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TECHNICAL NOTE 2386

STUDIES OF HIGH-TEMPERATURE PROTECTION OF A TITANIUM-CARBIDE CERAMAL BY CHROMIUM-TYPE CERAMIC-METAL COATINGS

By Dwight G. Moore, Stanley G. Benner,
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National Bureau of Standards

Washington
June 1951

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STUDIES OF HIGH-TEMPERATURE PROTECTION OF A TITANIUM-CARBIDE CERAMAL BY CHROMIUM-TYPE CERAMIC-METAL COATINGS

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SUMMARY

Certain ceramals consisting of carbides bonded with a refractory metal have good strength properties at elevated temperatures, but are lacking in resistance to oxidation. In order for carbide ceramals of this type to find extensive use in jet-engine applications, there must first be some means of protecting them against rapid oxidation. It was demonstrated in earlier work at the National Bureau of Standards that such protection might be achieved by application of a ceramic-metal coating to the ceramal surface. In this earlier work, a coating was developed consisting of 80 parts by weight of chromium powder, 20 parts by weight of frit (glass), and 5 parts by weight of kaolin. When applied to a ceramal of 80 percent titanium carbide and 20 percent cobalt and fired in hydrogen, the coating was found to inhibit oxidation of the ceramal for extended periods at 1800° F.

The present investigation was made to gain additional information concerning the durability of the coating as affected by (a) frit content, (b) firing temperature, (c) firing time, and (d) number of coats. In addition, it was desired to determine the relative effectiveness of the coatings over a temperature range that might conceivably be encountered by ceramals in jet-engine operation.

To obtain such information, four coatings of varying frit content were prepared and applied to the ceramal. The resulting specimens were then studied with respect to oxidation penetration (depth of oxidation), transverse breaking load, and thermal shock resistance after prolonged heating in air at 1650°, 1800°, 2000°, and 2200° F. The variables of firing temperature, firing time, and number of coats were studied for one coating only. The results may be summarized as follows:

1. The protective effect of frit as a constituent of the coating was more pronounced in oxidation tests at 2200° F than at 2000° F and lower temperatures.

PERMANENT RECORD

2. Of the frit-chromium ratios used in the coatings studied, the 10:90 composition (parts by weight) gave the most promise, especially in tests at 2200° F.

3. Increasing the firing time from 10 to 60 minutes at 2200° F in hydrogen was effective in improving oxidation resistance at test temperatures of only 1650° and 2200° F.

4. Increasing the firing temperature from 2200° to 2350° F improved the resistance to oxidation penetration at 2000° and 2200° F but adversely affected specimen strength.

5. The use of two applications of coating instead of one brought about a general improvement in durability, especially in thermal shock behavior.

INTRODUCTION

In a previous report (reference 1) the development of a ceramic-metal coating was described. This coating, which contained 80 parts of chromium powder that would pass through a No. 100 U. S. Standard Sieve, 20 parts of a high-barium, alkali-free frit (glass), and 5 parts of kaolin, protected a titanium-carbide ceramal[1] against oxidation for as long as 200 hours at 1800° F.

The present paper is a sequel to the earlier one in that it reports the results of studies made to determine the effect of a number of variables upon the durability of the coating in the temperature range of 1650° to 2200° F. These variables included frit content, firing temperature, firing time, and the number of applied coats. Although all of the coatings were applied to a single ceramal, it was expected that the findings might be of value in the application of chromium-frit coatings to ceramals of other compositions.

The work covered by this report was done at the National Bureau of Standards under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

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[1]A ceramal is defined as any sintered or hot-pressed material consisting of a combination of ceramic and metallic ingredients. All carbides are considered as ceramic materials.

MATERIALS

The ceramal specimens were prepared by Kennametal, Inc., Latrobe, Pennsylvania. Their K-138 composition, which is reported to contain 80 percent by weight of titanium carbide bonded with 20 percent by weight of cobalt, was furnished. The specimens were $2\frac{1}{2}$ inches long by 3/4 inch wide with a 1/8-inch-diameter hole near one end. The average thickness of the 90 specimens included in the test was 0.132 inch with a standard deviation of 0.002 inch. All corners and edges of the specimens were rounded but the flat surfaces were not finish-ground.

The frit used in the preparation of all coatings was an alkali-free composition, with the assigned number 331. The composition of this frit is shown in table 1. It was selected because previous tests had indicated that its ingredients were noncorrosive toward the more common heat-resistant alloys at temperatures above 1500° F (reference 2), and for this reason it was concluded that it should be nonreactive toward the cobalt-bearing Kennametal K-138 and the chromium powder.

The chromium powder was procured from Charles Hardy, Inc., New York, New York and was prepared electrolytically. The spectrochemical analysis and particle size are given in table 2.

The clay used was a commercial grade of Florida kaolin.

PREPARATION AND APPLICATION OF COATINGS

Four coatings were prepared using the mill formulas listed in table 3. The frit-chromium ratios used ranged from 0:100 to 30:70 parts by weight in 10-part increments.

All 70 of the K-138 specimens to be coated were first sandblasted. The coatings were applied by dipping. Although an effort was made to adjust the consistency of the slip for dipping to give a uniform single-coat thickness after firing, actually there was an appreciable variation as can be seen by the thicknesses listed in table 4.

