FIRING CORRELATION OF SOME PROPERTIES OF ENAMEL FRITS WITH PYROMETRIC CONES

A THESIS

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by

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FIRING CORRELATION OF SOLID PROPERTIES

OF ENAMEL FRITS WITH PYROMETRIC CONES

Approved:

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ACKNOWLEDGEMENTS

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I. DISCUSSION OF THE PROBLEM

The purpose of this study was to develop a satisfactory single test, employing standard pyrometric cones, to indicate enamel melting properties. The present standard method for the cone fusion test, as adopted by the American Ceramic Society, is reported in the Journal of the American Ceramic Society (1) and reprinted by Andrews (2a). This method incorporates a controlled rate of heating and thus requires about one hour to complete a test run. Such a timeconsuming method of testing has not been accepted readily by plants which use enamel frits. Thus, the beneficial results of a cone comparison test have been lost to shop operators. Inasmuch as the other operations incorporated in the test are simply and quickly performed, it was hoped that by shortening the time required in heating the cones, a plant practice of comparison of standard cones and cones made from enamel frits might be more feasible. None of the operations described in the following test required constant observation of the control operator for more than a few minutes.

For use in this research a series of brown ground coat enamels was developed. Included in the discussion, therefore, is a consideration of frit preparation, adherence, and related material. Variations in properties were desirable and guided

the choice of the enamel frit to be evaluated by the test.

It was thought best to develop an entirely new frit on which to run tests and determine the practicality of the high-speed cone comparison test. Variations were made in content of cobalt, nickel, manganese and antimony oxides in frit used in the experimentation. These oxides were known to give variation in color and adherence, and it was believed that they affected the firing range. A low firing frit was also tested. Thus the variation of adherence oxides and the temperature ranges of the two ground coats give an example of one important use of enamel frits on which to use the cone test.

Standardized cones are readily available, low in cost, and easy to use. The equipment used in this test is readily available in any enamel-shop control laboratory. Hence, at low cost and a short expenditure of time, the enamelist may add the valuable information gained from a cone comparison test to his evaluation of an experimental or sample frit.

II. REVIEW OF THE LITERATURE

The present Cone Fusion Test (1 and 2a) is one of a number of standard tests adopted by the American Ceramic Society. Montazuel (3) and The Properties and Uses of Pyrometric Cones (4) establish methods of support, mounting, placing and protection, as well as control of fire for use of cones.

The use of a constant temperature, instead of a controlled rate of heating over a long period of time, might lead to several difficulties. Freezing of cones as described by Rea and Shaw (5) might well result from introducing the cones into a high temperature without the customary slow rate of rise cycle. Bloating of cones as described in the Edward Orton Jr. Foundation manual (4) and by the British Clay Worker (6) might be caused by dampness in the cones or plaques or by carbonaceous material in the cones. The British Clay Worker article (6) indicates the necessity of pre-drying as well as calcining the cones. After drying, the cones and plaques must be transferred quickly to the already hot furnace to eliminate any possibility of moisture absorption. Nakamota and Sakata (7) experimented with various angles of mounting and their experiments led the author to believe that shorter cones of the Pyrometric Cone Equivalent (PCE) type might be more readily handled in a test of this type.

The frits used in this test were of the single fire brown ground coat type. Variations were made in four of the adhering metallic oxides, namely: cobalt, nickel, manganese, and antimony.

Morren (8) stated that more than 3.35% fluorine produced a discoloration on brown color oxides containing iron. Firing temperature must be carefully controlled as colors are sensitive to temperature variations, more so than enamels over ground coat. The hot iron tends to reduce and thus destroy the color of some color-producing oxides, due to the intimate contact of glass containing color oxides and the hot metal during firing. High amounts of cobalt tend to change the color from brown to blue.

Iron oxide and manganese oxide mixtures are much more stable when the manganese content is low according to Bernard (9). A mixture containing five percent manganese oxide kept for several hours at 800° centigrade did not show any decomposition. With increase of the manganese there was noted the irreversible formation of more and more magnetite at any reheating temperature.

King (10) found that manganese oxide alone was not effective in precipitating metal at the ground coat-iron interface. He stated that the indirect influence of manganese oxide in combination with cobalt and nickel oxides is not significant. Kautz (11) also found manganese without significant contribution to adherence when used alone. Howe and Fellows (12), however, found that nickel and manganese increase the firing range. Aldinger (13) found cobalt better than nickel for both adherence and long range. Cobalt also helped to prevent over-firing. English investigators (14) have concluded that the nickel dip as used in most commercial pickling practice does not improve adherence where

this is already good, although it improves the latitude in fusing conditions in those cases studied. Dietzel (15) stated the theory that adherence results from precipitation of cobalt metal according to the equations:

$$CoO + Fe = Co + FeO$$

$$CoO + 2\ FeO = Co + Fe_2O_3$$

Dietzel also believed, as did Clanson (16), that adherence is a corrosion phenomenon of cobalt reacting with the sheet steel, penetrating the pores of the steel, and etching the surface to secure good contact. Thus they believed adherence to be a function of the contact line. Kautz (11) believed adherence a function of the contact line by formation of iron oxide layer and penetration of aerial oxygen through enamel layer. Rueckel and King (17) and King (18) believed adherence not to be a function of the contact line, but rather due to dendrite formation. If this is true, nickel oxide must have a pronounced action on adherence because King (19) found nickel to have more effect on metal precipitation than cobalt and the action is evident even when the ratio of cobalt oxide to nickel oxide is as high as three to one. Andrews (20) concluded: "No theory has been accepted generally as explaining the function of cobalt in sheet iron ground coats ____. It is quite probable that the true explanation will involve several of these theories."

III. PROCEDURE

A. Preparation of Slip

1. Frit Preparation

The frit batch was weighed, sieved through a forty-mesh screen, mixed well, re-sieved, and loaded into the frit pot at the starting temperature (See Graph 1). The batches were 1200 grams since the frit pot could be conveniently loaded with a batch of this size. The firing curve, as shown by Graph 1, was followed as closely as possible, and, after firing to a clear thread, heating was continued for thirty minutes. The molten frit batch was then quenched in water, the water poured off, and the resultant batch dried for 24 hours. Before weighing, the batch was sieved through a twenty-mesh screen to remove all large pieces, so-called "rocks". These large particles were crushed until the entire batch was twenty-mesh or smaller in particle size.

2. Milling of Slip

Batch samples were weighed according to the schedule shown on Data Sheet 1. This batch with 30 percent water was milled to a fineness of 6 or 7 percent. The fineness was checked by the standard method of the Porcelain Enamel Institute for fineness of wet-milled enamels adopted February 17, 1930. A two-hundred mesh screen was used for all testing. After milling,

the sample enamel batch was held in storage for twenty-four hours before spraying, and the water content was adjusted to 45 percent. The set of the enamel was adjusted by small substances of tetrasodium pyrephosphate, to make the slip more fluid, and sodium nitrite, to make the slip less fluid. These substances were included in the final water addition to prevent clotting of the slip.

