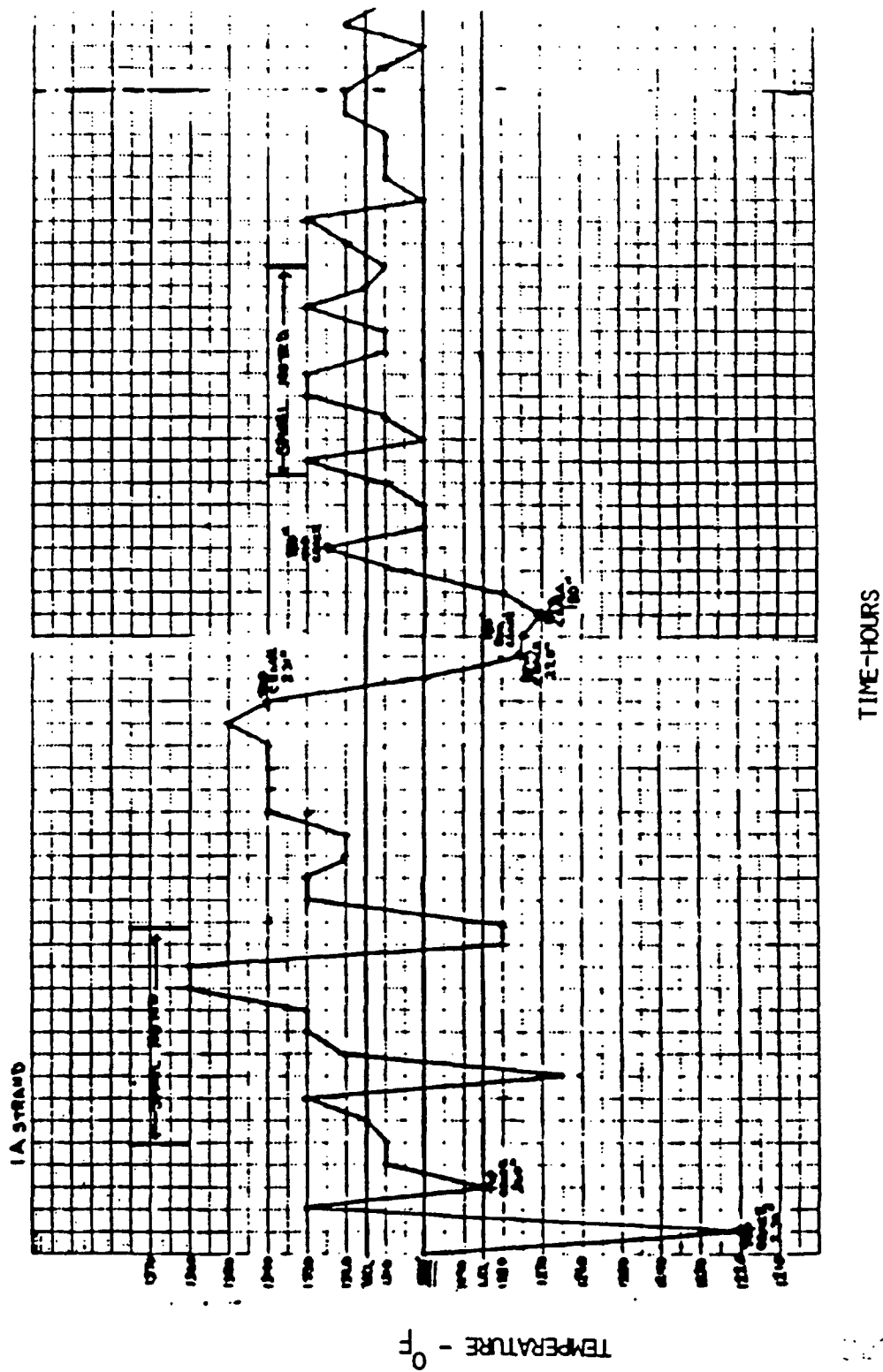


FIGURE 15. PROCESS TEMPERATURE DATA RECORDED AT VARRICK SHOWING METAL TEMPERATURE FLUCTUATION WITH TIME.

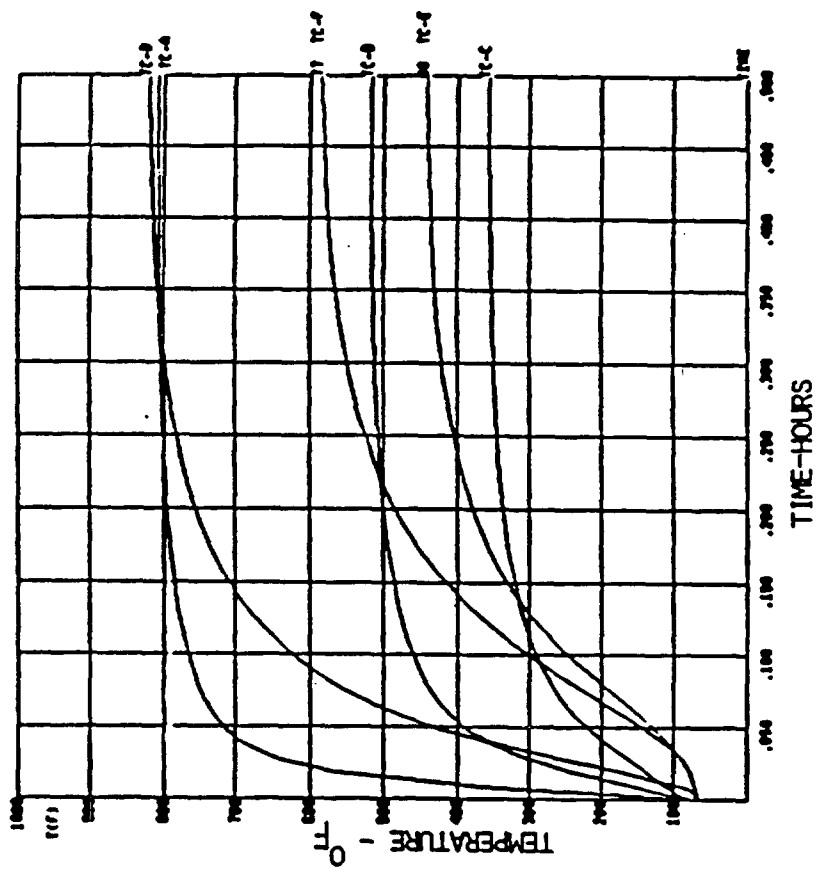


FIGURE 16 - TRANSIENT TEMPERATURES IN HEADER AT THERMOCOUPLE LOCATIONS (METAL CONTACT TO OIL RING)

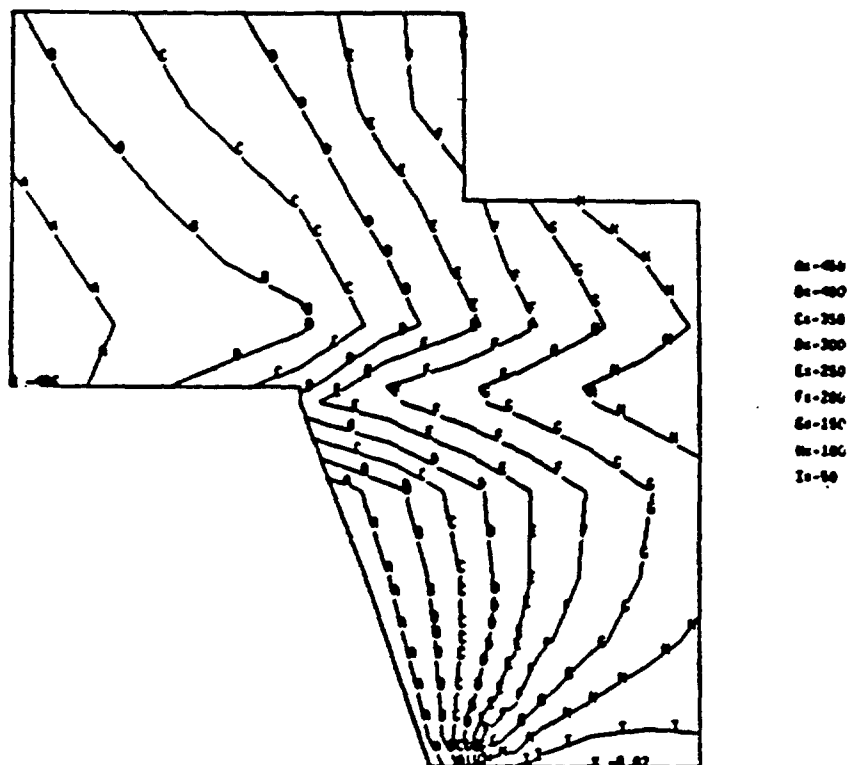
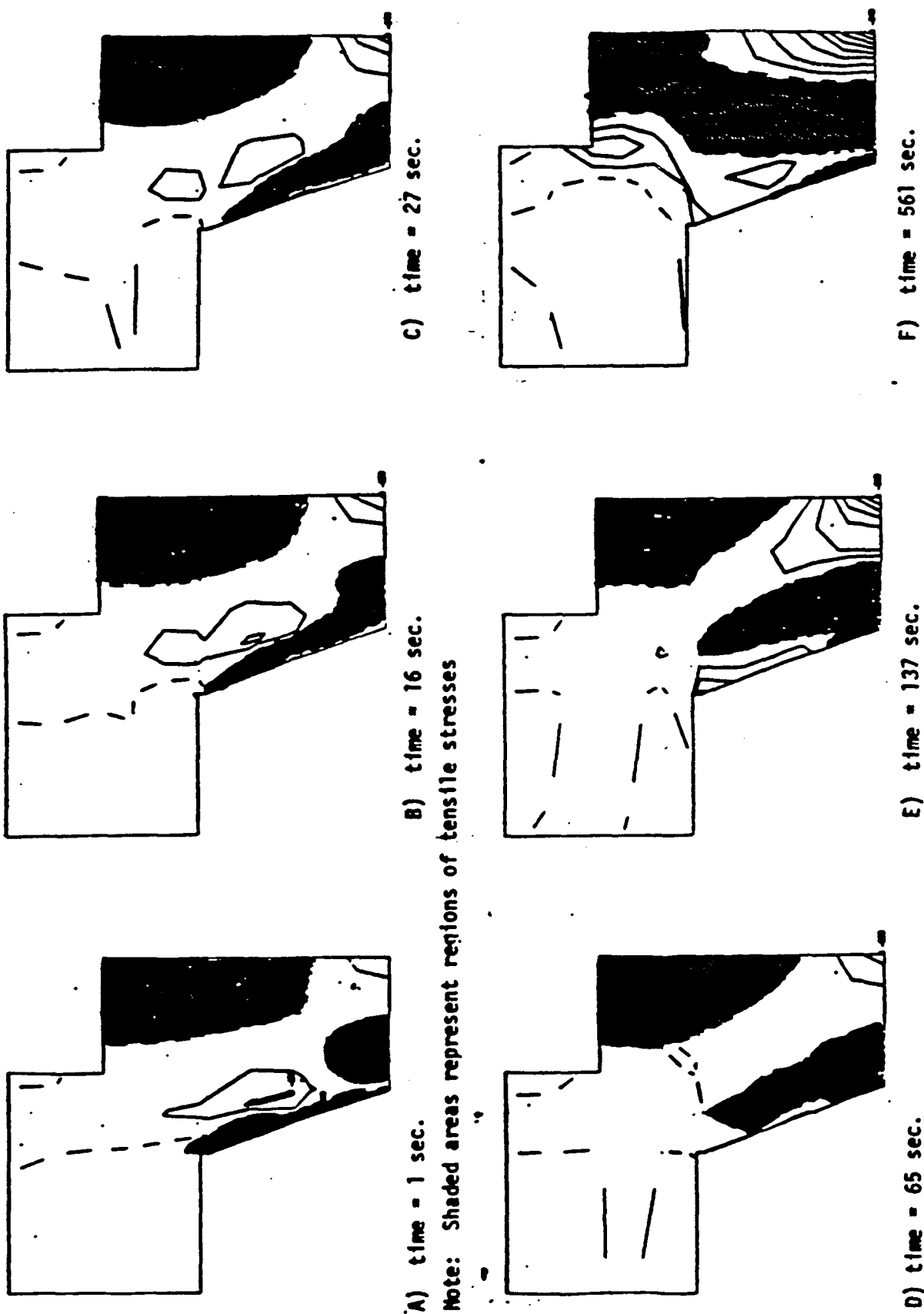


FIGURE 17 - STEADY STATE CIRCUMFERENTIAL STRESSES
FOR "FIXED" BOUNDARY CONDITION

FIGURE 18 - ZONES OF TENSILE STRESSES IN CLAMPING DIRECTION AN IDC HEADER DURING AND AFTER START-UP.