After application of the slip the specimens were dried at approximately 110° F, and then fired according to the plan given in table 4. All but 10 of the specimens were fired in the induction-type furnace as shown in figure 1. With this furnace, the specimens were raised from room temperature to 2200° F in approximately 4 minutes, held at

2200° ± 20° F for 10 minutes, and then allowed to cool to room temperature. During this entire period of firing, a purified hydrogen atmosphere was maintained in the furnace tube. The hydrogen was purified by first removing the oxygen with a palladium catalyzer and then drying the gas by passing it through activated alumina. The maximum safe operating temperature of the induction furnace with the uninsulated steel susceptor was 2250° F. Therefore, the 10 specimens requiring the 2350° F treatment (specimens 110 to 119) were fired in the small gas-fired furnace described in an earlier report (reference 1). In all cases, the coated specimens after firing had a gray metallic appearance with no signs of surface oxidation.

TEST PROCEDURE

Oxidation Tests

Oxidation tests in air were made on duplicate coated specimens, as well as on uncoated specimens. Separate tests were made at 1650° F for 400 hours, 1800° F for 200 hours, 2000° F for 100 hours, and 2200° F for 50 hours. The furnace used for these tests had a $7\frac{3}{4}$-cubic-foot capacity and was heated by silicon-carbide (Globar) elements. The furnace had air venting at the top and bottom. No forced ventilation was used.

The specimens were removed from the furnace for visual examination upon expiration of the respective cumulative treatment periods given in table 5. All specimens were treated for the maximum periods indicated.

The oxidation penetration[2] was determined by micrometer measurements. The initial measurements were made after the original sandblasting. At the completion of the test treatment at each of the four

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[2]Oxidation penetration is a term used initially by Whitman and Repko (reference 3) to express the distance or depth to which the oxide layer has penetrated into the ceramal. It may be defined mathematically by the formula

$$O.P. = \frac{t_o - t_1}{2}$$

where O.P. is oxidation penetration, t_o is the original ceramal thickness, and t_1 is the thickness of the unoxidized ceramal remaining after the heating treatment.

temperatures, the coating layer (or oxide layer in the case of the uncoated specimens) was removed by sandblast after which a final micrometer measurement was made to determine the residual thickness of the ceramal specimen. In the sandblasting operation, low-pressure air was used in conjunction with 60-mesh sand. Complete removal of the coating or oxide layer was indicated by a distinct difference between the appearance of the ceramal and that of the overlying layer of either coating or oxide.

Table 6 gives the average loss in thickness and average rate of oxidation penetration for all specimens included in the study. The value listed in each case is the average for duplicate specimens.

Figure 2 is a graph of average rate of oxidation penetration plotted against temperature for uncoated specimens and for specimens protected with coating B. The three points at 1625°, 1785°, and 2000° F, represented by the square symbols, were obtained from the data of Whitman and Repko (reference 3) for an uncoated ceramal containing 20 percent cobalt and 80 percent titanium carbide.

Figure 3 illustrates the reduction in thickness that occurs with the uncoated K-138 after 50 hours' heating in air at 2200° F in contrast with the minor change in thickness that occurs on a K-138 specimen protected with coating B.

Transverse Breaking Strengths

Following completion of the oxidation tests, all specimens were broken at room temperature using center-point, transverse loading on a 2-inch span. The specimens were broken in a conventional Tate-Emery testing machine using a loading rate of approximately 120 pounds per minute.

In these transverse breaking tests the load necessary to cause failure was used to express the data rather than the modulus of rupture, because the procedures required for exact measurement of cross sections of the specimens after long-time heating would have had adverse effects. For example, it was believed undesirable to remove the scale from the uncoated specimens before the transverse breaking test, or to remove the coatings from the coated specimens, since the testing would then be done under conditions that would not exist in service. Even if the scale (and the coatings) had been removed, an accurate cross section would have been difficult to obtain owing to the prevalence of surface pitting, especially at the higher test temperature.

Table 7 gives the results of the transverse breaking tests while figure 4 illustrates the trend of decreasing specimen strength with increasing temperature of heating for both uncoated specimens and specimens coated with the B composition. Figure 5 shows the effect of varying the frit content of the coating on the transverse breaking load for specimens heated 50 hours in air at 2200° F.

Thermal Shock Tests

The upper half of each specimen that was broken earlier in transverse testing was used in the thermal shock test. The test procedure was considerably more severe than the thermal shock encountered under service conditions but was the same as that used in a previous study (reference 1).

The test selected was carried out as follows: The $1\frac{1}{4}$-inch-long specimens were heated for 5 minutes at a temperature of 1000° F, then withdrawn quickly from the furnace, and the lower ends submerged to a depth of 1 inch in water at room temperature. After 5 minutes in the water, the specimens were examined and if no failure had occurred they were reinserted into the furnace at a temperature 100° F higher and the cycle repeated. This procedure was continued until failure occurred or until the specimens had been quenched from 2200° F without failure.

Table 8 gives the data for the thermal shock test.

RESULTS AND DISCUSSION

The effectiveness of the chromium-frit type coating in retarding the oxidation of the titanium-carbide K-138 ceramal at elevated temperatures is well brought out in figure 2. In this figure the K-138 protected with coating B shows very low oxidation rates at temperatures below 2200° F. Even at 2200° F the rate is very slow when compared with the uncoated material. The effect of this difference in rates at 2200° F is evident in figure 3. The thickness of the uncoated specimen in figure 3 decreased 0.085 inch from its original 0.132-inch thickness in 50 hours of heating while the specimens with coating B decreased only 0.001 inch from its initial 0.131-inch thickness.

The method of measuring decrease in thickness by sandblasting the layer of oxide and using a micrometer on the residual ceramal gave good agreement with the microscopic method as used by Whitman and Repko (reference 3).