B. Preparation of Metal Plates

1. Pickling of Metal Plates

All sample plates were three inch by five inch, twenty-gauge enameling iron and were subjected to the following pickle procedure:

a. Immersion in boiling commercial cleaning-compound solution for twenty minutes or until no draining breaks showed on inspection after removal from bath.

b. Warm-water rinse at about 70° F.

c. Immersion into 5% sulfuric acid solution at 150° F. for ten minutes.

d. Cold-water rinse at about 40° F.

e. Immersion into a 2% double nickel salt solution, at 150° F. and a ph of 2.6, for five minutes.

f. Cold-water rinse at about 40° F.

g. Immersion in a cyanide pickle bath at 185° F. for two minutes.

h. Drying in air to remove all traces of moisture.

The sample plates were thus prepared, and carefully examined for any sign of rust, grease, or finger marks. Any of these imperfections of the pickle process as well as any scratches or dings caused rejection.

2. Spraying

Metal plates (prepared as in the previous paragraph) were then sprayed to a thickness of thirty grams per square foot. The plates were sprayed on both sides to help prevent warping and thoroughly dried before firing.

3. Firing of Plates

The plates, after spraying was complete, were fired according to the schedule shown in Data Sheet 1. They were fired in an electric muffle furnace in an oxidizing atmosphere. The firing period as given in the data sheets is that at which all variations of that particular frit gave the best results. Periods tried in determination of the best time were from one and one-half to seven minutes total time in the furnace, measured from the closing of the door after loading to the opening of the door for removal. The temperature was established by a similar trial-and-error method in 20° F. intervals and those temperatures shown are the limits of good plates with all frits. A plate was considered satisfactory if it exhibited a surface free from any bubbles, an even color, and a good gloss.

C. Impact Adherence Test

The test herein used is sometimes known as the falling weight test. A rod with an hemispherical end one inch in diameter is allowed to drop vertically upon the rigidly fixed enameled plate. The rod was used in this case in order to concentrate more weight in the one-inch tube than would be available if the weight were a one-inch ball. All pieces were subjected to this weight dropped from a height of twenty-five inches. This height was determined as the minimum necessary to cause deformation of the plate into the one and one-eighth inch hole drilled into the base directly below the tube holding the weight. The degree of adherence was noted by visual observation and recorded in one of the five grades from none to excellent. Variation was indicated by the approximate area of the plate covered by enamel slivers twelve hours after the test. The delay was necessary because in some cases the enamel continued to flake off for sometime. In some cases, where a large amount of flaking occurred over an extended period, the adherence is noted by two letters.

The best adherence obtainable from commercial samples determined the grade called "excellent". From this grade, the other degrees of adherence are arbitrarily assigned and "no adherence" is complete removal of the enamel during the test, leaving the clean, bare metal.

D. Acid Resistance

Acid resistance was evaluated in the manner recommended by the Porcelain Enamel Institute, Test for Acid Resistance of Porcelain Enamels (20). The test procedure consists of application to the test plate of a few drops of a ten percent solution of citric acid in water; covering with a watch glass for fifteen minutes; washing the plate with water; noting the effect of the acid solution on the plate. If a pencil mark can be removed with a dry cloth and there is no visible effect, the acid resistance is considered "AA". If a wet cloth is required to remove the pencil mark, the acid resistance of the plate is classed as "A". When the acid has a visible effect on the enamel the plate is classed as "B" or "C" depending on blurring of highlights. Very severe effect and blurring of highlight to disappearance is class "D".

E. Reflectance and Gloss

Reflectance is the measure of light-reflecting efficiency of surfaces. It is commonly expressed in percent reflection, a value arbitrarily dependent on reflection from a clean magnesium-oxide surface being considered one hundred percent. Gloss is a part of surface appearance and is here measured similarly to reflectance at an angle of 45°, and the reflectance at this angle is called specular gloss.

The apparatus for measurement consists of a light source and two photo-electric cells in series with a galvanometer. This

equipment is described by Hunter (21). When the light to each of the cells is equal, the galvanometer is in balance. Thus, by allowing the light to reflect from a calibrated standard and a sample plate, two plates may be compared and evaluated. The standard applicable to the evaluation to be determined is first used to calibrate and samples are then inserted and readings made.

F. Color and Surface

The color was noted by visual observation and thus values given are only approximate within the range from cobalt blue to nickel brown. The surface was determined by visual observation and comparison with the best obtainable commercial ground coat enamel. It was considered good if bubble-free, without matte, and free of copper heading, fishscale (determined after several weeks), crazing, egg shell or orange peel. Thus, if up to commercial practice, it was considered good.

G. Fusion Block Test

For the fusion block test a porcelain shape such as that suggested by Andrews (2c) is used. A portion of the milled enamel is dried and this cake ground in a mortar. The resulting powder is moistened and packed into the space above the incline to level full. Any excess is cleaned off and the sample and block allowed to dry in an oven for twelve or more hours. The block is then placed in a furnace and heated at a rate of 3°F. per

minute (See Graph 2 for heating schedule). The temperature is read as the enamel starts to flow and as the flow passes each intersection.

Water is the only binder used with the enamel powder to pack into the space in the block. Several gum binders were tried, but were found to cause bloating and inconsistent results.

H. Standard Cone Fusion Test

In the standard cone fusion test for enamel frits, as described by Andrews (2a), a representative sample of frit is ground to pass a 150-mesh sieve. This powdered frit is made into cones 5/8 inch along the base and 2 1/2 inches high. The test cones are dried, and mounted, using a plaque of asbestos or steel coated with enamel, at an angle of 82° between the troweled face of the cone and the horizontal plaque. A thermocouple is placed in the center of the plaque equidistant from the four test cones and the whole assembly dried. The furnace used and placement of the plaque in the furnace are such as to give good control of the rate of heating. The control thermocouple is equidistant from the furnace walls, and 1/2 inch above the tips of the test cones. The test starts at 800° F. or below, and the rate of heating is arranged according to a set schedule from 800° F. to 1370° F. at a decreasing rate of temperature rise from 20° F. per minute to 10° F. per minute.

J. Modified Cone Comparison Test

The following modifications were made in the procedure of the standard cone fusion test to shorten, simplify and adapt it for easy plant use:

1. The test was made by comparison with standard pyrometric cones.

2. The test material used was milled enamel, prepared for firing on test plates of sheet steel. This material was dried and ground in a mortar. The material contained clay as a mill addition.

3. Test cones were made of PCE size to facilitate easy handling and standard cones used were of the PCE size.

4. Standard cones were prepared for the test by calcining to remove carbonaceous material, which is apt to cause bloating of the cones and hence inconsistent results.

5. The mounting of the cones was on a plaque made from clay to facilitate handling. The mounted standard and test cones along with the plaque were dried overnight to remove all traces of mositure.

6. Water was used to make up the test cones from the milled enamel batch. The resultant cones handled well due to the clay in the enamel slip batch. The clay used in the slip preparation must be reasonably free of carbonaceous material.

7. The dried cone plaque is put into the furnace at the desired temperature, usually the same as that used in fir-

ing test plates or a slightly lowered temperature. The time of bending from start of bending of cone tip until the tip is on a horizontal line with the upper surface of the plaque is recorded.

8. At any given furnace temperature the time of bending of the test cone is compared with that of standard pyrometric cones, which standard cones bracket the bending time of the test cone. Thus a reproduceable comparison test of a cone, made from milled enamel batch, can be made with a standard pyrometric cone.