APPENDIX
SEM PHOTOMICROGRAPHS OF
NEW AND USED HEADER MATERIALS

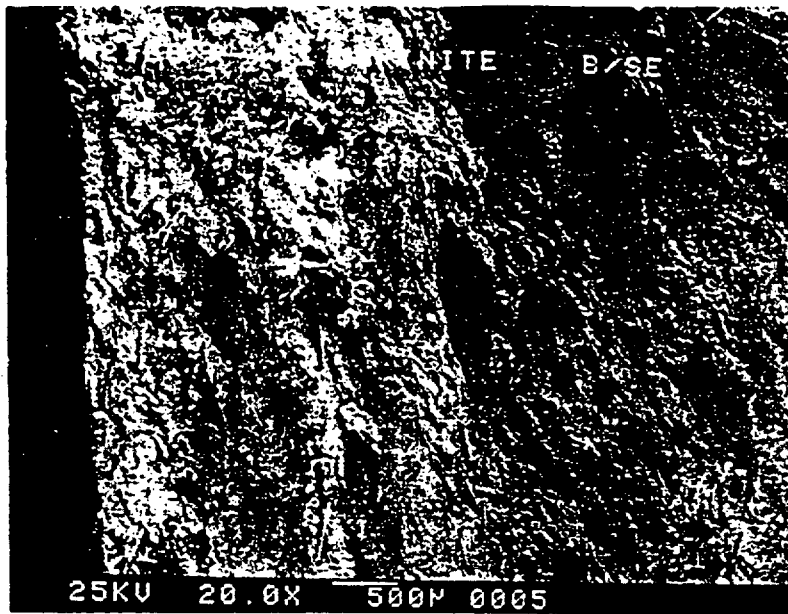
APPENDIX

SEM Photomicrographs of New and Used Header Materials

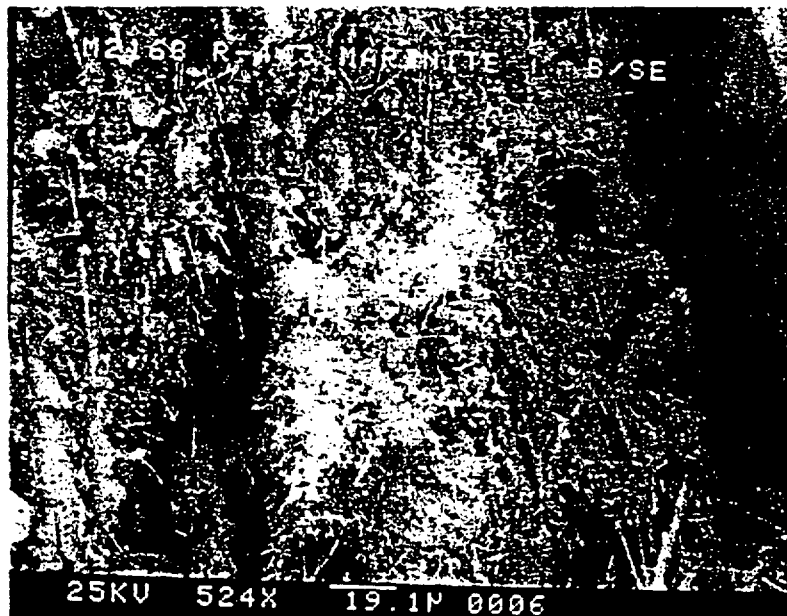
<u>Material</u>	<u>Mag</u>	<u>Comments</u>
MMM-HG (New)	20X	Relatively high concentration of oriented fibrous bundles without an obvious continuous binder phase (Figure A-1).
	524X	Fluffy binder phase interdispersed with fibers (<5 μ m) (Figure A-1).
MMM-HG (Used- Overhang Area)	30X	Material has a more monolithic appearance. Orientation of fibrous bundles is still obvious (Figure A-2).
	400X	Small fibers still retain original morphology. An amosite pencil also retains its unique size (cross section 25 x 50 μ m) and shape. It acts as a flaw in the overall matrix and could be a cause of premature failure in the highly stressed overhanging region (Figure A-2).
	400X	Fiber bundles retain original morphology. Binder phase seems to agglomerate around the individual fibers and bundles (Figure A-3).
	1000X	Highly magnified view of fibers and associated binder agglomerates (Figure A-3).
MM (New, A4.3-1)	1000X	Another example of agglomerated matrix around asbestos fibers (Figure A-4).
	20X	A more continuous matrix. Fiber reinforced concentrations are lower than MMM-HG (Figure A-5).
	100X	Fibers and relics of kraft paper (cellulose) are highly oriented. The fibers are relatively large in diameter (5-10 μ m) compared to asbestos fibers and do not occur in bundles (Figure A-5).
	1000X	Voids are created in the more continuous binder phase by the kraft paper which retains a flat appearance about 60 x 10 μ m in cross section (Figure A-6).
MM (Used, A3.1-4)	30X	Fracture surface shows a porous matrix with a lower fiber content than MMM-HG. Fibers still retain their morphology (Figure A-7).

Appendix - continued

L-17 (New)	1000X	Matrix is similar to N-14 (Figure A-14).
L-17 (Used)	30X	Fiber concentration is decreased at the tip because of possible oxidation. Matrix is heavily sintered (Figure A-15).
L-17 (Used Tip)	200X	Matrix is heavily sintered at the tip. Carbon fibers are no longer visible (Figure A-15).
	400X	Tiny fibers in the matrix are no longer visible (Figure A-16).
L-17 (Used) Interior)	100X	Reinforcement is still present in the interior. Sintered texture of matrix is predominant throughout the cross section of the header (Figure A-16).
L-18 (New)	20X	Carbon fiber reinforcement is similar to L-17. Fibers seem to be shorter and less concentrated than L-17. The matrix is continuous but more open than L-17 (Figure A-17).
	521X	The matrix is composed of minute fibers similar in morphology to L-17 (Figure A-17).
L-18 (Used)	-	No trial use as yet.