Microscopic examination of the specimens after prolonged heating showed oxidation of the ceramal at localized breaks in the coating. This oxidation was of the same type as illustrated in reference 1. The localized areas of oxidation are responsible for the pits that are visible after sandblasting. It is believed that these localized effects can be prevented or greatly reduced in number through appropriate modification of the coatings or of the coating application techniques.

The average initial transverse breaking load for those K-138 specimens that were coated but received no subsequent heating treatment was 393 pounds (specimens 68, 69, 78, 79, 88, 89, 98, and 99 in table 7). This figure is somewhat lower than the 463-pound average load required to break specimens 148 and 149 which were uncoated but which received the same 10-minute firing at 2200° F as the coated specimens. This difference in strength was believed to result from stress concentrations caused by a slight unevenness in the coating surface. Because of this lack of flatness, the load at the center knife edge was not uniformly distributed across the width of the specimen as was the case with the uncoated specimens. If this explanation is correct the difference in apparent strength should disappear at a test temperature of 1800° F, because the coating would be capable of creep and thus stress concentrations would be eliminated. That such is the case was indicated by several transverse breaking tests at 1800° F. In these tests, with a loading rate of about 20 pounds per minute, the breaking load for two coated specimens (coating C) was 285 and 352 pounds while two uncoated specimens showed breaking loads of 271 and 318 pounds. The average modulus of rupture from these data for the coated specimens was 78,150 psi while the average for the two uncoated specimens was 68,800 psi. This latter value for the uncoated specimens is in fair agreement with the data reported by Deutsch, Repko, and Lidman (reference 4) but is about 40 percent lower than the modulus of rupture reported by Redmond and Smith (reference 5).

The effect of variation in frit content of the coatings was most prominent in the resistance to oxidation penetration at 2200° F. At this temperature, the presence of frit in the coating in any of the amounts tried was beneficial in retarding oxidation of the ceramal, but the 10:90 frit-chromium combination (coating B) was outstandingly effective. This same coating also gave stronger specimens than any of the other coatings applied under the same conditions, the greater strength prevailing for all test temperatures. The 30:70 frit-chromium coating (coating D) provided the best protection against oxidation at the lower test temperatures.

The reason why the presence of a frit in the coating is more essential at 2200° F than at the lower temperatures is believed to be associated with the diffusion layer, or bond layer, that forms at the interface when the coatings are fired on the ceramal. This layer, which

is illustrated by several photomicrographs in the earlier study (reference 1), may, of itself, give fairly good protection at the lower test temperatures, but at 2200° F the chromium diffusion layer apparently becomes nonprotective. Practically all of the protection at this higher temperature results from the presence of the glassy phase.

That the chromium, of itself, provides reasonably good protection at a test temperature of 1800° F was demonstrated by electrodepositing chromium on one K-138 specimen to a thickness of 1 mil. This was done by flashing the plating at a temperature of 50° C for 5 minutes, using a current density of 60 amperes per square decimeter. After bonding the chrome plate by heating to 2200° F for 10 minutes in dry hydrogen, the specimen was found to have excellent durability at 1800° F except at corners and edges where satisfactory coverage in the plating operation was not achieved. The coating prepared from chromium powder but containing no frit also gave good protection at the lower temperatures but it is significant that the performance of this frit-free coating (coating A) was exceeded by that of one or more of the frit-bearing coatings in the tests for strength of specimen at all test temperatures and for resistance to oxidation at 1650°, 1800°, and 2200° F.

In addition to frit content, other variables included in the study were (a) firing time, (b) firing temperature, and (c) the number of coatings applied. It was not within the scope of the investigation to test all possible combinations of the four coatings with these variables. Accordingly, a coating was selected for study of these factors which was neither the best nor the poorest of the four under consideration and which had the same frit-chromium ratio (20:80) as the A-479M coating reported in reference 1.

Tables 6, 7, and 8 include results for coating C (1) fired 10 minutes at 2200° F, (2) fired 60 minutes at 2200° F, (3) fired 10 minutes at 2350° F, and (4) applied as two separate coats, each fired 10 minutes at 2200° F.

The increased firing time at 2200° F was beneficial in improving resistance to oxidation penetration at 1650° and 2200° F, while the increase of firing temperature from 2200° to 2350° F improved resistance to oxidation penetration at 2000° and 2200° F. The higher firing temperature adversely affected resistance to oxidation penetration at 1650° and 1800° F. Also, in general, it adversely affected specimen strength both before and after the heat treatments. This reduction in strength, which was apparent in spite of a considerable spread in transverse strength results (see table 7), implies that the ceramal underwent some deterioration in properties as a result of being heated to 2350° F.

Table 8 shows that in the thermal shock test the K-138 cracked in many cases before the coating flaked, thus making any comparison between the various coatings difficult. In those cases where failure was in the

coating rather than in the ceramal, no consistent pattern could be detected except that the two-coat application appeared to give outstanding thermal shock behavior. This coating did not flake after any of the heating treatments except after 50 hours at 2200° F; further, it appeared to protect the ceramal against cracking. The use of two applications of coating was also generally beneficial in retarding oxidation penetration, especially as compared with a similarly fired one-coat application when tested at 2200° F. It did not appear to have any significant effect upon specimen strength.