Cones for this test were fired in a Globar furnace, which is mounted in a horizontal position. The full length alumina muffle is three inches inside diameter, with a heating zone seven inches long and uniform temperature over the heating zone. Full load power consumption is about 3,300 watts. The furnace is controlled by a Leeds and Northrup Micromax recording potentiometer and an on-off relay controlled magnetic switch. A scale-drawing of this furnace is shown in Figure 1, and a diagram of the electric wiring is shown in Figure 2.

IV. DISCUSSION OF RESULTS

In standardizing the test procedure, a comparison was made between standard and calcined cones at the 150° per hour rate of temperature rise. The following observations were made:

1. Less differences in end point resulted than might be expected from variations in cone formation.

2. Standard cone 022 comesdown at 1090° F.

3. The 022 cone calcined at 900° F. bloated slightly, but the cone calcined at 800° F. did not bloat.

4. The order in which standard and calcined cones came down is shown in Data Sheet 5.

From the above results it was decided to calcine at 850° F. for 2 1/2 hours to avoid slight bloating of 900° F. calcine. To have the advantage of less brittleness, which the 900° F. cones exhibited, it was decided to calcine at higher temperature than 800° F. for a longer time than the original two hours. The cones calcined at 850° F. for 2 1/2 hours showed variations limited to the range of the experimental error when checked against standard cones. Hereafter, "calcined cones" refers to this type.

The angle of mounting of the cones was found to be more satisfactory at 60° than at the standard 82°. With the more nearly vertical angle, the sample cones slumped when fired at the enamel maturing temperatures. With the 60° mounting

position the test could be run, in those cases examined, at the enamel maturing temperature. Too high a temperature caused slumping and too low a temperature took excessive time for the test to be run. One to two minutes seems to be the optimum time of test from start of bending to down, and 60° mounting angle gives this time in the results shown on Data Sheet 3.

On Data Sheet 3 are shown the results of the cone comparison test and on Graph 3 is shown the total elapsed time of test. In general, the total time of test increases with addition of metallic oxide, except in the case of cobalt oxide, which cone series is somewhat erratic in trend. Starting time of bending is approximately the same in each case and total time of test-cone bending increases. The total time for manganese oxide is somewhat longer than for the other metallic oxides, and starting time somewhat lower. The longer firing range of enamels containing manganese oxide is substantiated by the experimental work of Howe and Fellows (22).

On Data Sheet 4 are shown the results of the fusion block test. These results are shown in graphic form on Graphs 4 and 5. The results are much more erratic and do not show the trends nearly so well as the modified cone test. The two commercial frits have short total times of bending and low starting time as seen on Data Sheet 6.

Runs of the fusion block test show a greater variation than the modified cone test. The manganese and nickel series

(Graphs 4 and 5) again show the greater firing range as indicated by the greater temperature range from start of flow to finish of test. Addition of two percent of antimony oxide decreases total time, but the total time remains the same with the addition of three percent. Samples containing the cobalt oxide are erratic in the fusion block series (Graph 5) as in the modified cone series.

On Data Sheet 2 are shown the observed results of the adherence test on fired plates as well as the gloss measurements; surface observation; color and acid resistance. From this Data Sheet 2 it may be seen that cobalt oxide is necessary for adherence when a single metallic oxide addition is made within the percentage range examined. Antimony oxide seems to decrease the adherence when added in amounts of more than one percent. With the addition of two percent of manganese oxide the range of the enamel is extended enough to be fired satisfactorily at 1550° F. in three and one quarter minutes.

The gloss also is improved by the addition of manganese oxide, gloss being higher than any of the other oxides with plates P 1, P 2, P 3. Long cone bending time of cobalt oxide at one, two, or three percent and high gloss is correlated by P 8, P 9, P 10 and likewise nickel oxide at three percent in P 13. The exception is antimony oxide at three percent in P 6, which has the lowest gloss value determined.

Acid resistance in all cases was class B. Color is

noted in Data Sheet 2 in the column so headed.

When the cone comparison test was run with a lower maturing temperature enamel it was found necessary to lower the temperature at which the test run was made. Thus a commercial frit (See Data Sheet 6) which is normally fired at 1470° F., when made into sample cones, will give good test results at 1470° F. enamel cones at 1550° F., as with the higher temperature enamel used in the majority of tests herein, the 1470° F. cones slump rather than bend and no true reading can be taken. Likewise an enamel fired at still lower temperatures was found to test satisfactorily at the firing temperature of the enamel and slump at high test temperatures.

On Data Sheet 6 are given two frits, made by chemical additions to Basic Frit (Data Sheet 1), which were made up in an effort to check unsatisfactory characteristics. These two frits when fired on metal sample plates crawled in the case of G-1 and blistered in the case of M-2. When tested by the cone comparison test, cone G-1 froze and slumped. Enamel M-2 bloated and slumped.

The method used in evaluation of the end point of calcined cones is that used in practice and recommended by the Edward Orton Jr. Foundation (4). The clock method, although satisfactory for cone users heretofore, is shown on Data Sheet 3 to give comparable results to stop watch timing of bending period. However, the differentiation is not so sharp. Thus 1% antimony would appear the same as 2% antimony by the clock

method, but is shown to be different by the total time of bending. Measurement would increase the definition of end results of calcined cones, but it would be necessary to remove the cone plaque immediately upon completion of the test. It is thought that the comparative results of a close observation of end point and evaluation by the clock method are sufficient.

V. CONCLUSIONS

1. Reproducible results on frit properties may be obtained from a cone comparison test with less variation than with fusion block tests.

2. Trends of variation of firing range in enamel frits with metallic oxide additions are clearly shown with the cone comparison test.

3. In enamels studied, good gloss correlates with long, smooth bending of cones.

4. Adherence does not seem to be correlated with any cone test property and must be determined separately.

5. Recording the time of bending as well as the end position of the standard cones is not absolutely necessary in test runs. Standard cones can not be read to the precise accuracy of a stop watch, but they clearly show variations of heat input to cones. Neither temperature-indicating thermocouples nor time of runs can show the effect of active heat on ceramic bodies as well as cones.

6. Test runs may be made with fair accuracy without any indicating device other than standard cones.

7. Test cones made with frits having unsatisfactory properties exhibit misbehavior during test in those cases studied.

8. The angle of mounting of cones is decreased from 85° to 60° in order to enable the cone comparison test to be run at

the same temperature as the test plates are run. Any greater angle tends to cause slumping of the test cone.

9. The maturing temperature may be correlated with test frit cones action during test.

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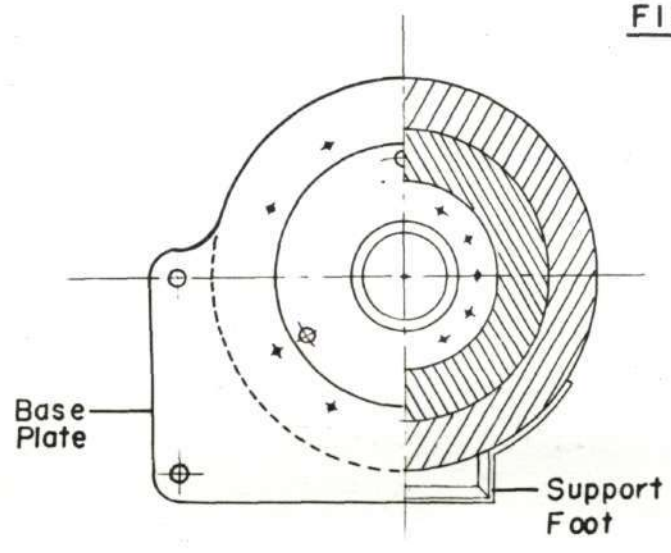
APPENDIX I.