20X

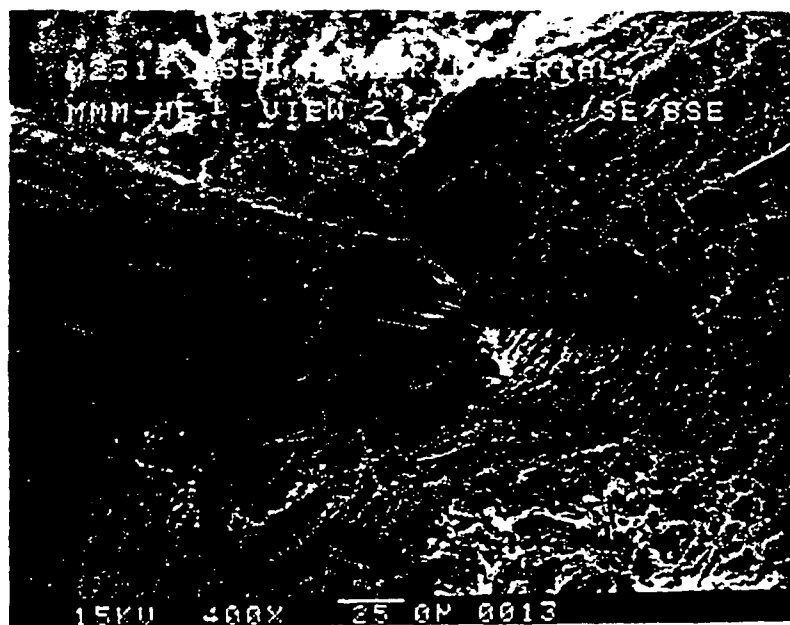


524 X

Figure A-1 - SEM Photos of New MMM-HG Fracture Surfaces



30X

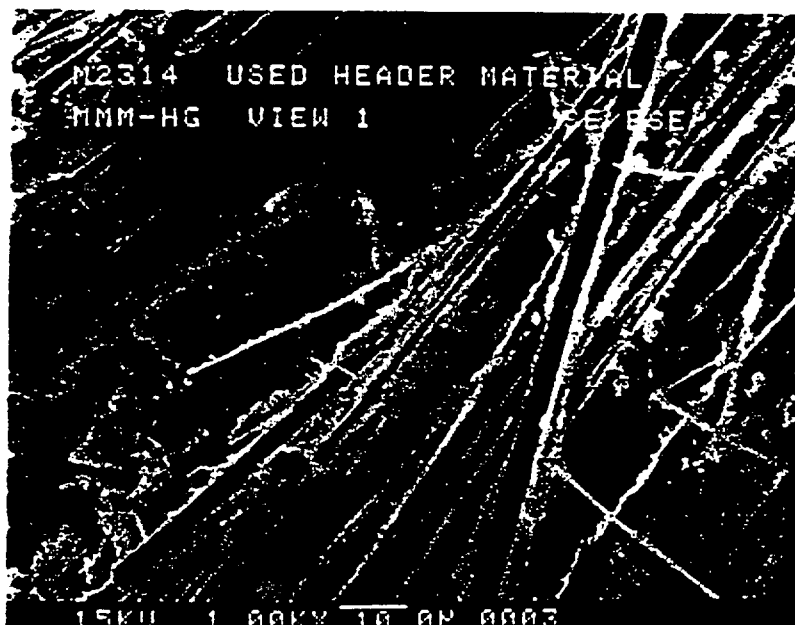


400X

Figure A-2 - SEM Photos of Used MMM-HG Fracture Surfaces

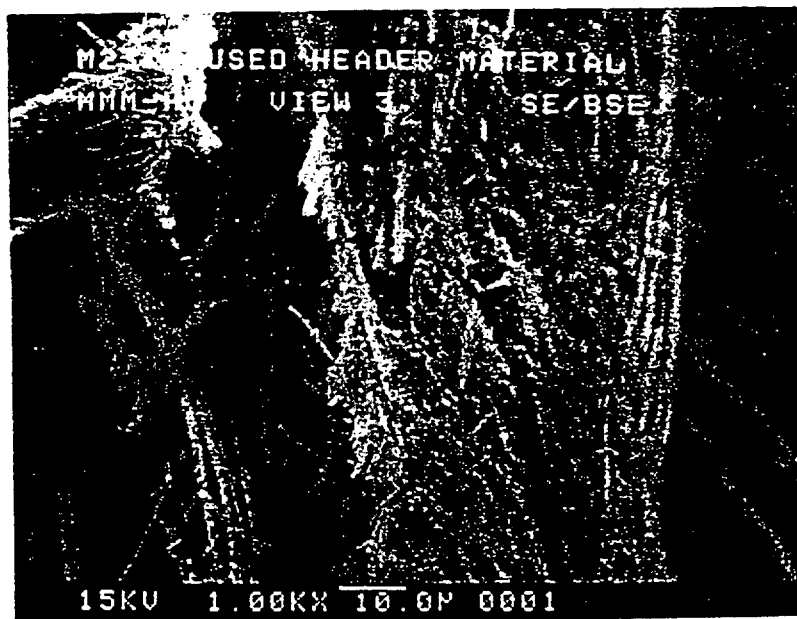


400X



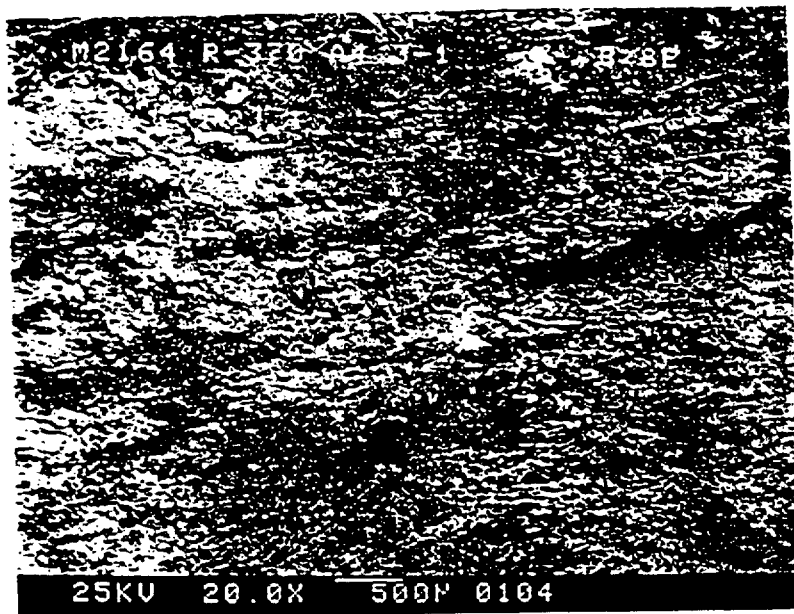
1000X

Figure A-3 - SEM Photos of Used MMM-HG Fracture Surfaces

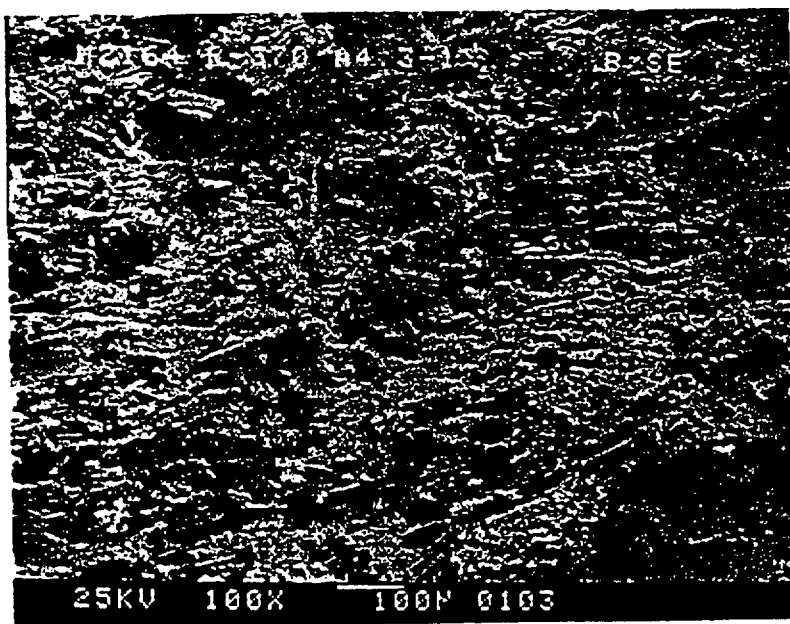


1000X

Figure A-4 - SEM Photos of Used MMM-HG Fracture Surfaces



20X



100X

Figure A-5 - SEM Photos of New MM (A4.3-1) Fracture Surfaces



1000X

Figure A-6 - SEM Photos of New MM (A4.3-1) Fracture Surfaces



30X



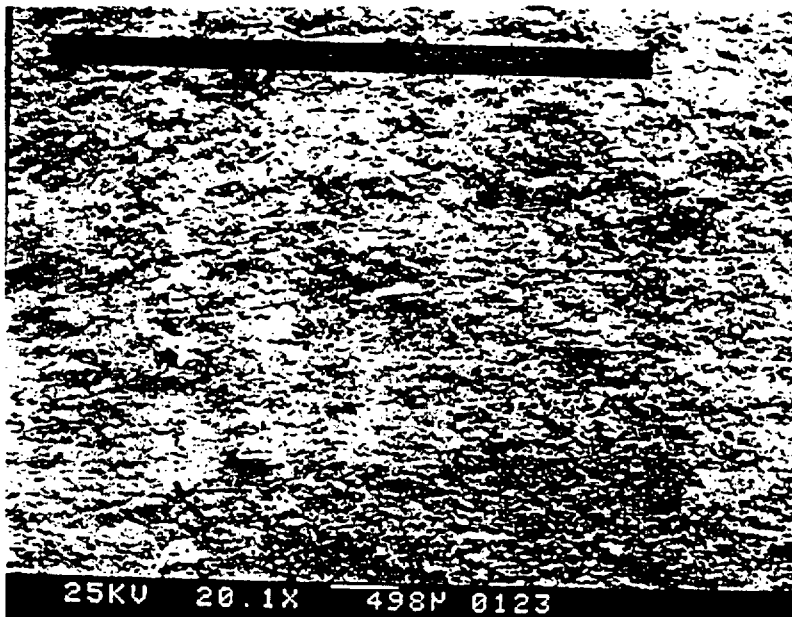
200X

Figure A-7 - SEM Photos of Used MM (A3.1-4) Fracture Surfaces



1000X

Figure A-8 - SEM Photos of Used MM (A3.1-4) Fracture Surfaces

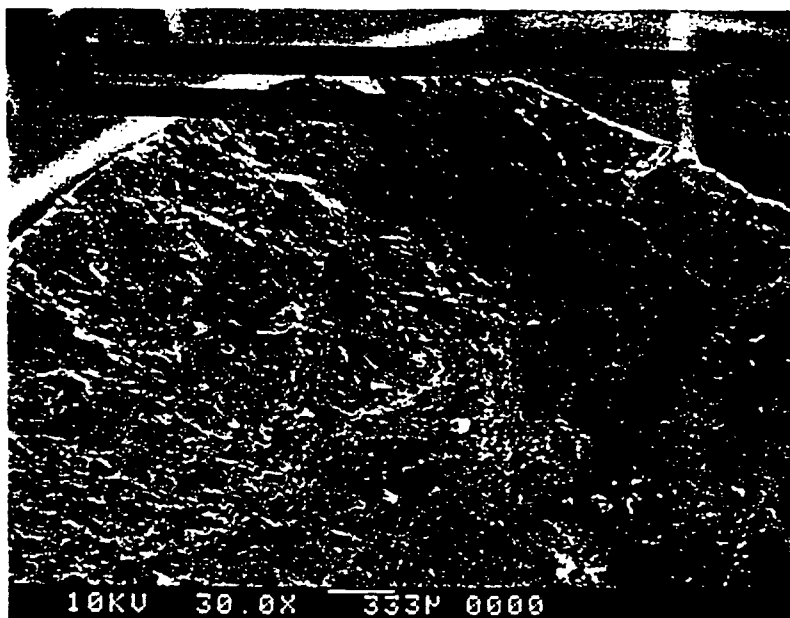


20X

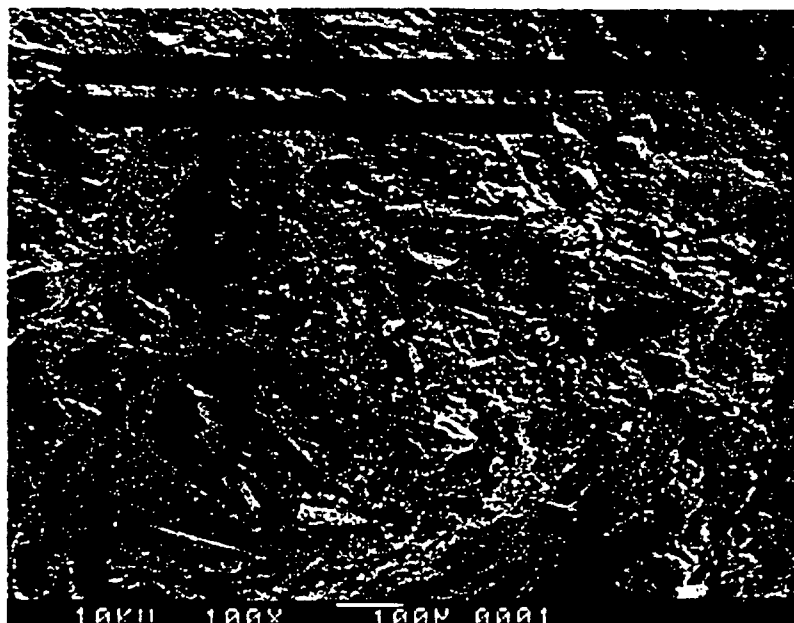


525X

Figure A-9 - SEM Photos of New XM-6 Fracture Surfaces

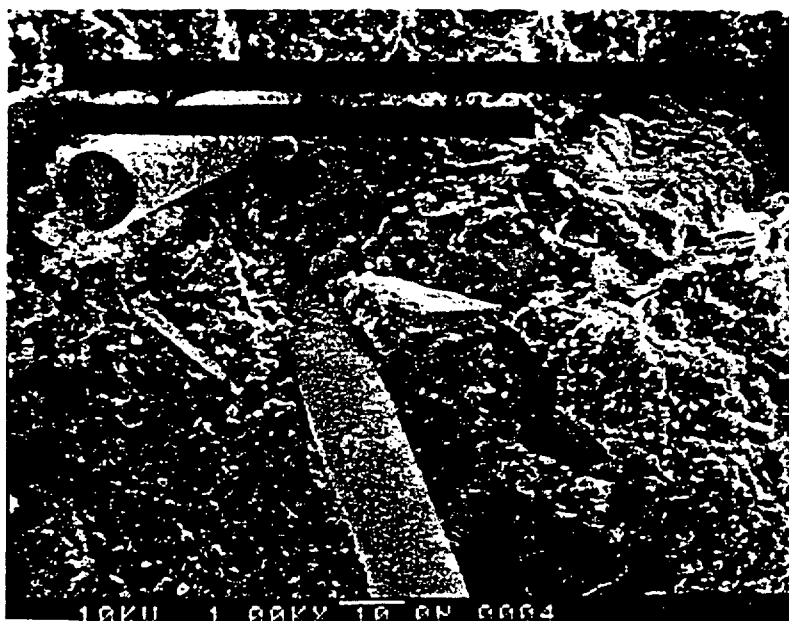


30X



100X

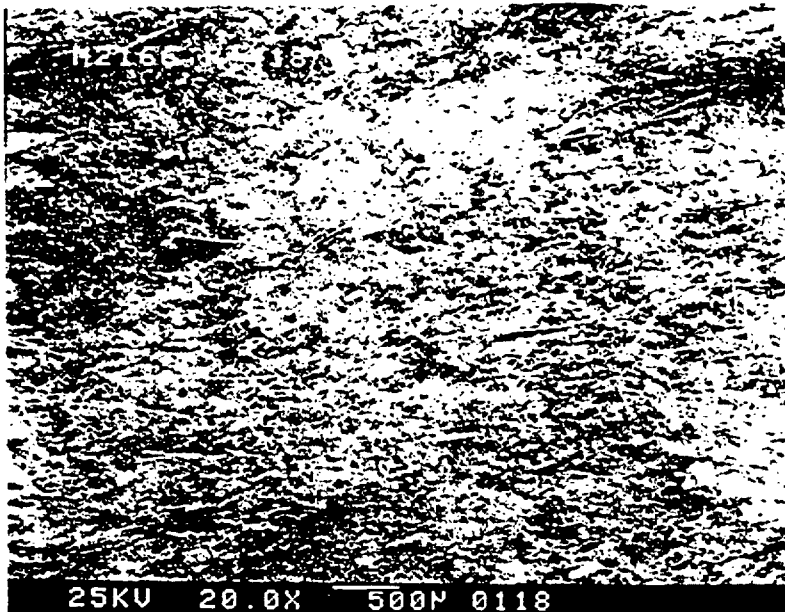
Figure A-10 - SEM Photos of Used XM-4 Fracture Surfaces