This study of the variables of firing time, firing temperature, and number of coats (for coating C) should serve as a valuable guide in any further development of this type of coating that may be undertaken. The higher firing temperature should be avoided since it has some harmful effects and the beneficial effects can be achieved in other ways. Studies of frit-chromium ratios near 10:90 (between 5:95 and 15:85) are indicated in order to determine more closely what ratio gives certain categories of peak performance that occurred with the 10:90 composition (coating B) in this study. Preparation of coatings having the selected frit-chromium ratio and their application as two separate coats, each fired for longer than 10 minutes (possibly 1 hr) at 2200° F, should lead to the development of a coating having superior properties.

Recent work by Redmond and Smith (reference 5) has demonstrated that the oxidation resistance of the ceramal containing 80 percent titanium carbide and 20 percent cobalt can be greatly improved by replacing 15 percent of the titanium carbide with a solid solution of columbium carbide, tantalum carbide, and titanium carbide. These authors report that when this is done the modulus of rupture is decreased by as much as 30,000 psi both at room temperature and at 1800° F. Also, if the titanium-carbide ceramals were used in any substantial quantity for high-temperature service, it would seem that columbium carbide should be eliminated from the composition, if possible, because of the probable shortage of columbium in the event of a national emergency. For both of these reasons, the use of a protective coating of the chromium-frit type merits consideration as a method of achieving the necessary resistance to oxidation.

SUMMARY OF RESULTS

Four ceramic-metal coatings of varying frit content were prepared and applied to ceramals containing 80 percent chromium and 20 percent cobalt. After prolonged heating in air at temperatures of 1650°, 1800°, 2000°, and 2200° F, oxidation penetration, transverse breaking load, and thermal shock resistance of the various specimens were

determined. The effects of varying firing time, firing temperature, and number of coats applied were studied for one coating. The results obtained may be summarized as follows:

1. The protective effect of frit as a constituent of the coating was more pronounced in heating tests at 2200° F than at 2000° F and lower temperatures.

2. Of the frit-chromium ratios used in the coatings studied, a ratio of 10:90 (parts by weight) gave the most promise, especially in tests at 2200° F.

3. Increasing the firing time from 10 to 60 minutes at 2200° F in hydrogen was effective in improving oxidation resistance at test temperatures of only 1650° and 2400° F.

4. Increasing the firing temperature from 2200° to 2350° F improved the resistance to oxidation penetration at 2000° and 2200° F, but adversely affected specimen strength.

5. The use of two applications of coating instead of one brought about a general improvement in durability, especially in thermal shock behavior.

National Bureau of Standards
Washington, D. C., June 30, 1950

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TABLE 1.- BATCH COMPOSITION AND COMPUTED OXIDE

COMPOSITION OF FRIT 331

[Maximum smelting temperature, 2425° F;
smelting time, $2\frac{1}{2}$ to 3 hr]

(a) Raw batch composition.

Constituent	Parts by weight
Flint (SiO_2)	38.00
Barium carbonate	56.63
Boric acid	11.50
Calcium carbonate	7.14
Beryllium oxide	2.50
Zinc oxide	5.00
	120.77

(b) Computed oxide composition.

Constituent	Percent by weight (1)
SiO_2	38.0
B_2O_3	6.5
BaO	44.0
CaO	4.0
ZnO	5.0
BeO	2.5
	100.0

[1]After melting.

TABLE 2.- SPECTROCHEMICAL ANALYSIS AND PARTICLE SIZE OF MINUS 200 MESH ELECTROLYTIC CHROMIUM POWDER USED IN PREPARING CHROMIUM-FRIT COATINGS

(a) Spectrochemical analysis[1].

Relative amounts of reported elements			
Trace (<0.01 percent)	Very weak (<0.01 percent)	Weak (0.01 to 1 percent)	Very strong (>1 percent)
Ag Al Cu Mg Ni Pb Sn Ti	Ca Co Mn Mo	Fe Si	Cr

(b) Particle size by micro-projection[2].

Particle	Size (microns)
Largest	100
Smallest	1
Estimated average	8-10

[1]Analysis by the Spectrochemistry Section of NBS.
[2]Determination made by the Fineness Laboratory of NBS.

TABLE 3.- MILL FORMULAS FOR EXPERIMENTAL

CERAMIC-METAL COATINGS

Coating	Parts by weight of -					Milling time (hr) (3)
	Chromium powder (1)	Frit 331 (2)	Florida kaolin	Sodium nitrite	Water	
A	1500	0	75	0.6	500	1
B	1350	150	75	.6	525	3
C	1200	300	75	.6	525	3
D	1050	450	75	.6	525	3

[1]Electrolytically prepared; minus 200 mesh.

[2]Minus 40 mesh after 3-hr dry milling.

[3]In a 1-gal ball mil.

TABLE 4.- FIRING CONDITIONS AND COATING THICKNESSES

FOR FOUR COATINGS AS APPLIED TO SEVEN

SETS OF SPECIMENS

Specimen	Coating applied	Coating composition (parts by weight) (1)		Firing conditions		Coating thickness (mils) (2)
		Frit	Cr powder	Time (min)	Temperature (°F)	
60-69	A	0	100	10	2200	3.2-5.8
70-79	B	10	90	10	2200	2.5-3.7
80-89	C	20	80	10	2200	6.6-8.5
90-99	D	30	70	10	2200	3.7-6.7
100-109	C	20	80	60	2200	7.0-11.2
110-119	C	20	80	10	2350	3.7-8.2
120-129	C[3]	20	80	10	2200	7.5-13.6
140-149	None	0	0	10	2200	0

[1]All coatings contained 100 parts of frit plus chromium powder in addition to 5 parts by weight of clay.

[2]Values listed give range in average thickness for each group of 10 specimens.