PAGE

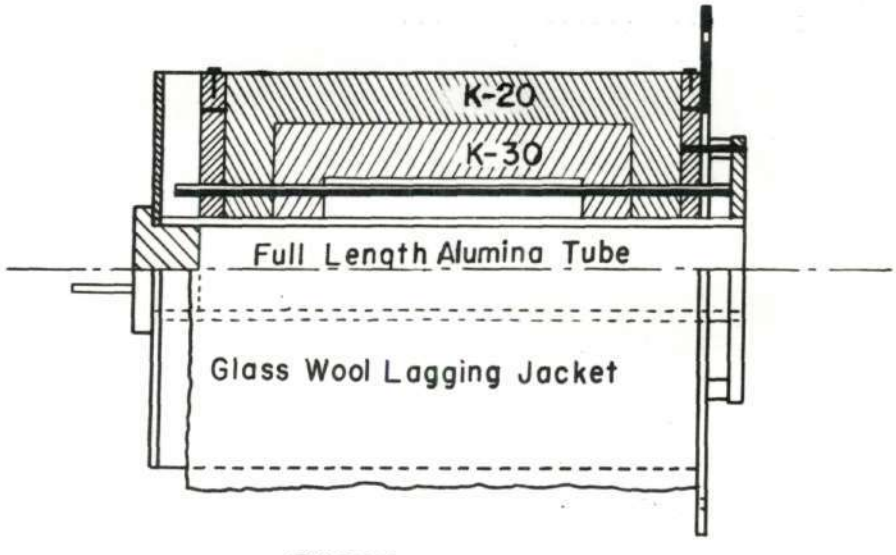
DETAIL OF GLOBAR FURNACE CONSTRUCTION HORIZONTAL & VERTICAL SECTIONS

Scale : 1"= 6"