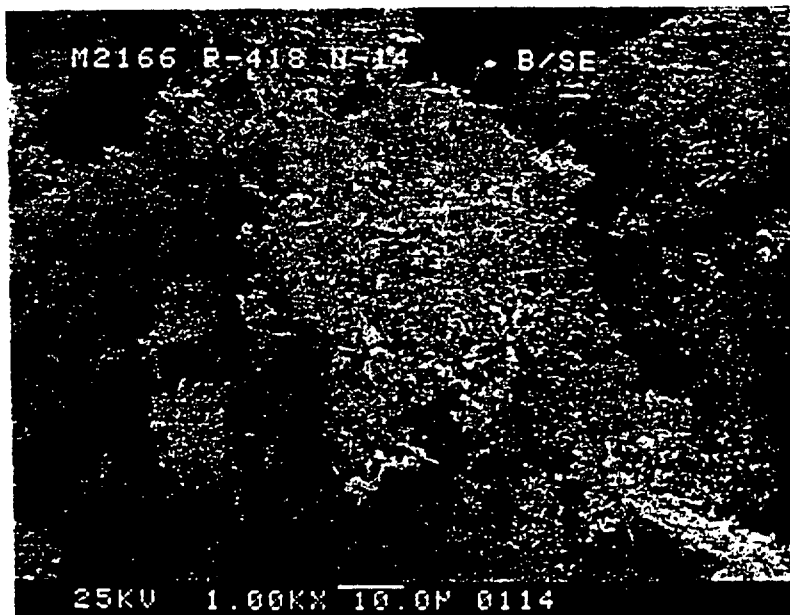
1000X

Figure A-11 - SEM Photos of Used XM-4 Fracture Surfaces

1000X

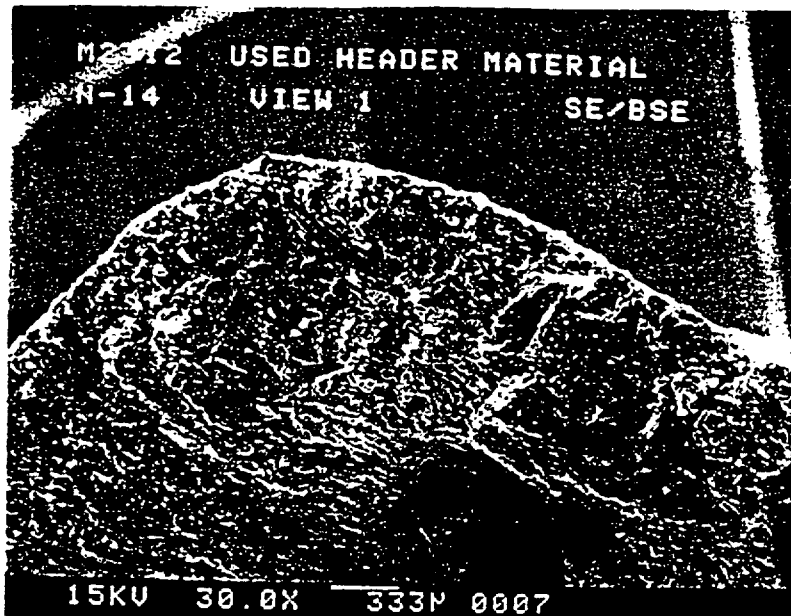


20X



1000X

Figure A-12 - SEM Photos of New N-14 Fracture Surfaces

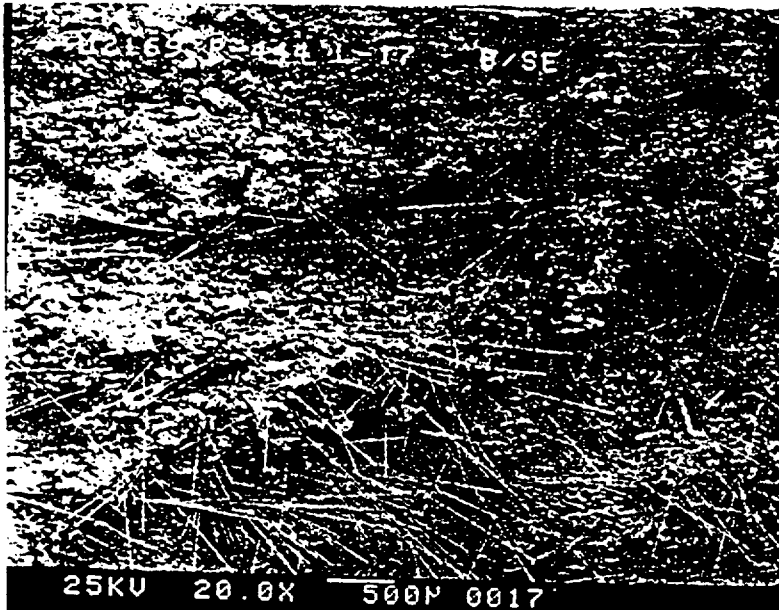


30X



1000X

Figure A-13 - SEM Photos of Used N-14 Fracture Surfaces

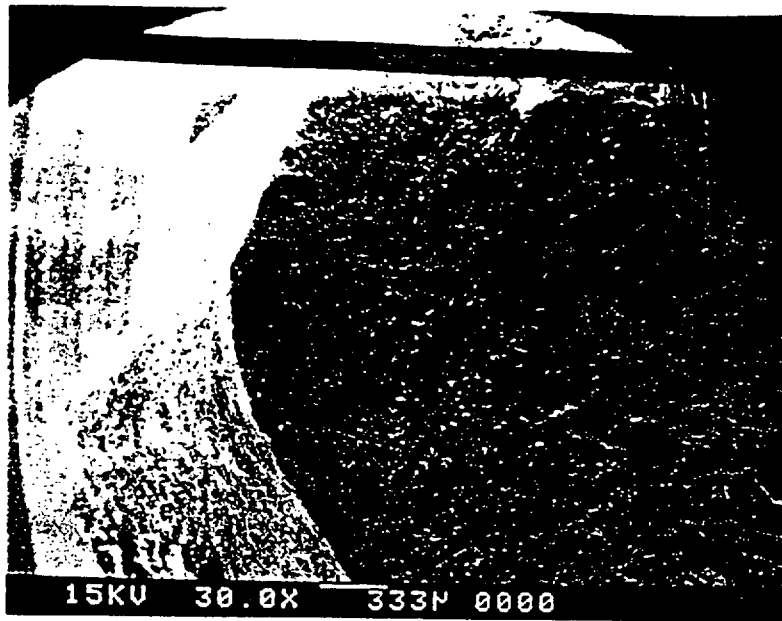


20X

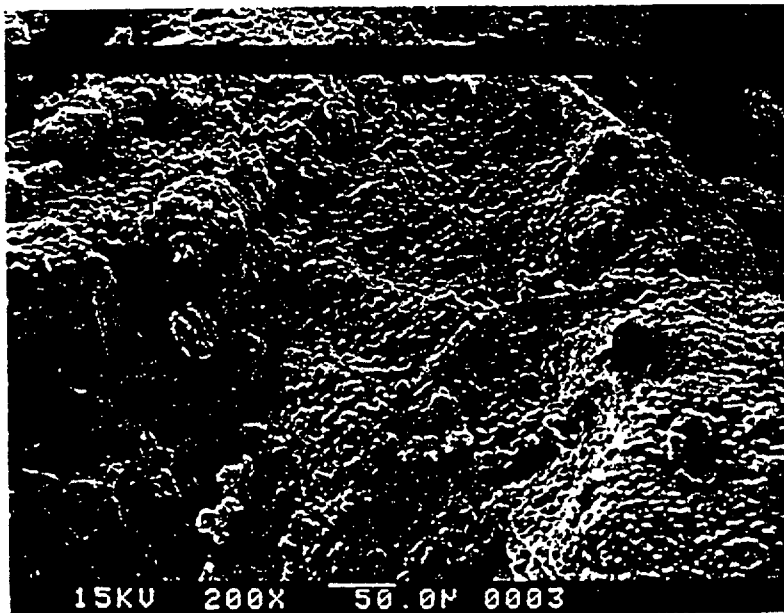


1000X

Figure A-14 - SEM Photos of New L-17 Fracture Surfaces

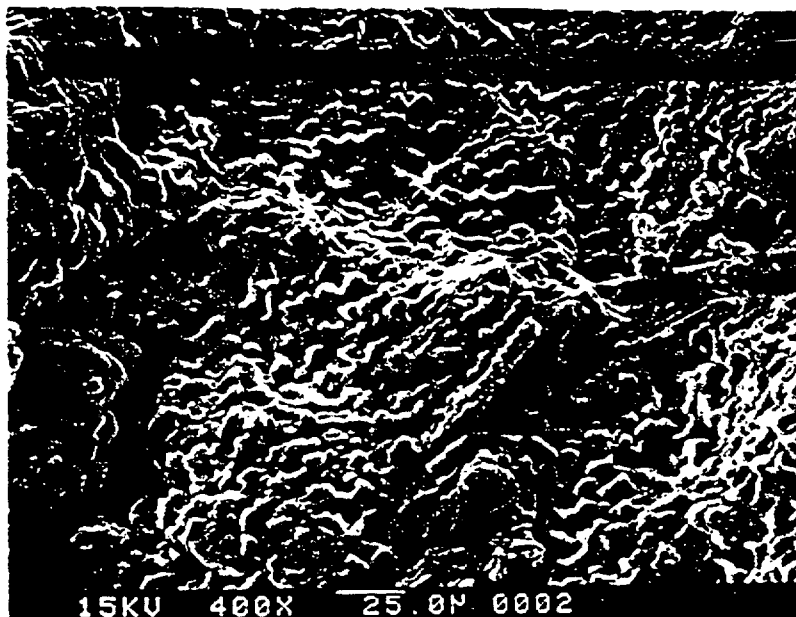


30X



200X

Figure A-15 - SEM Photos of Used L-17 Fracture Surfaces



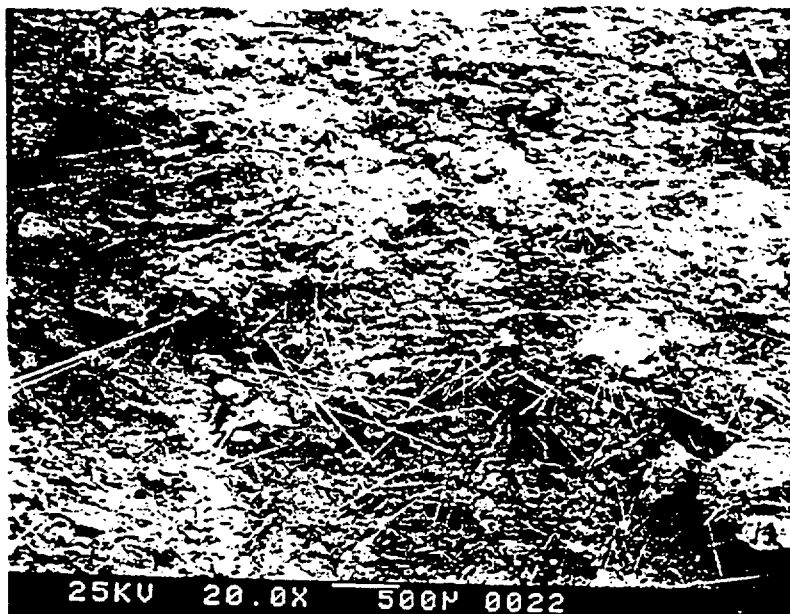
400X - Tip



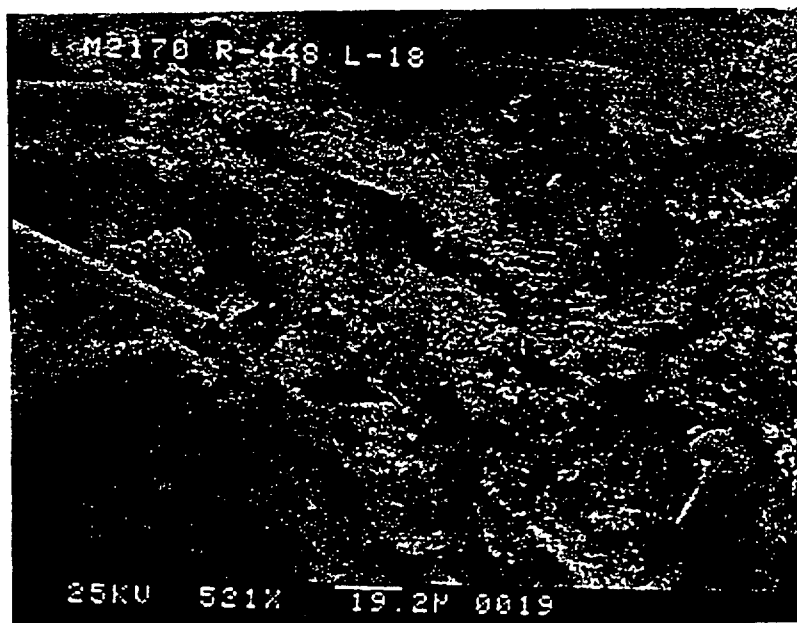
100X - Interior

8018538

Figure A-16 - SEM Photos of Used L-17 Fracture Surfaces



20X



521X

1146533

Figure A-17 - SEM Photos of New L-18 Fracture Surfaces

FROM M. W. VANCE
CERAMICS DIVISION
ALCOA TECHNICAL CENTER

TO MEMORANDUM

CONFIDENTIAL

1985 October 10

RE: DIVISION REPORT - "EVALUATION OF CANDIDATE MATERIALS FOR HDC INGOT
CASTING HEADER APPLICATION AT WARRICK OPERATIONS"

This report summarizes a program intended to improve the understanding and data base for selection of new HDC header materials as substitutes for asbestos containing Molten Metal Marinite.

To date, two new candidate calcium silicate bonded materials have been identified for future trials at Warrick Operations. Although some of the properties presented in this report may be used for header material quality control purposes, material consistency should be addressed as a separate item.

It is intended that this information may also be used as background for selection of headers for other HDC and FDC casting operations, for headers for FDC casting Alithalite alloys, and for potential selection of materials for other applications involving molten metal contact such as: troughs, basin linings, floats and dams.

Future ATC involvement will consist of performing limited testing, making recommendations for implementing substitute materials, and continued participation in the HDC Header Task Force at Warrick Operations.

Further inquiries should be addressed to the writer at 8-221-2815, Alcoa Technical Center.

M. W. Vance
M. W. VANCE

TH:MB:11

0035109



ATC 0035109

SP-4563 (REV 11-68)

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REINFORCEMENT OF ALUMINUM WITH WHISKERS
AND FIBERS OF BORON NITRIDE, ASBESTOS,
ALUMINA AND GRAPHITE

By C. N. Cochran

Phys. Chem. Div.
Report No. 8-68-3

March 29, 1968
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March 29, 1968
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Aluminum Company of America
Alcoa Research Laboratories
Physical Chemistry Division

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Signed C.N. Cochran
C. N. Cochran, Chief
Physical Chemistry Division

Date - March 29, 1968

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ABSTRACT

Composite tensile specimens of boron nitride paper in high purity aluminum, R414 Safibers in 2025 alloy, graphite yarn in 2025 alloy (aged to the T6 condition) and alumina whisker tape in 2219 alloy were prepared by a high pressure liquid metal infiltration technique. In all but the boron nitride reinforced sample, the composites were weaker than the unreinforced alloys. The 5600 psi gain in tensile strength due to the boron nitride is equivalent to a tensile strength of 72 ksi for this material. The poor performance of the alumina whisker tape was unaccounted for. The R414 Safibers were probably weakened in an attempt to enlarge the crystals by firing to 1850°C. The graphite yarn reacted partially with the aluminum to form aluminum carbide. Microcrystalline asbestos fibers were incorporated into prealloyed 2024 powder by hot pressing but did not provide the claimed improvement in elongation.

INTRODUCTION

Hot pressing of aluminum powder and infiltration with liquid aluminum (described in Report 8-67-8) were used to incorporate whiskers and fibers of boron nitride, asbestos, alumina and graphite into aluminum. A stronger bomb of 310 stainless steel (Figure 1) was constructed to permit liquid metal infiltration at pressures up to 2500 psi at 1000°C. The high pressure reduced the porosity of castings by permitting metal infiltration in channels between whiskers, as narrow as 0.07 microns. Infiltration was performed at 750°C rather than at 1000°C to reduce strain in the bomb. The boron nitride mold (Figure 2) was elongated by 1/2" to give a tensile specimen with a 1-1/2" long reduced section area. The watted seal between the aluminum and alumina tube was formed at temperatures of 1000 to 1100°C in vacuum before infiltration. For the most part, alloys in the 2000 series were employed rather than high purity aluminum, but the castings were not heat treated in all cases.

The results for the specimens prepared for the present report are given in Table I. Summaries of the reinforcing agents and the composite tensile specimens included in this study to date are listed in Tables II and III.

BORON NITRIDE

Boron nitride paper obtained from Mr. Lynn Ogden of the Carborundum Company was infiltrated with high purity aluminum at

230 psi. The yield, tensile, and elongation of the specimen containing 0.233 volume fraction boron nitride and 2.1% porosity were, respectively, 5,300 psi, 10,500 psi and 10%. Transverse and longitudinal sections of this sample in Figure 3 show that the orientation approximates a two-dimensional random array. Using 6,600 psi as the tensile of high purity aluminum cast in the same fashion, a tensile strength of $\frac{3[10,500 - (1-0.233-.021)6600]}{0.233} =$ 72 ksi was calculated for the boron nitride for this orientation. This compares to tensile strengths of 75-120 ksi claimed for this material in the literature.⁽¹⁾ The other constituents showing in the photomicrograph have not been identified but may be Al-B or Al-N compounds.