[3]Two coats were applied, each coat being fired for 10 min at 2200° F.

TABLE 5.- TEST TEMPERATURES AND CUMULATIVE HEAT TREATMENT PERIODS FOR BOTH COATED AND UNCOATED K-138 SPECIMENS

Test temperature (°F) (1)	Duration of test (hr)	Cumulative treatment periods prior to inspections (hr)
1650	400	20, 60, 100, 200, 300, 400
1800	200	20, 40, 80, 120, 160, 200
2000	100	4, 10, 30, 50, 70, 100
2200	50	1, 3, 6, 10, 16, 33, 50

[1]Furnace temperatures controlled to ±3° F with a proportional-type controller.

TABLE 6.- AVERAGE LOSS IN THICKNESS AND AVERAGE RATE OF OXIDATION PENETRATION FOR DUPLICATE SPECIMENS OF K-138 AFTER PROLONGED HEATING AT FOUR TEST TEMPERATURES

Coating	Average loss in thickness[1] (mils) after -				Average rate of oxidation penetration[2] (mils/hr) after -			
	400 hr at 1650° F	200 hr at 1800° F	100 hr at 2000° F	50 hr at 2200° F	400 hr at 1650° F	200 hr at 1800° F	100 hr at 2000° F	50 hr at 2200° F
A[3]	7.2	1.5	0.7	81.5	0.0090	0.0038	0.0035	0.8150
B[3]	12.3	8.9	3.5	4.5	.0154	.0222	.0175	.0450
C[3]	2.1	.5	1.1	48.2	.0026	.0012	.0055	.4820
D[3]	.2	.1	.9	45.7	.0002	.0002	.0045	.4570
C[4]	0	2.1	1.1	8.8	0	.0053	.0055	.0880
C[5]	17.3	9.6	.9	11.6	.0216	.0240	.0045	.1160
C[6]	0	0	1.3	10.2	0	0	.0065	.1020
None	22.2	40.6	80.3	87.0	.0278	.1015	.4015	.8700

[1]Determined from difference in specimen thickness before coating application and after test treatment as indicated.

[2]Calculated by dividing loss in thickness by 2 and dividing this value by time at temperature.

[3]Fired 10 min at 2200° F.

[4]Fired 60 min at 2200° F.

[5]Fired 10 min at 2350° F.

[6]Two-coat application.

TABLE 7.- TRANSVERSE LOAD REQUIRED TO BREAK BOTH COATED AND UNCOATED K-138 SPECIMENS AFTER HEAT TREATMENTS AS INDICATED.

Coating	Specimen	Heat treatment (1)	Break load (lb)	Coating	Specimen	Heat treatment (1)	Break load (lb)
A[2]	60	1	334	C[3]	100	1	268
	61	1	306		101	1	282
	62	2	268		102	2	283
	63	2	293		103	2	278
	64	3	304		104	3	249
	65	3	304		105	3	170
	66	4	166		106	4	218
	67	4	160		107	4	237
	68	5	411		108	5	329
	69	5	431		109	5	335
B[2]	70	1	420	C[4]	110	1	(5)
	71	1	381		111	1	156
	72	2	297		112	2	140
	73	2	342		113	2	264
	74	3	341		114	3	264
	75	3	297		115	3	278
	76	4	281		116	4	221
	77	4	240		117	4	169
	78	5	302		118	5	292
	79	5	472		119	5	360
C[2]	80	1	(5)	C[6]	120	1	282
	81	1	386		121	1	271
	82	2	293		122	2	327
	83	2	256		123	2	282
	84	3	284		124	3	251
	85	3	274		125	3	286
	86	4	224		126	4	198
	87	4	193		127	4	236
	88	5	385		128	5	360
	89	5	375		129	5	332
D[2]	90	1	346	N[7]	140	1	354
	91	1	352		141	1	399
	92	2	268		142	2	239
	93	2	330		143	2	228
	94	3	254		144	3	118
	95	3	282		145	3	173
	96	4	186		146	4	138
	97	4	214		147	4	87
	98	5	354		148	6	432
	99	5	411		149	6	495

[1]Figures denote the following heat treatments:
1 400 hr in air at 1650° F
2 200 hr in air at 1800° F
3 100 hr in air at 2000° F
4 50 hr in air at 2200° F
5 firing treatment only
6 firing treatment of 10 min at 2200° F in hydrogen

[2]Fired 10 min at 2200° F.

[3]Fired 60 min at 2200° F.

[4]Fired 10 min at 2350° F.

[5]Loading rate uncontrolled, failed by impact.

[6]Two-coat application; each coat fired for 10 min at 2200° F.

[7]Not coated.

TABLE 8.- RESULTS OF THERMAL SHOCK TESTS WITH COATED AND UNCOATED K-138 SPECIMENS BOTH BEFORE HEATING IN AIR AND AFTER PRIOR HEAT TREATMENTS IN AIR AS INDICATED