FIG. 1

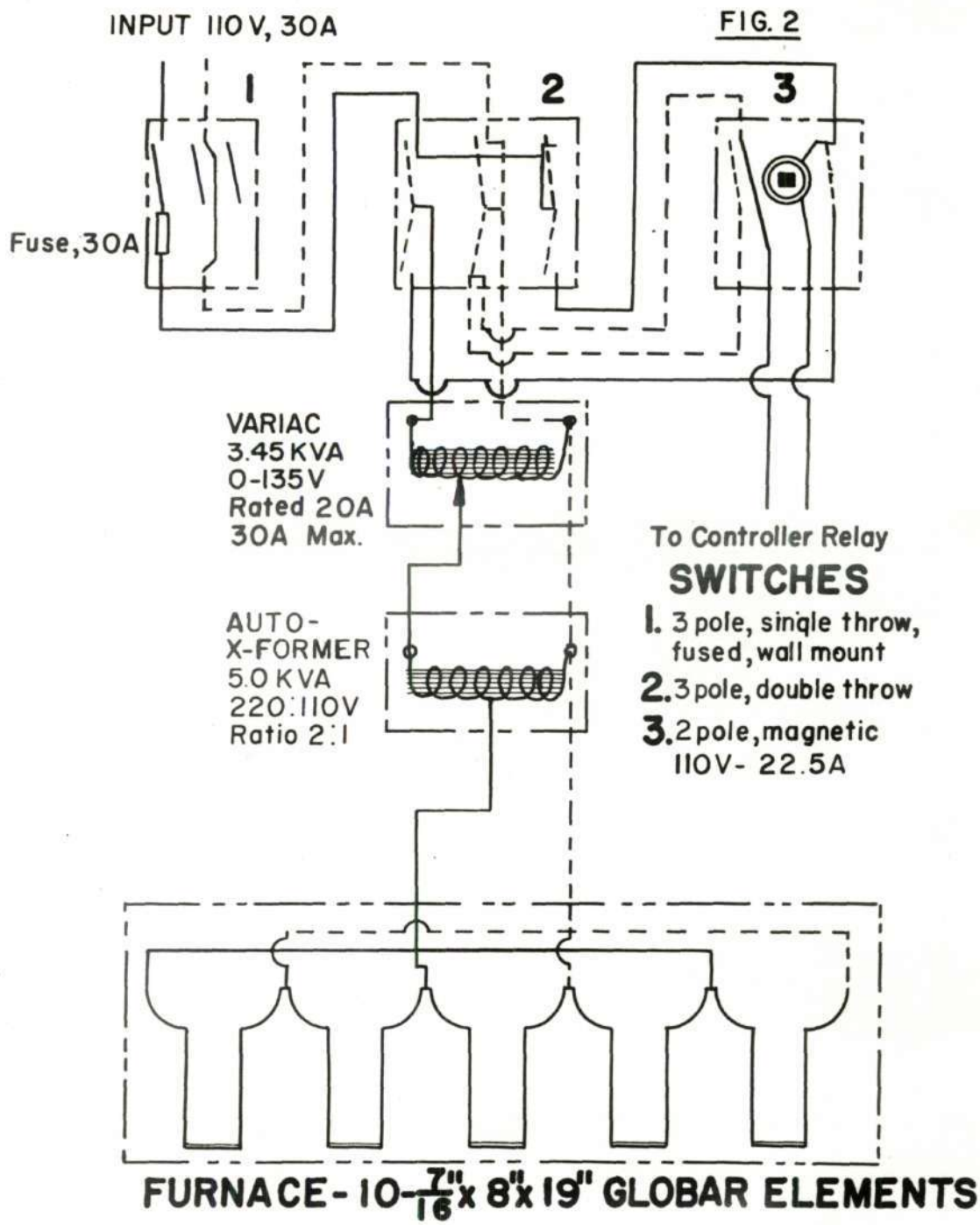


FRONT



SIDE

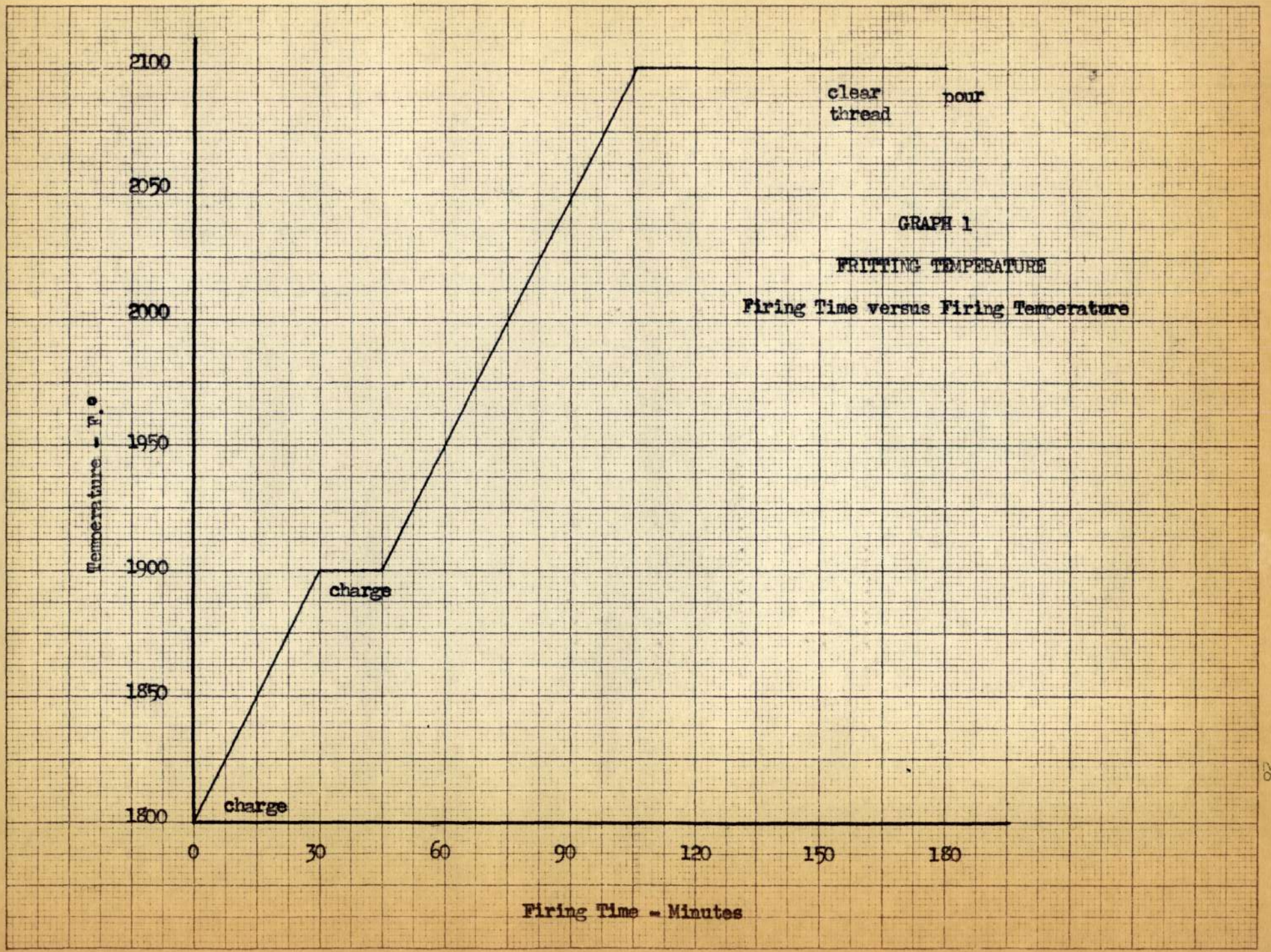
WIRING DIAGRAM- POWER SUPPLY



FULL LOAD: approx. 60 amps at 62.5 volts

APPENDIX II.