ASBESTOS

During a visit to these Laboratories in the summer of 1967, Dr. A. O. Battista of Food Machinery Corporation offered to supply "Avibest" microcrystalline asbestos fibers for incorporation in aluminum. These are not strong fibers, but an improvement in elongation of aluminum was claimed. It was necessary to employ hot pressing of aluminum powder-asbestos mixtures for incorporation because the asbestos contains water of

(1) Donald G. Sturges, Steel S-1 to S-6 (May 30, 1966).

hydration which would be released above 660°C if molten aluminum were employed.

Chemical treatment of the powder is required to permit mixing of the aluminum powder and asbestos. A quantity of pre-alloyed 2024 powder supplied to Food Machinery Corporation was treated, mixed with the "Avibest" and returned to Alcoa. Mixtures of 10 and 25 wt. % "Avibest" with 2024 powder as well as treated 2024 powder without "Avibest" were pressed into tensile specimens by Mr. J. H. Dudas. The specimens were sintered 45 minutes at 1100°F in nitrogen, cooled to 920°F and cold-water quenched. Samples of each material containing "Avibest" were aged to the T6 temper. The tensile strengths were only 400 psi for the specimens containing "Avibest," and the elongation could not be evaluated. The treatment of the 2024 powder required for mixing with the "Avibest" evidently caused deterioration because the tensile strength of one compact from the treated powder without "Avibest" was only 1,400 psi, and the other broke before it could be tested.

ALUMINA

Samples of R414 polycrystalline alumina "Safibers" with smaller diameters (approximately 20 microns) than previously available were received from Mr. A. Pearson of the Alumina and Chemicals Division. One lot that had been reheated to 1850°C to promote grain growth proved disappointing in giving a tensile

strength of only 4,100 psi in a 2025 alloy composite. This is less than expected for the matrix alone. Tests will be made with a second lot which is the same material as in the first lot but without the grain growth treatment at 1850°C.

A quantity of alumina sapphire whiskers in a PIB rubber tape was obtained without charge from General Technologies, Inc. The high weight fraction (0.44) of whiskers in the tape raised hopes of producing a composite with a high volume fraction of reinforcing agent. However, the low density of the rubber matrix in the tape reduces the high weight fraction to a volume fraction of only about 0.15 in the tape. The rubber was burned away by firing in air at 1000°C, and the remaining whiskers were used to reinforce 2219 alloy (0.124 volume fraction). The infiltration was not successful because tensile strength (10,400 psi) well below that of the matrix alone was obtained. There appears to be no advantage of the tape over the whisker mats previously purchased from Thermo-Kinetic Fibers, Inc., a subsidiary of General Technologies, Inc.

GRAPHITE

A free sample of Thornel-50 graphite yarn obtained from Union Carbide Corporation was incorporated into 2024 alloy, and the composite sample solution was heat treated and aged to the T6 temper. Metal grade alumina ore was piled over the mold beneath

the aluminum to prevent reaction with the graphite during the 1000-1100°C treatment to form the Al-Al₂O₃ seal prior to infiltration. Vaporization of magnesium during evacuation prior to infiltrating changed the matrix composition nearer to 2025 alloy. The yarn, consisting of 1,440 filaments, each 7 microns in diameter, permitted packing to a high volume fraction of 0.336.

Polished transverse and longitudinal sections (20X, 100X, and 500X) in Figure 4 show that the graphite was pushed to one side of the section during infiltration, but good longitudinal orientation of the fibers was obtained. The grey areas surrounding the fibers and the needles in the matrix in the 500X section may be aluminum carbide. The modulus of 50×10^6 pounds/inch², tensile strength of 400,000 psi, and density of 1.55 g/cc make this material extremely attractive for reinforcing aluminum. However, the density of 2.643 g/cc for the sample corresponds to 8.6% volume contraction during infiltration which may indicate reaction to form aluminum carbide. If all of the graphite in this specimen reacted to form aluminum carbide, the theoretical contraction would be 16.1%. The tensile strength of the composite was 20,800 psi, which is below that for the matrix alone. The modulus of 24.3×10^6 psi is identical with the value calculated from the rule of mixtures for this composition.

Powder diffraction analysis (Film B54371) showed about 40% Al, 40% Al_4C_3 , and 20% graphite while chemical analyses (68-011810) showed 7.1% Al_4C_3 . In an attempt to reduce Al_4C_3 formation in Run 11418-44, the infiltration temperature was reduced from the 785°C used in previous runs to 705°C, and the time at temperature before the start of casting reduced from about 1 hour to 2 minutes. This reduced the carbide to about 25% (Film B54424), but the sample did not release from the mold and broke.

SILICON CARBIDE

Photomicrographs of longitudinal and transverse sections (500X) of #13 alloy containing 0.103 volume fraction Carborundum SiC whiskers and 0.9% porosity are shown in Figure 5. This specimen had been described in Report 8-67-8. The alignment is predominantly a two-dimensional random array with a slight trend toward uniaxial alignment. With 22,100 psi for the tensile strength of unreinforced #13 alloy cast in a similar specimen, the tensile strength of the silicon carbide is $\frac{3[23,900 - (1.0 - 0.112)22,100]}{0.103} = 126,000$ psi.