Specimen	Coating	Frit in coating (parts by weight) (1)	Temperature of first thermal shock failure (°F) (2)	Type of failure
(a) No prior heat treatment.				
68	A[3]	0	1100	Cracking of K-138
78	B[3]	10	1200	Do.
88	C[3]	20	1300	Do.
99	D[3]	30	2100	Flaking of coating
109	C[4]	20	1100	Do.
118	C[5]	20	1800	Cracking of K-138
128	C[6]	20	2000	Flaking of coating
148	None	0	2100	Cracking of K-138
(b) After 400 hours' heating in air at 1650° F.				
60	A[3]	0	1500	Cracking of K-138
70	B[3]	10	1500	Flaking of coating
80	C[3]	20	1200	Cracking of K-138
91	D[3]	30	2000	Do.
101	C[4]	20	2000	Do.
110	C[5]	20	2000	Do.
120	C[6]	20	>2200	No failure
140	None	0	1300	Flaking of oxide layer
(c) After 200 hours' heating in air at 1800° F.				
62	A[3]	0	1500	Cracking of K-138
72	B[3]	10	2100	Do.
82	C[3]	20	1500	Do.
92	D[3]	30	2000	Do.
102	C[4]	20	2000	Do.
112	C[5]	20	>2200	No failure
122	C[6]	20	>2200	Do.
142	None	0	1200	Flaking of oxide layer
(d) After 100 hours' heating in air at 2000° F.				
64	A[3]	0	1500	Cracking of K-138
74	B[3]	10	2100	Do.
84	C[3]	20	1300	Do.
94	D[3]	30	2000	Do.
104	C[4]	20	2200	Flaking of coating
114	C[5]	20	1800	Do.
124	C[6]	20	>2200	No failure
145	None	0	1500	Flaking of oxide layer
(e) After 50 hours' heating in air at 2200° F				
66	A[3]	0	1900	Flaking of coating
76	B[3]	10	2200	Do.
86	C[3]	20	2000	Do.
96	D[3]	30	2100	Do.
106	C[4]	20	2100	Cracking of K-138
117	C[5]	20	2100	Do.
127	C[6]	20	2200	Flaking of coating
146	None	0	1600	Flaking of oxide layer

[1]See table 4 for other constituents present.
[2]See text for description of test procedure.
[3]Fired 10 min at 2200° F.
[4]Fired 60 min at 2200° F.
[5]Fired 10 min at 2350° F.
[6]Two-coat application; each coat fired for 10 min at 2200° F.

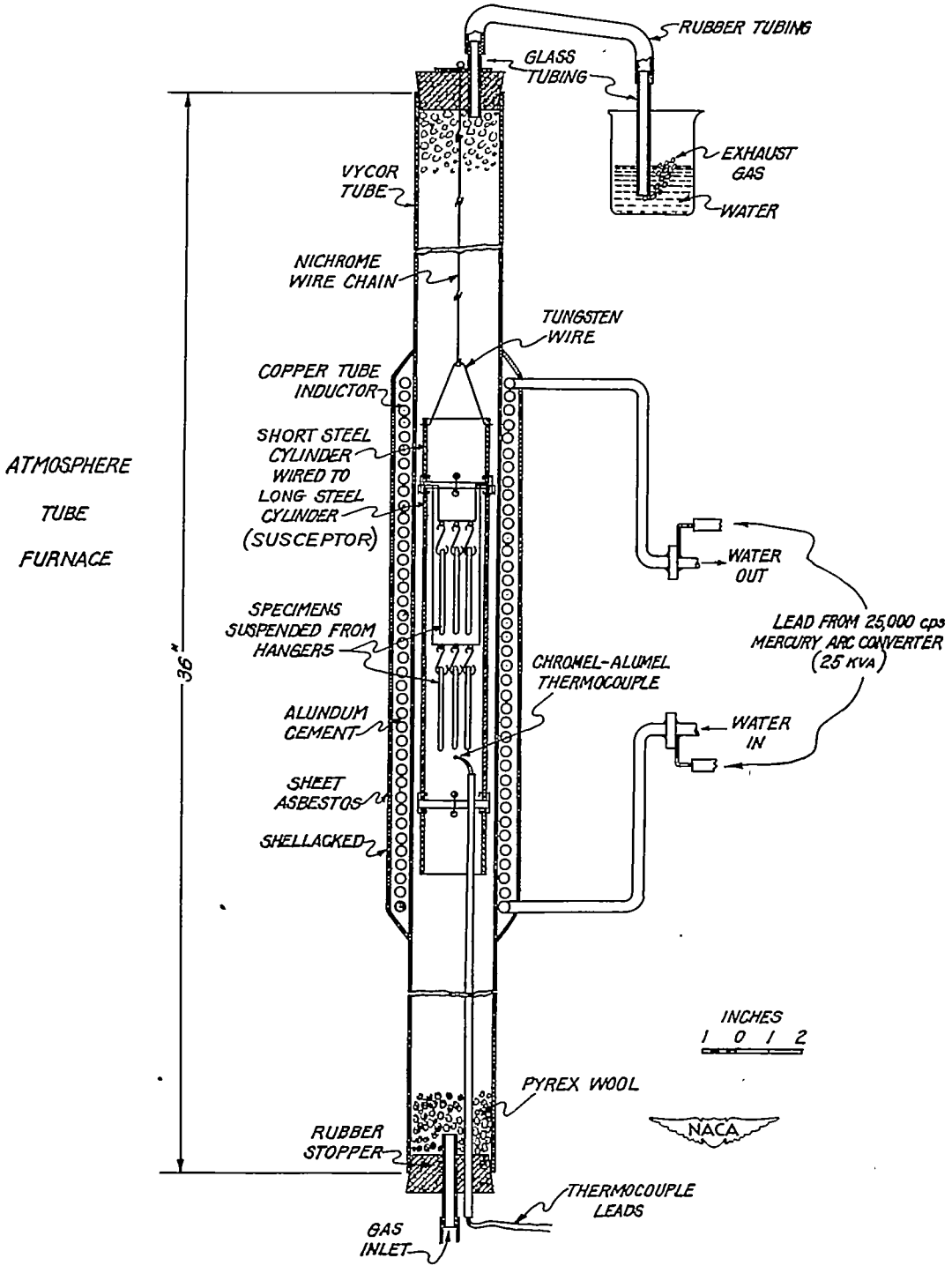


Figure 1.- Schematic drawing of induction furnace used for firing chromium-frit coatings to the K-138 ceramal. Normal firing consisted of heating for 10 minutes at 2200° F in an atmosphere of purified hydrogen.