PAGE



GRAPH 1

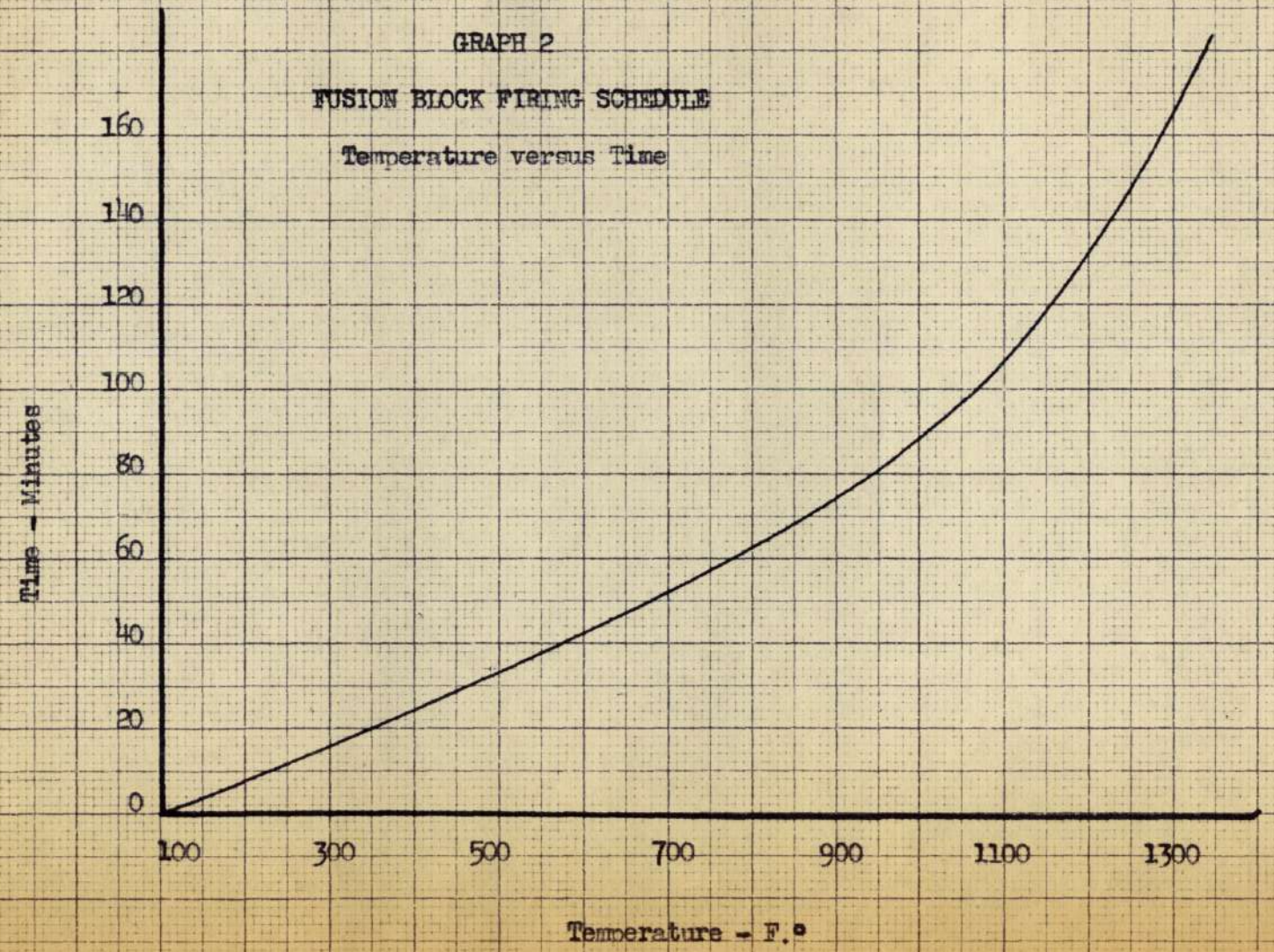
FRITTING TEMPERATURE

Firing Time versus Firing Temperature

GRAPH 2

FUSION BLOCK FIRING SCHEDULE

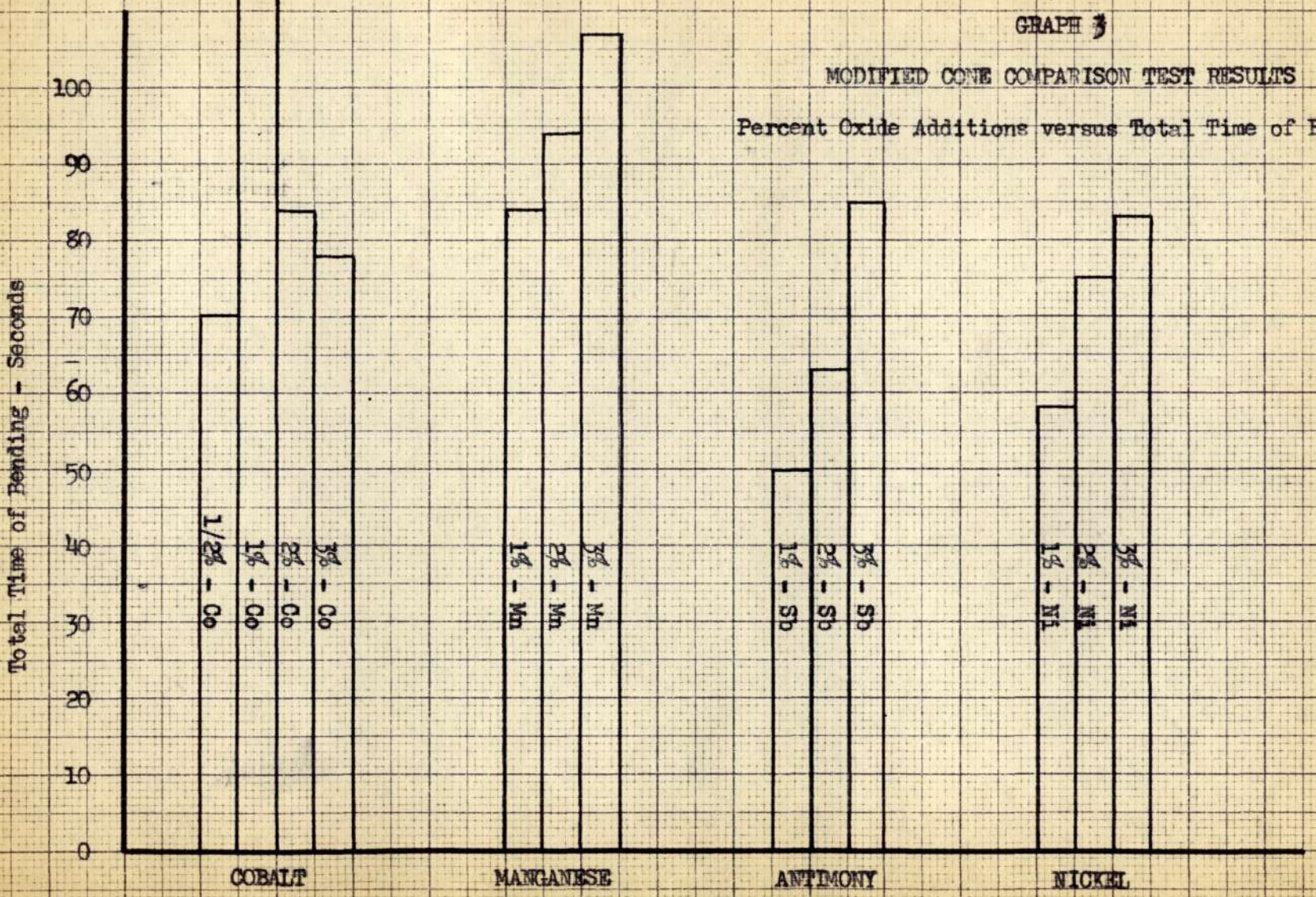
Temperature versus Time



GRAPH 3

MODIFIED CONE COMPARISON TEST RESULTS

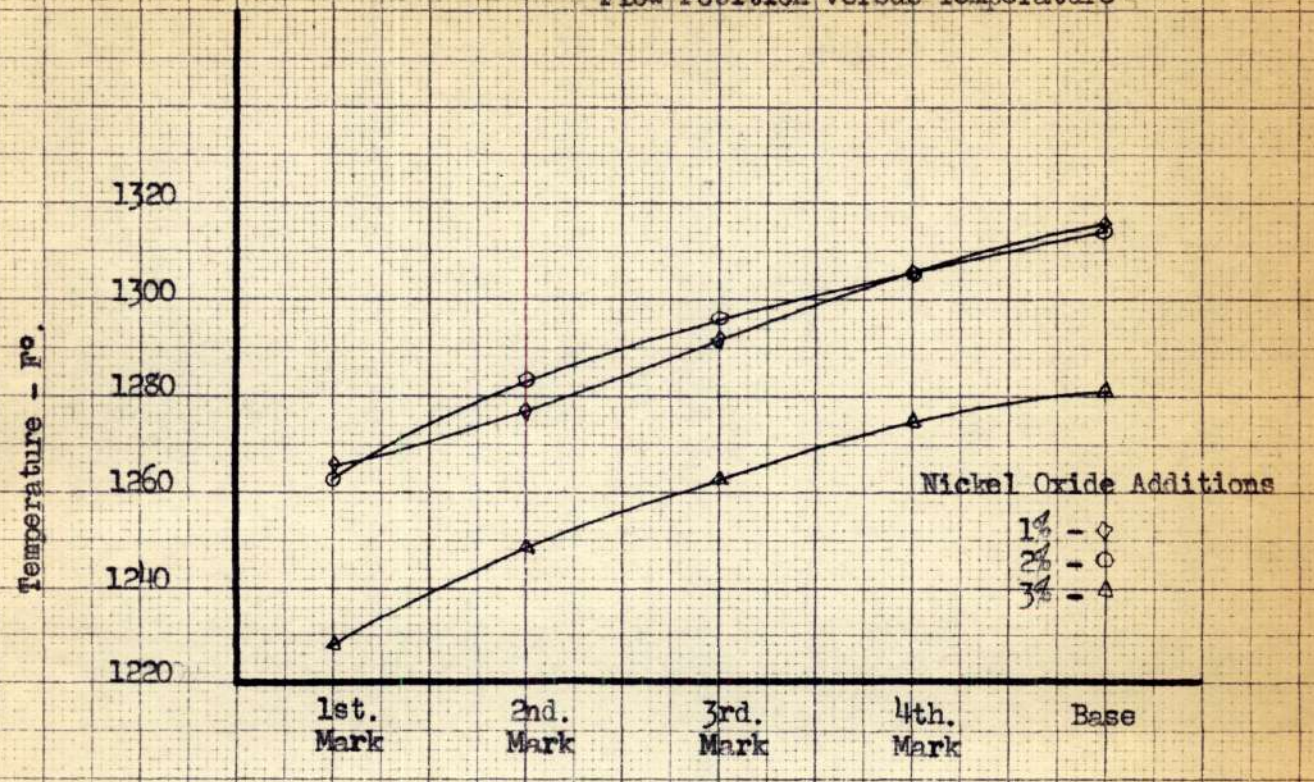
Percent Oxide Additions versus Total Time of Bending