TABLE I
REINFORCEMENT OF ALUMINUM WITH WHISKERS AND FIBERS

----- Reinforcing Agent -----										----- Properties of Tensile Specimen -----					
Matrix	Reinforcement	Dia. Microns	Length Microns	Density g/cc	Claimed Modulus psi x 10 ⁻⁶	Derived Tensile Strength psi	Weight Fraction		g/cc	% Porosity	Yield psi	Tensile psi	Elongation in 1/2"	Reference	
							Reinforcing Agent	Volume Fraction Reinforcing Agent							
High Purity Al	Carborundum BN Paper	3-8		1.85	8-10	72,000 ⁽¹⁾	0.172	0.233	2.554	2.1	5,300	10,500	10	12339-31 ⁽²⁾	
2024 ⁽³⁾	{ FMC "Avibest" Microcrystalline Asbestos }	0.5					0.10					400		CRD 8214	
2024 ⁽³⁾							0.25					400		CRD 8215	
2024 ⁽³⁾	None						0	0				1,400		CRD 8213	
2025	ESL R414 Saffibars 4670G-2 (1850°C)	20	300-1000	4.00			0.370	0.275	2.980	5.5		4,100		12339-48 ⁽⁴⁾	
2025-R6	Union Carbide Thoronel 50 Graphite Yarn	~7		1.55	50	(400,000)	0.197	0.336	2.643	-9.31		20,800 ⁽⁵⁾	0	11418-40 ⁽⁴⁾	
2219	Gen. Tech. ⁽⁶⁾ Al ₂ O ₃ Whisker Tape	1-3	20-400	3.97	100-150		0.172	0.124	2.862	0 (assumed)		(10,400)		11418-38 ⁽⁴⁾	
2025-0	None						0	0	2.812	0	19,000	21,800	3.0	11418-18 ⁽⁴⁾	
(1) Random two-dimensional array assumed															
(2) Cast at 230 psi and 750°C															
(3) Preallayed Powder															
(4) Cast at 2500 psi and 750°C															
(5) Modulus of Elasticity is 24,300,000 psi															
(6) R1B Rubber Tape, 41 wt. % Type IB Whiskers															

(1) Random two-dimensional array assumed
(2) Cast at 230 psi and 750°C
(3) Prealloyed powder
(4) Cast at 2500 psi and 750°C
(5) Modulus of Elasticity is 24,300,000 psi
(6) FIB Rubber Tape, 41 wt. % Type IB Whiskers

TABLE II

SUMMARY OF REINFORCING AGENTS USED TO DATE IN PHYSICAL CHEMISTRY DIVISION

Agent	---Suppliers' Claims--- Modulus x 10 ⁻⁶ psi	Tensile, ksi	Density g/cc	Ultimate Diameter of Whisker or Filament	Whisker Length	Tensile Strength Developed in Al Matrix, ksi
TKF Sapphire Wool	100-300	2,000-3,500	3.97	1-3	500-5,000	400
TKF Sapphire Paper	100-300	2,000-3,500	3.97	1-3	500-5,000	400
GTC Sapphire Whisker Tape	100-300	2,000-3,500	3.97	1-3	20-400	400
ESL R414 Safibers				30-150	2,000-5,000	24
Carborundum SiC Whisker Tape	70	<3,000	3.17	0.5-3	100-750	120
Carborundum BN Whisker Mat	8-10	75-120	1.85	3-8		72
Texaco Boron Filaments	60	350	2.6	102	∞	Reacted with Al
FMC "Avibest" Microcrystalline Asbestos				0.5		0
Union Carbide Thornel 50 Graphite Yarn	50	400	1.55	~7		0

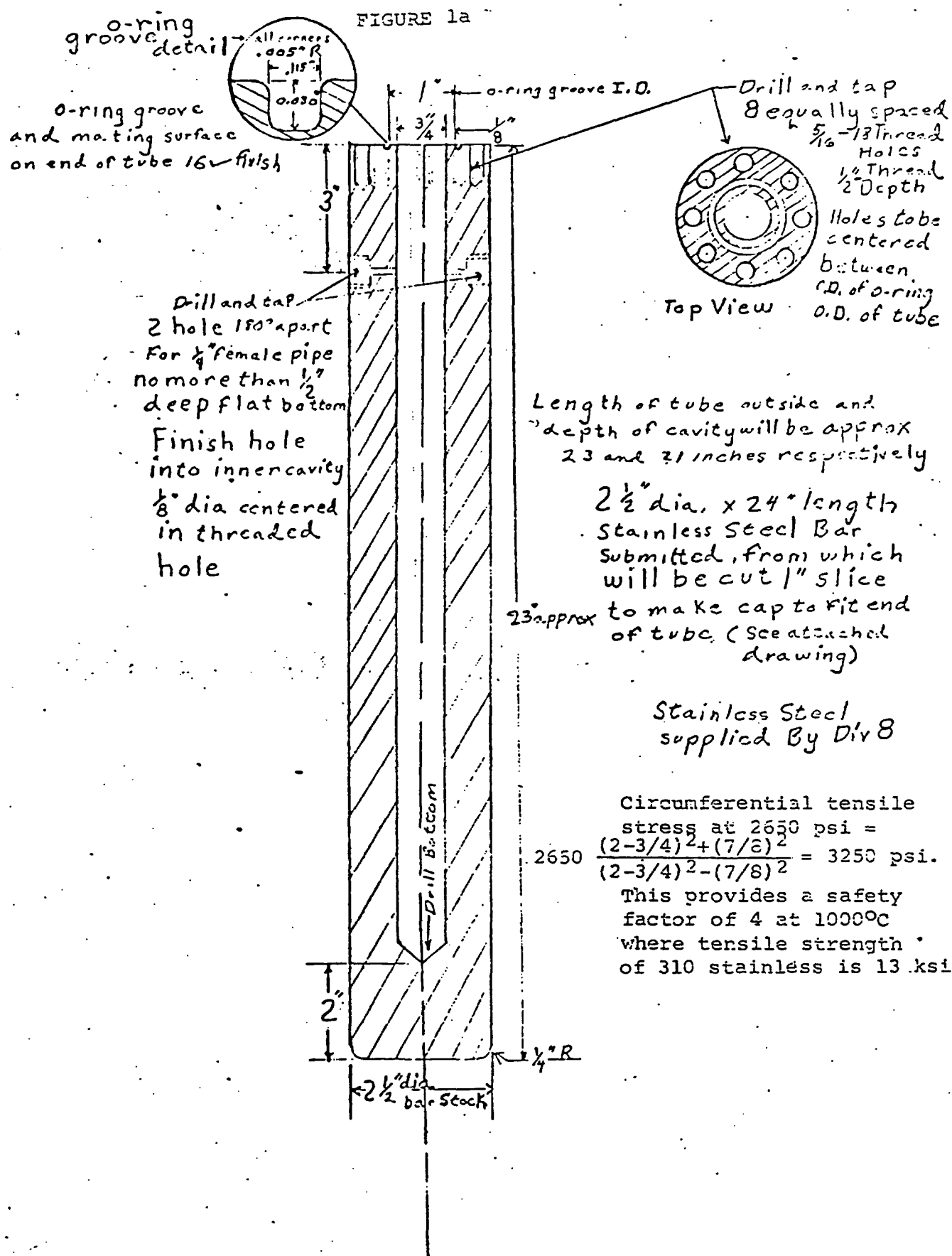
TABLE III

SUMMARY OF COMPOSITE TENSILE SPECIMENS MADE TO DATE IN PHYSICAL CHEMISTRY DIVISION

Liquid Infiltration Used Unless Otherwise Noted

Reinforcing Agent	Matrix	Volume Fraction Reinforcing Agent	Results Increase in Tensile, psi
TKF Sapphire Wool	H.P. Al	0.084	9,400
TKF Sapphire Wool	H.P. Al	0.091	9,200
TKF Sapphire Paper	H.P. Al	0.112	11,700
ESL R414 Safibers 4468G	H.P. Al	0.301	7,200
ESL R414 Safibers 4670G-2	2024	0.143	Loss
ESL R414 Safibers 4670G-1			
Gen. Tech. Sapphire Whisker Tape	2219	0.124	Loss
Carborundum SiC Whisker Tape	#13	0.103	4,100
Carborundum BN Paper	H.P. Al	0.233	5,400
FMC "Avibest" Microcrystalline Asbestos	2024 Prealloyed (1) Powder	0.10 Wt. Fraction	Loss
FMC "Avibest" Microcrystalline Asbestos		0.25 Wt. Fraction	Loss
Texaco Boron Filament	H.P. Al		Reaction with Al
Union Carbide Thornel 50 Graphite Yarn			

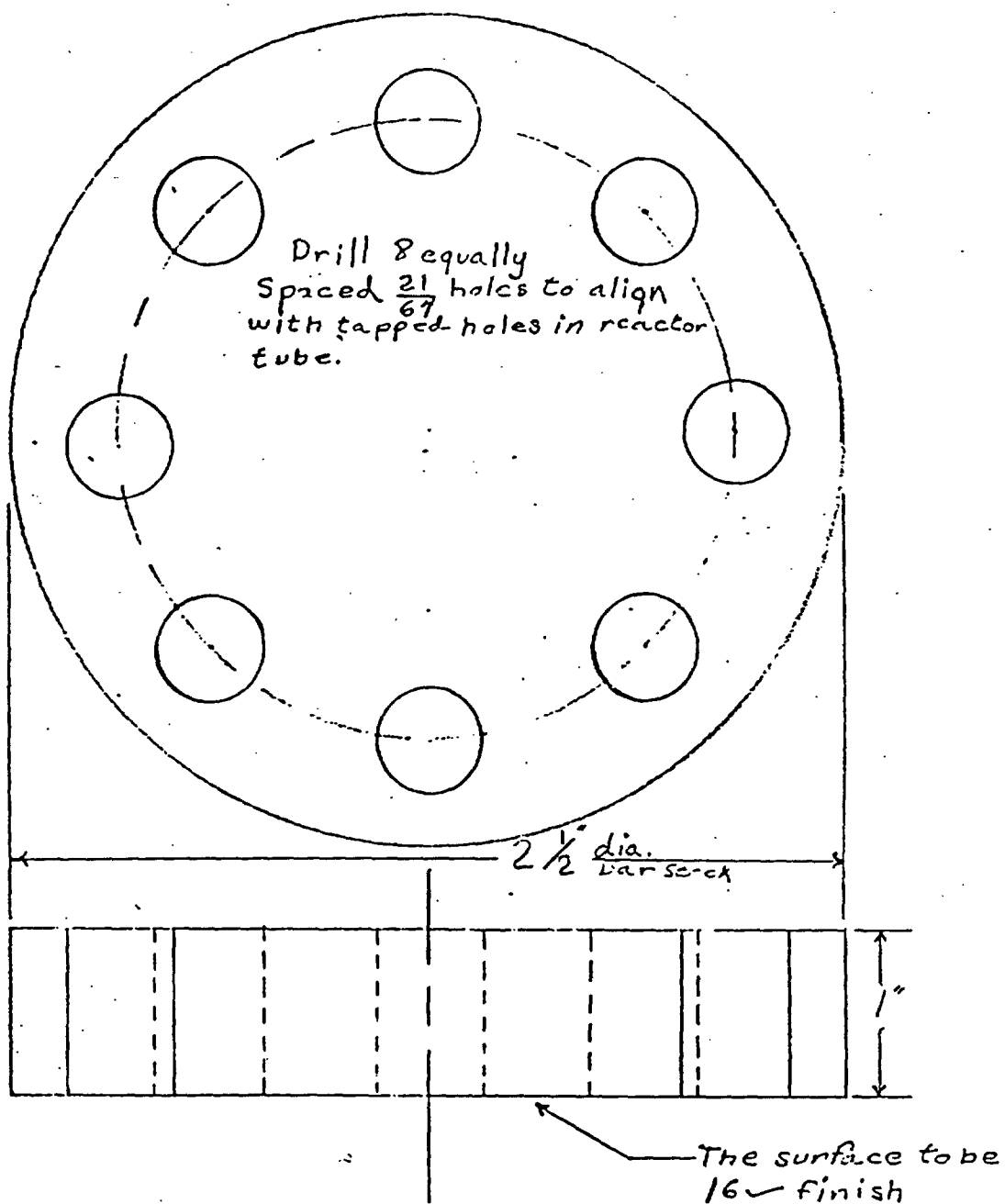
(1) Hot Pressed and Sintered



HIGH-PRESSURE AND TEMPERATURE BOMB FOR PREPARING REINFORCED
ALUMINUM TENSILE BARS BY LIQUID METAL INFILTRATION
(310 Stainless Steel)