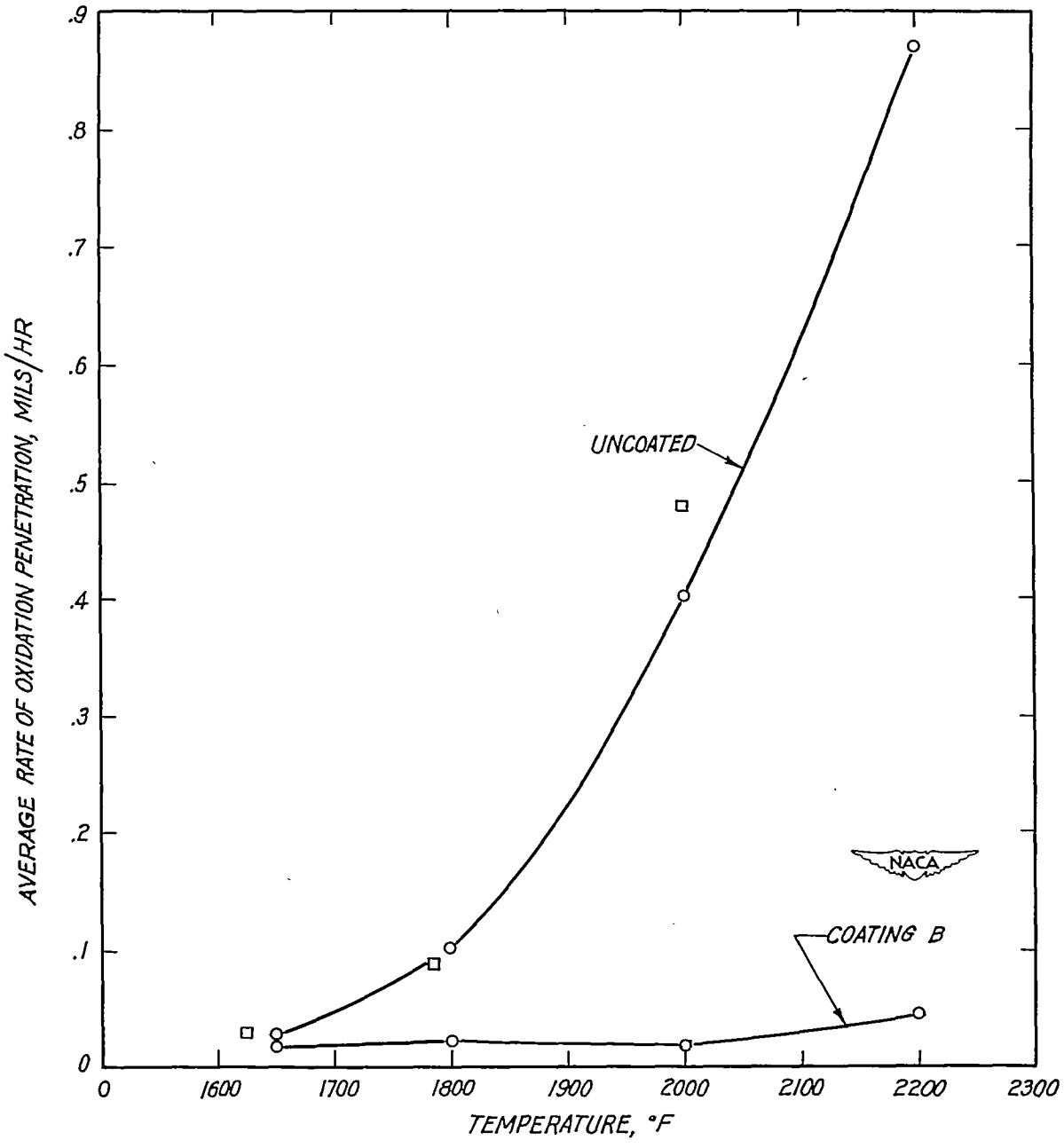
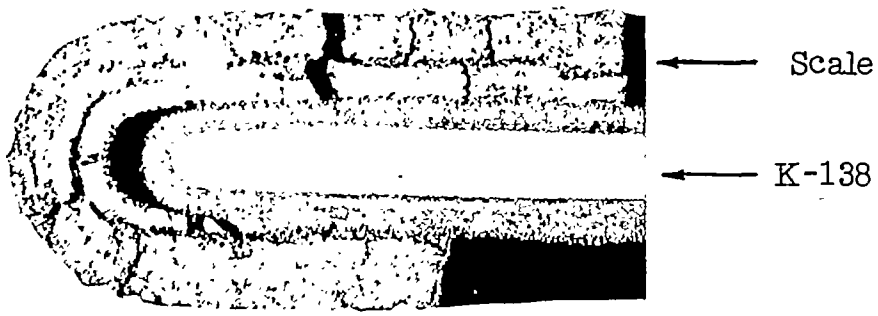
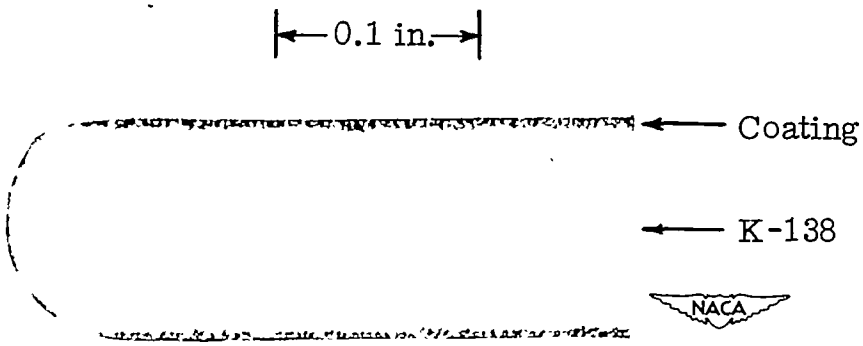