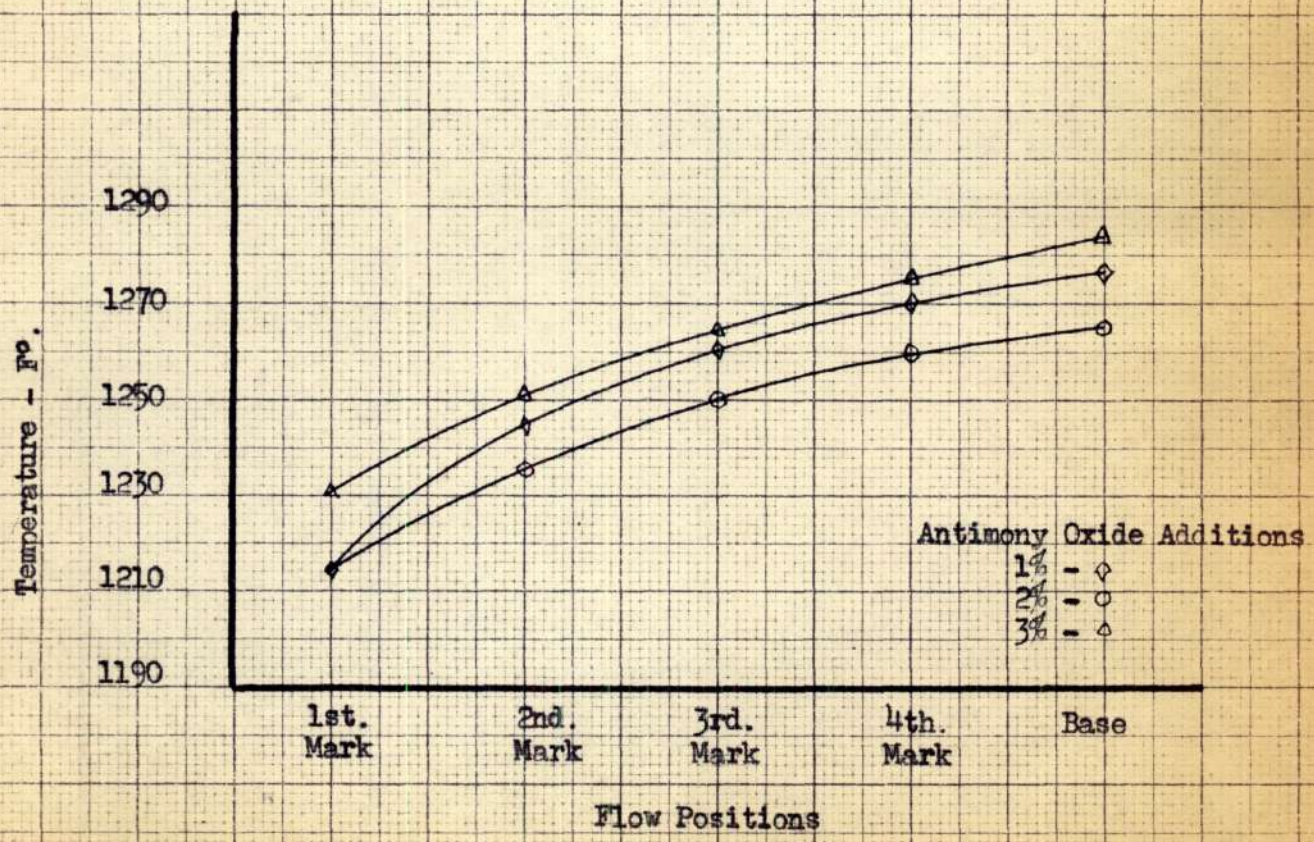


GRAPH 4

FUSION BLOCK TEST RESULTS.