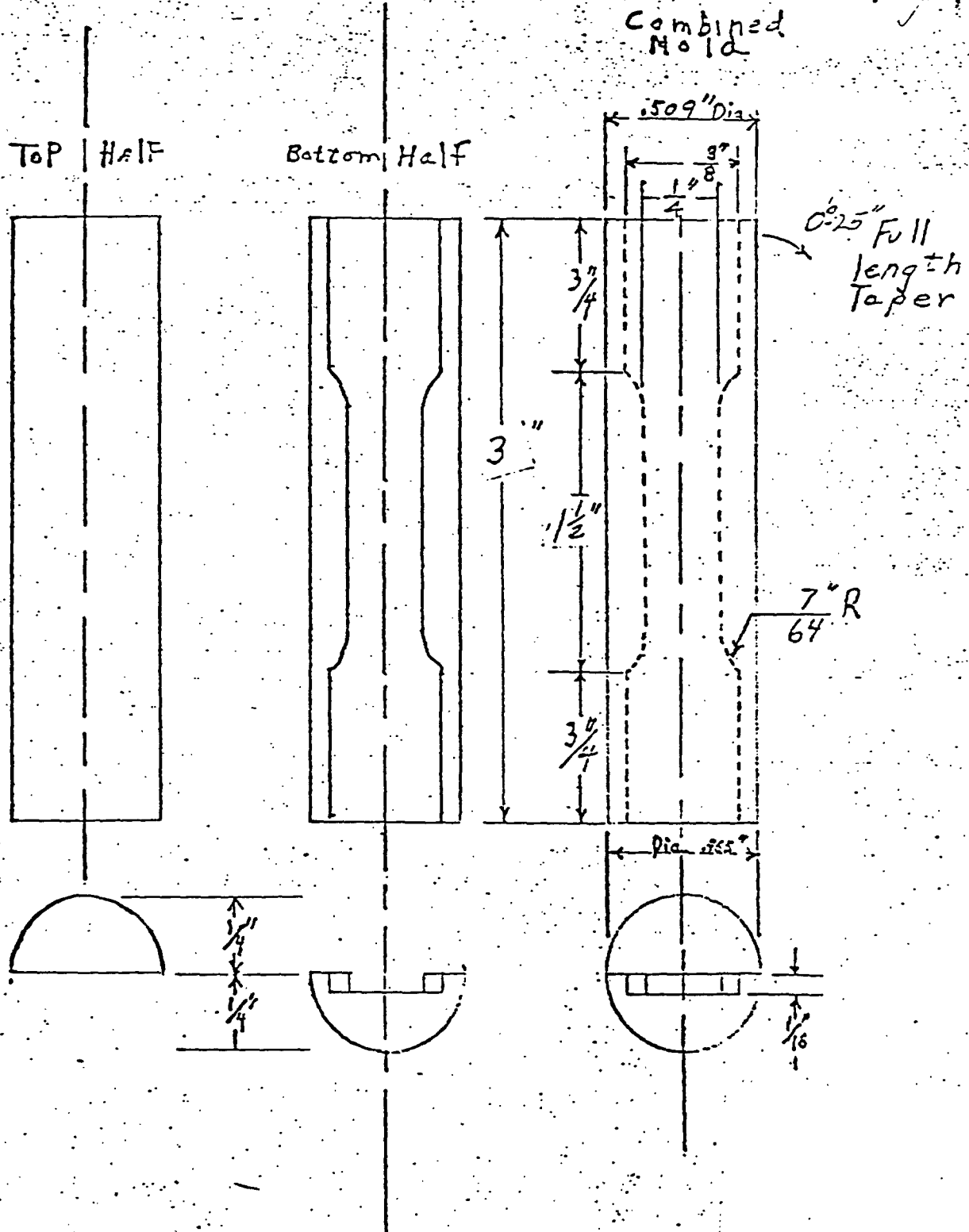
ATC 0035126

FIGURE 1b



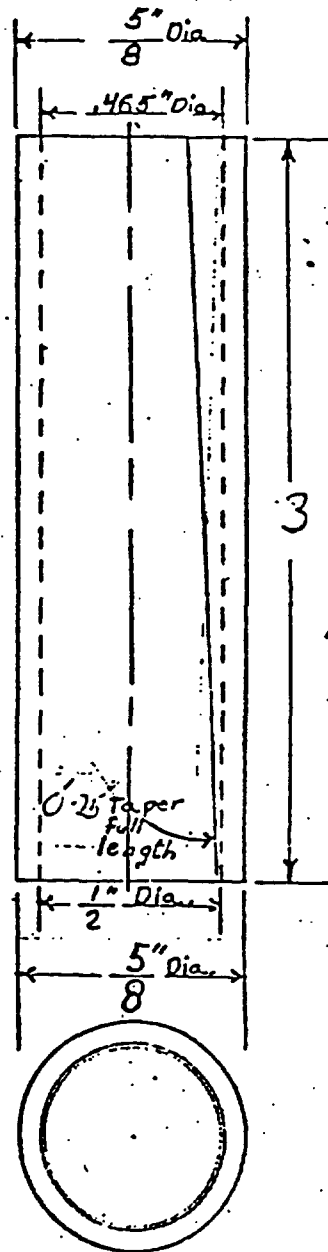
END CAP FOR BOMB SHOWN IN FIGURE 1a
(310 Stainless Steel)

FIGURE 2a



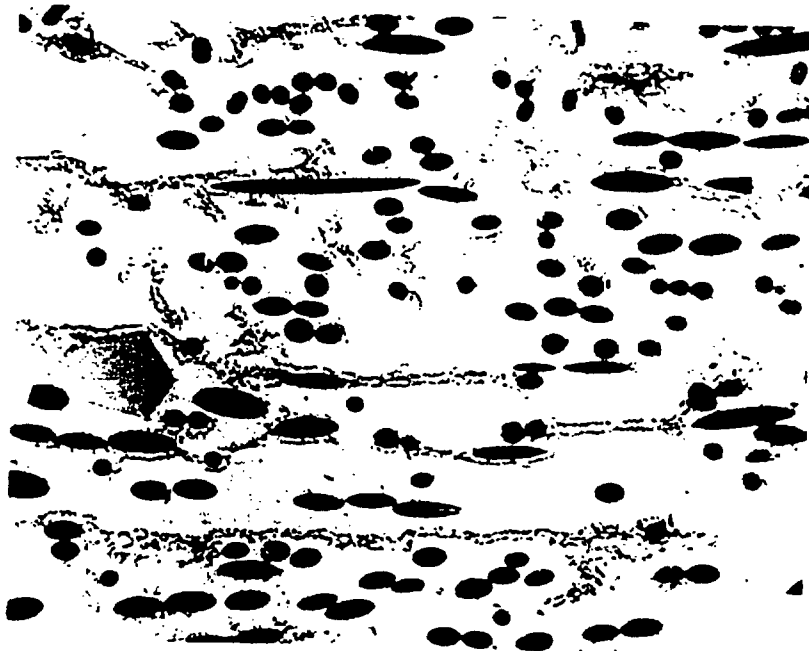
ELONGATED SPLIT BORON NITRIDE MOLD FOR
CASTING REINFORCED ALUMINUM TENSILE BARS

FIGURE 2b



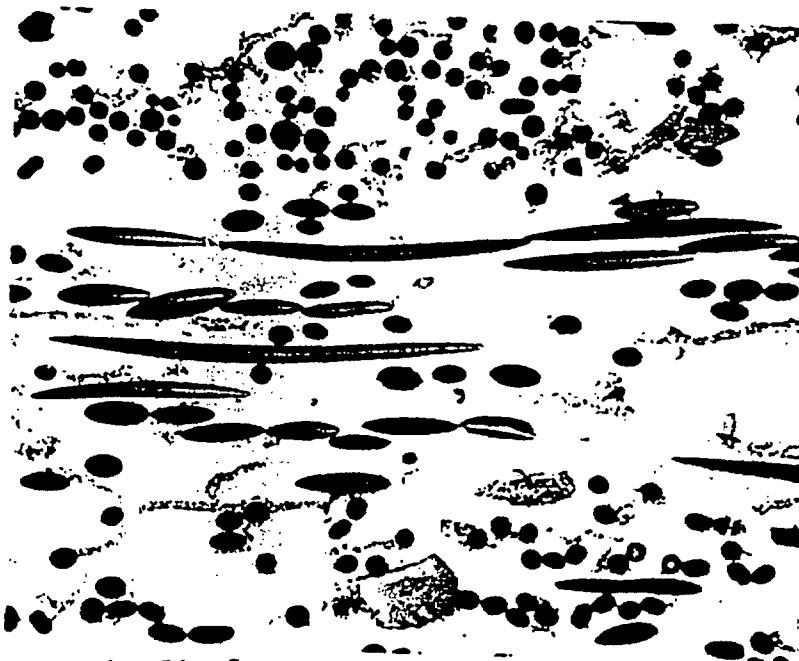
ELONGATED BORON NITRIDE SLEEVE FOR HOLDING
SPLIT MOLD OF FIGURE 2a TOGETHER IN CASTING
REINFORCED ALUMINUM TENSILE BARS.

FIGURE 3
(500X)



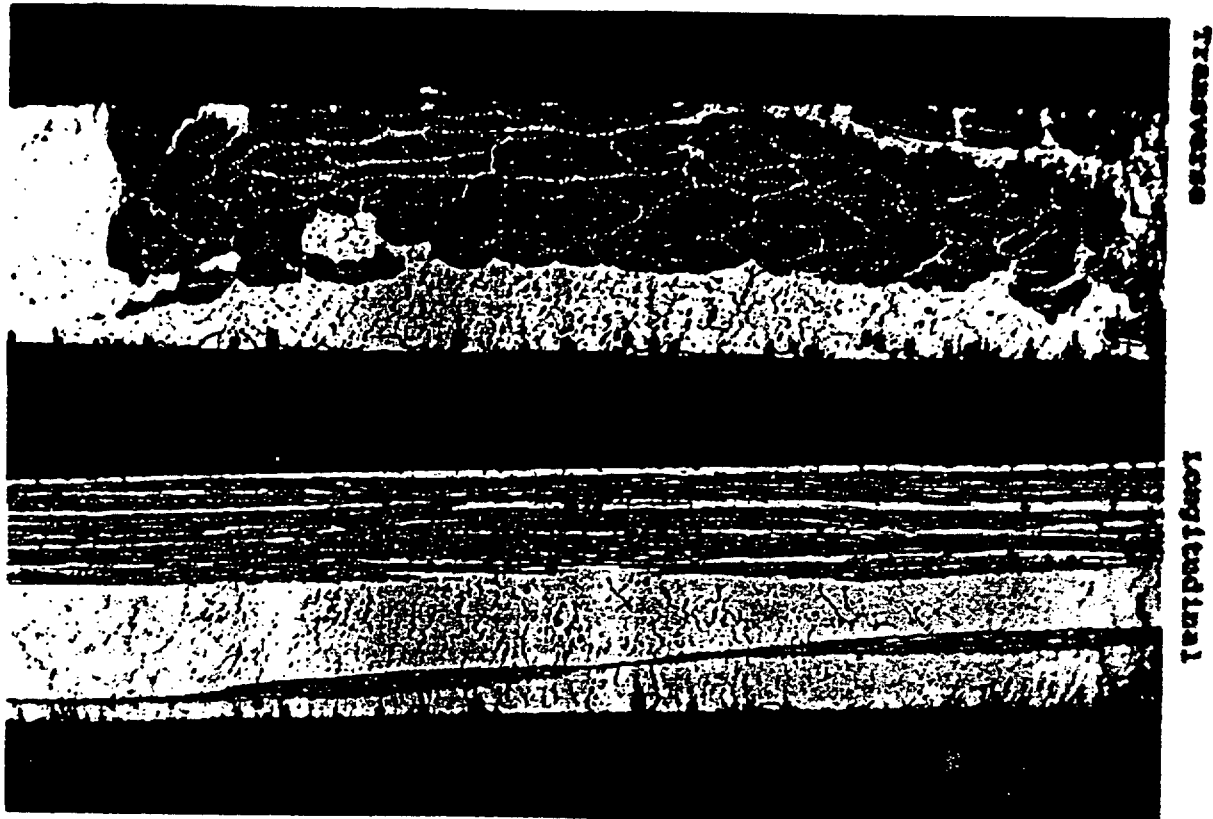
Transverse

SECTIONS OF HIGH PURITY ALUMINUM (SAMPLE
12339-31) CONTAINING 0.233 VOLUME FRACTION
OF CARBORUNDUM BORON NITRIDE PAPER



Longitudinal

FIGURE 4a
(20X)



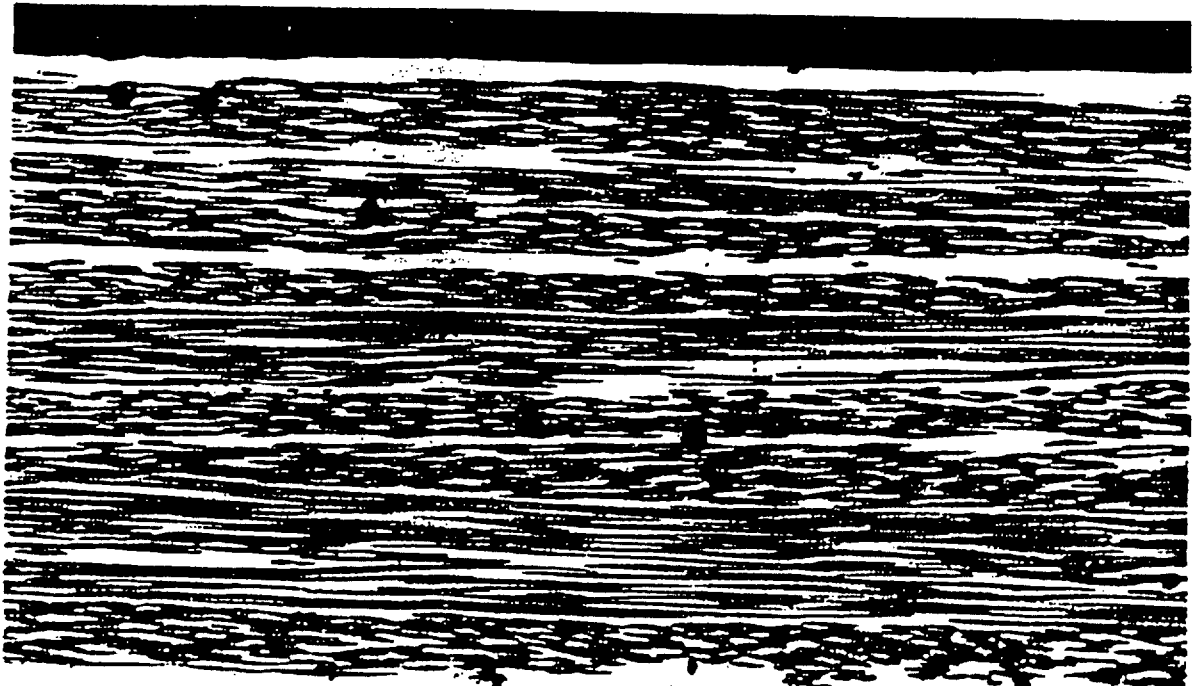
POLISHED SECTIONS OF 2025 ALLOY (APPROX.) CONTAINING 0.336
VOLUME FRACTION OF UNION CARBIDE THORNEL-50 GRAPHITE YARN

FIGURE 4:
(100x)



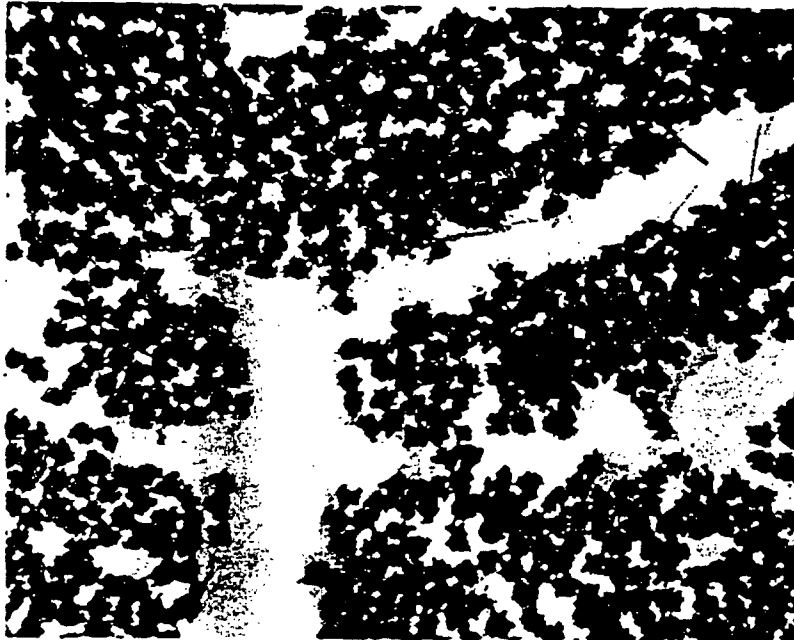
Transverse

POLISHED SECTIONS OF 2025 ALLOY (APPROX.) CONTAINING 0.336 VOLUME
FRACTION OF UNION CARBIDE THORNEL-50 GRAPHITE YARN



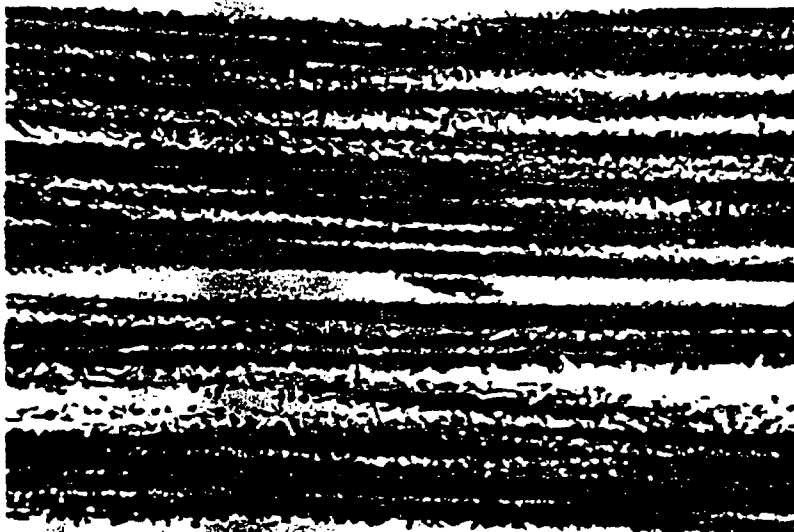
Longitudinal

FIGURE 4c
(500x)



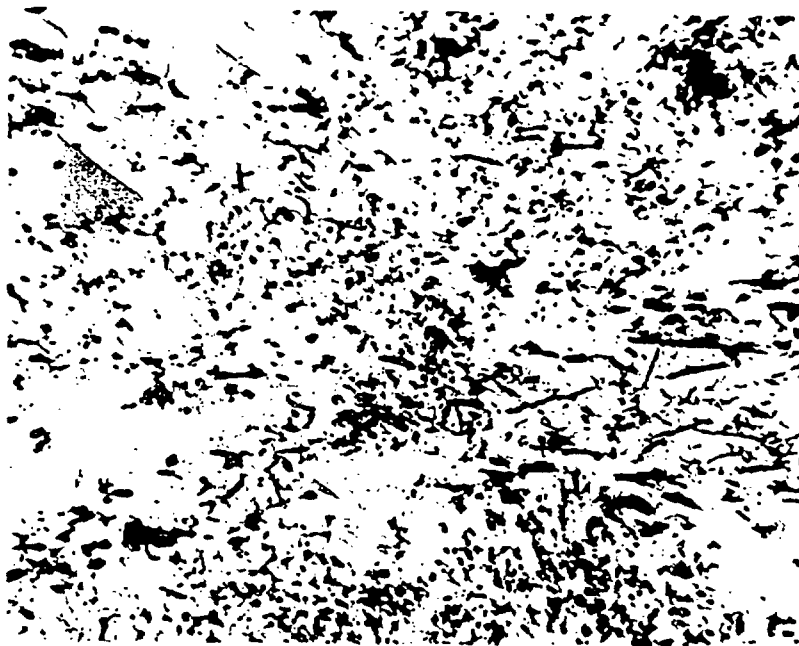
Transverse

POLISHED SECTIONS OF 2025 ALLOY (APPROX.)
CONTAINING 0.336 VOLUME FRACTION OF UNION
CARBIDE THORNEL-50 GRAPHITE YARN



Longitudinal

FIGURE 5
(500X)



Transverse

0.103 VOLUME FRACTION CARBORUNDUM SiC WHISKERS
IN #13 ALLOY, 0.5 TO 3 μ DIAMETER, 100 TO 750 μ
LONG, RANDOM TWO-DIMENSIONAL ARRAY, DERIVED
TENSILE STRENGTH OF WHISKERS - 126,000 PSI



Longitudinal

FROM: C. N. COCHRAN
PHYSICAL CHEMISTRY DIVISION
ALCOA RESEARCH LABORATORIES
NEW KENSINGTON

TO: DR. P. T. STROUP

ALCOA RESEARCH LABORATORIES
NEW KENSINGTON

March 29, 1968

RE: REINFORCEMENT OF ALUMINUM WITH WHISKERS AND FIBERS OF
BORON NITRIDE, ASBESTOS, ALUMINA AND GRAPHITE
Physical Chemistry Division Report No. 8-68-3

The enclosed copy of the captioned report covers the preparation and properties of composites of aluminum with whiskers or fibers of boron nitride, alumina, graphite, asbestos and silicon carbide. Evidence of some small degree of reinforcement was obtained only with the boron nitride. These results illustrate the problems of whisker or fiber damage and reaction with the matrix that can prevent reinforcement. Possibly, the reinforcing agents will have to be coated with other materials to circumvent these difficulties in future work.

C. N. Cochran
C. N. COCHRAN

CNC:dls

Enclosure

cc: W. A. Dean, ARL, N.K.
J. W. Newsome, ARL, N.K.
H. Y. Hunsicker, ARL, N.K.
L. K. Hudson, East St. Louis
K. J. Brondyke, ATC, N.K.
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