Flow Position versus Temperature

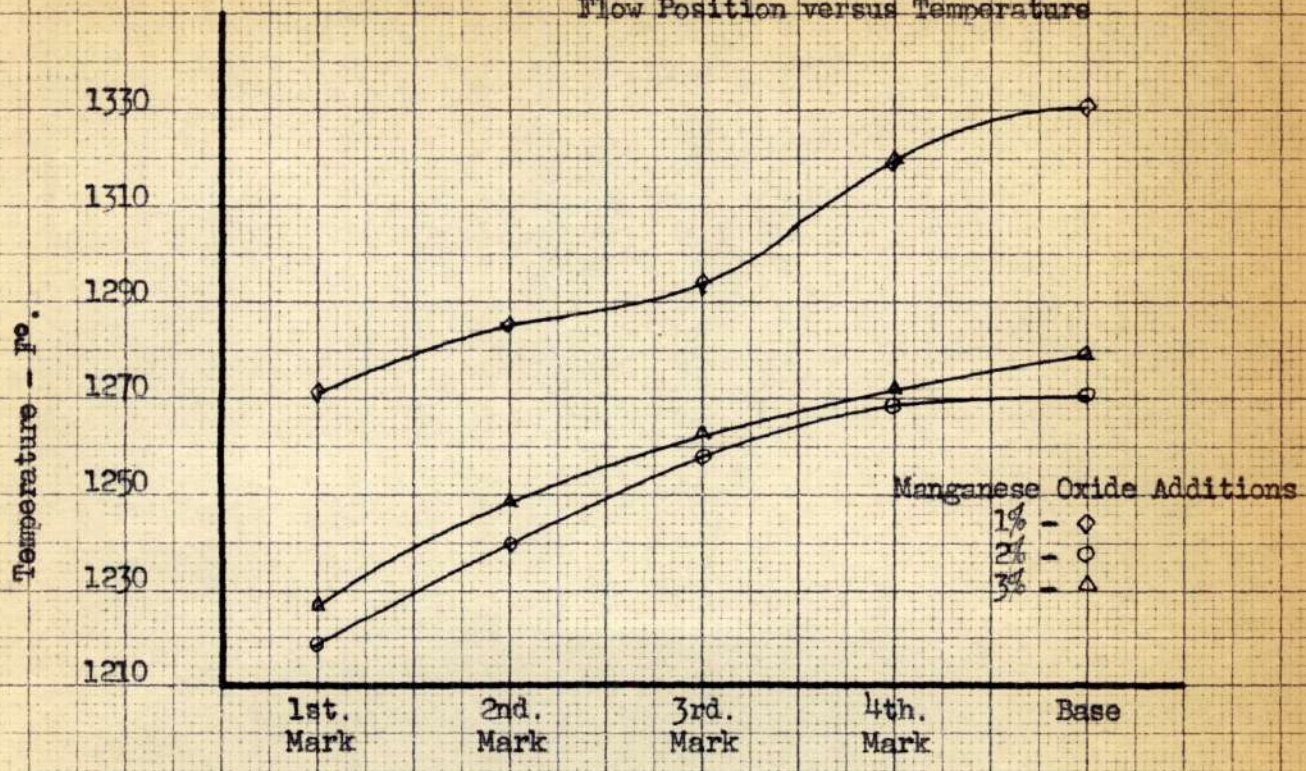


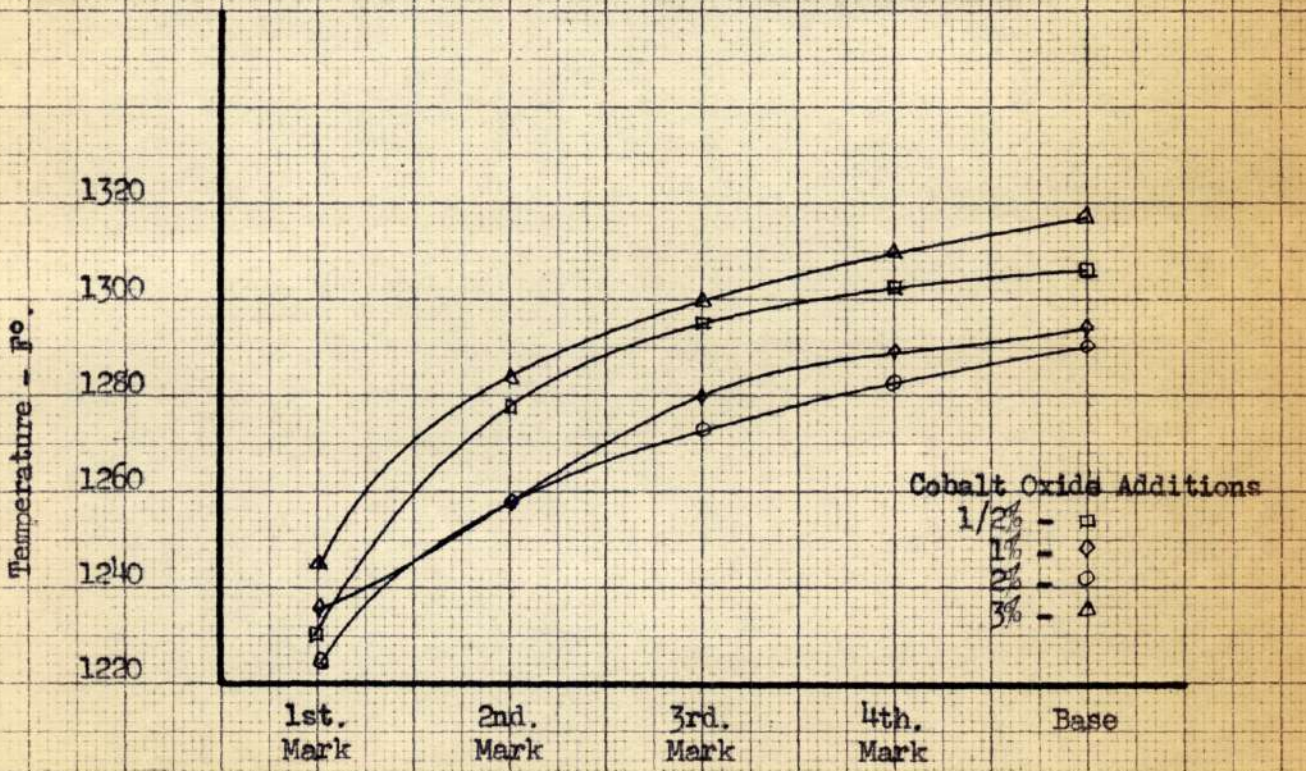


GRAPH 5

FUSION BLOCK TEST RESULTS

Flow Position versus Temperature





APPENDIX III.

DATA SHEET 1.

BASIC FRIT

Na_2O - .690	Feldspar	29
K_2O - .033	Quartz	16.1
CaO - .281	Borax (Anhyd.)	35.3
B_2O_3 - .130	Sodium Nitrate	4.1
Al_2O_3 - .058	Sodium Carbonate	10.6
SiO_2 -.350	Fluorspar	4.9

Approximate firing range 1500° to 1580° F.

Time and temperature used for test plates: 3.25 min. 1550° F.

MILL ADDITIONS

Frit	100%
Ferro Black Label Clay	6%
Brown Oxide	4%
Sodium Nitrite	1%
Water	45%

DATA SHEET 2.

BASIC FRIT

Plate No.	Co_3O_4	MnO_2	NiO_2	Sb_2O_3	Adherence	Surface	Gloss	Color	Acid Resistance
P 1		1%			None	Poor	5.37	R. Br.	B
P 2		2			None	Good	5.32	R. Br.	B
P 3		3			None	Good	5.36	R. Br.	B
P 4				1 %	Good	Good	5.29	Lt. Br.	B
P 5				2	Poor/None	Good	5.19	Lt. Br.	B
P 6				3	None	Good	5.15	Lt. Br.	B
P 7	.5%				Good	Good	5.29	Lt. Br.	B
P 8	1				Excellent	Good	5.33	D. Bl.	B
P 9	2				Excellent	Good	5.30	Very Dark	B
P 10	3				Excellent	Good	5.32	Very Dark	B
P 11			1 %		Poor	Good	5.25	Med. Br.	B
P 12			2		Good	Good	5.21	Med. Br.	B
P 13			3		Good/Poor	Good	5.30	Med. Br.	B

Colors:

R. Br.	Red Brown
Lt. Br.	Light Brown
Lt. Bl.	Light Blue
D. Bl.	Dark Blue
Very Dark	Very Dark Blue
Med. Br.	Medium Brown

DATA SHEET 3.

MODIFIED CONE COMPARISON TEST RESULTS

Tests Identified by Percent of Oxide Added

to Each Frit

All Runs Start at Zero Time and Are Fired at 1525° F.

Frit	Time Start of Bending	Time Finish	Total Time of Bending	Finish Position Calcined Cone
1/2% Co	55 Sec.	125 Sec.	70 Sec.	18 at 3 o'clock
1% Co	37	155	118	18 at 6 o'clock
2% Co	54	138	84	18 at 5 o'clock
3% Co	28	106	78	18 at 3 o'clock
1% Sb	81	131	50	18 at 2 o'clock
2% Sb	67	130	63	18 at 2 o'clock
3% Sb	75	160	85	18 at 5o ' clock
1% Ni	80	138	58	18 at 2 o'clock
2% Ni	64	139	75	18 at 3 o'clock
3% Ni	52	135	83	18 at 4:30 o'clock
1% Mn	24	108	84	18 at 4:30 o'clock
2% Mn	28	122	94	18 at 5 o'clock
3% Mn	26	133	107	18 at 5:30 o'clock

DATA SHEET 4.

FUSION BLOCK TEST RESULTS

Tests Identified by Percent of Oxide Added

to Each Frit

Frit	1st. Mark	2nd. Mark	3rd. Mark	4th. Mark	At Base
1/2% Co	1230	1278	1296	1312	1323
1% Co	1235	1258	1280	1288	1294
2% Co	1224	1258	1272	1282	1290
3% Co	1244	1284	1298	1310	1316
1% Sb	1214	1246	1261	1270	1277
2% Sb	1214	1236	1251	1260	1266
3% Sb	1231	1252	1265	1277	1283
1% Ni	1266	1277	1292	1305	1315
2% Ni	1263	1284	1296	1305	1314
3% Ni	1228	1249	1263	1275	1281
1% Mn	1272	1286	1293	1315	1320
2% Mn	1219	1239	1258	1269	1270
3% Mn	1227	1248	1264	1273	1279

DATA SHEET 5.

RESULTS OF CALCINED CONE CHECK

Heating rate of 150° F. per hour rise in temperature

Calcining Temperature	Cone Number	Position of Cone at End of Test
900° F.	022	down
Not Calcined	022	down
800° F.	022	5 o'clock
900° F.	021	3 o'clock
800° F.	021	2 o'clock
Not Calcined	021	1 o'clock

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DATA SHEET 6.

COMMERCIAL FRITS

MILL ADDITIONS

Frit	100%
Clay	6%
Sodium Nitrite	1%
Water	45%

Approximate firing range 1420° F. to 1500° F.

Time Fire Plate	Temperature	Start Cone	Finish Cone	Total
3.25 min.	1470° F.	24 Sec.	96 Sec.	72
3.50 min.	1470° F.	32 Sec.	108 Sec.	76

FRITS SHOWING MISBEHAVIOR

Material	G-1 %	M-2 %
Feldspar	37%	29.4%
Quartz	15.1%	15.1%
Borax	25%	20%
Sodium Carbonate	11.3%	20.6%
Sodium Nitrate	3%	8%
Fluorspar	8.6%	6.9%

Mill additions for G-1 and M-2 are same as above.