Figure 2.- Curves showing effect of temperature on the average rate of oxidation penetration for uncoated K-138 ceramal specimens and similar specimens protected with coating B. (Squares represent data taken from reference 3.)



(a) Uncoated K-138 ceramal.



(b) K-138 ceramal with chromium-frit coating.

Figure 3.- Two sections of a K-138 ceramal after 50 hours' heating in air at 2200° F. The uncoated specimen lost 0.085 inch of its original 0.132-inch thickness while a similar specimen protected with coating B shows practically no loss in thickness. Black areas in (a) are voids in scale layer.

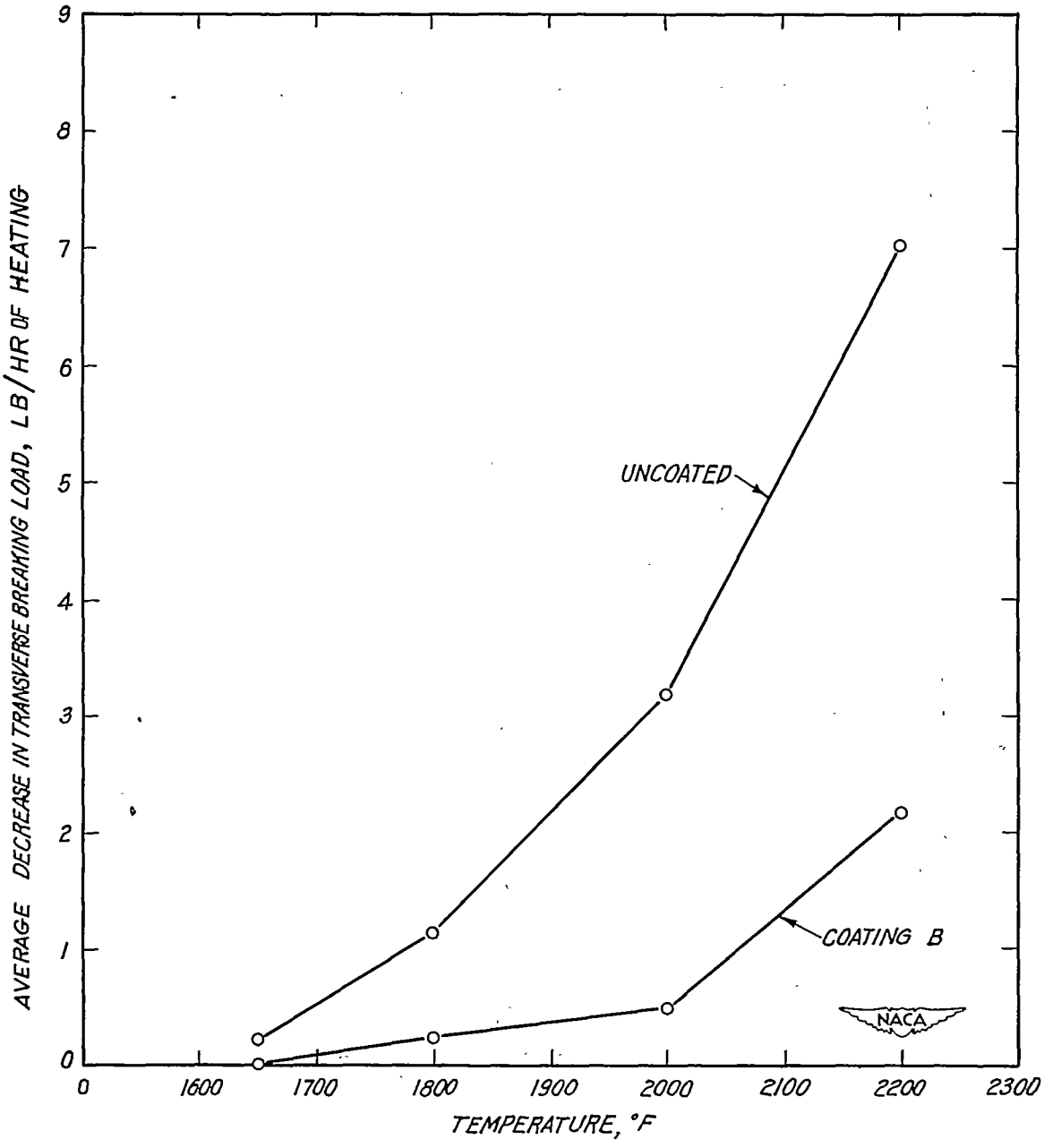


Figure 4.- Curves showing effect of temperature on average decrease in transverse breaking load for uncoated K-138 specimens and similar specimens protected with coating B.

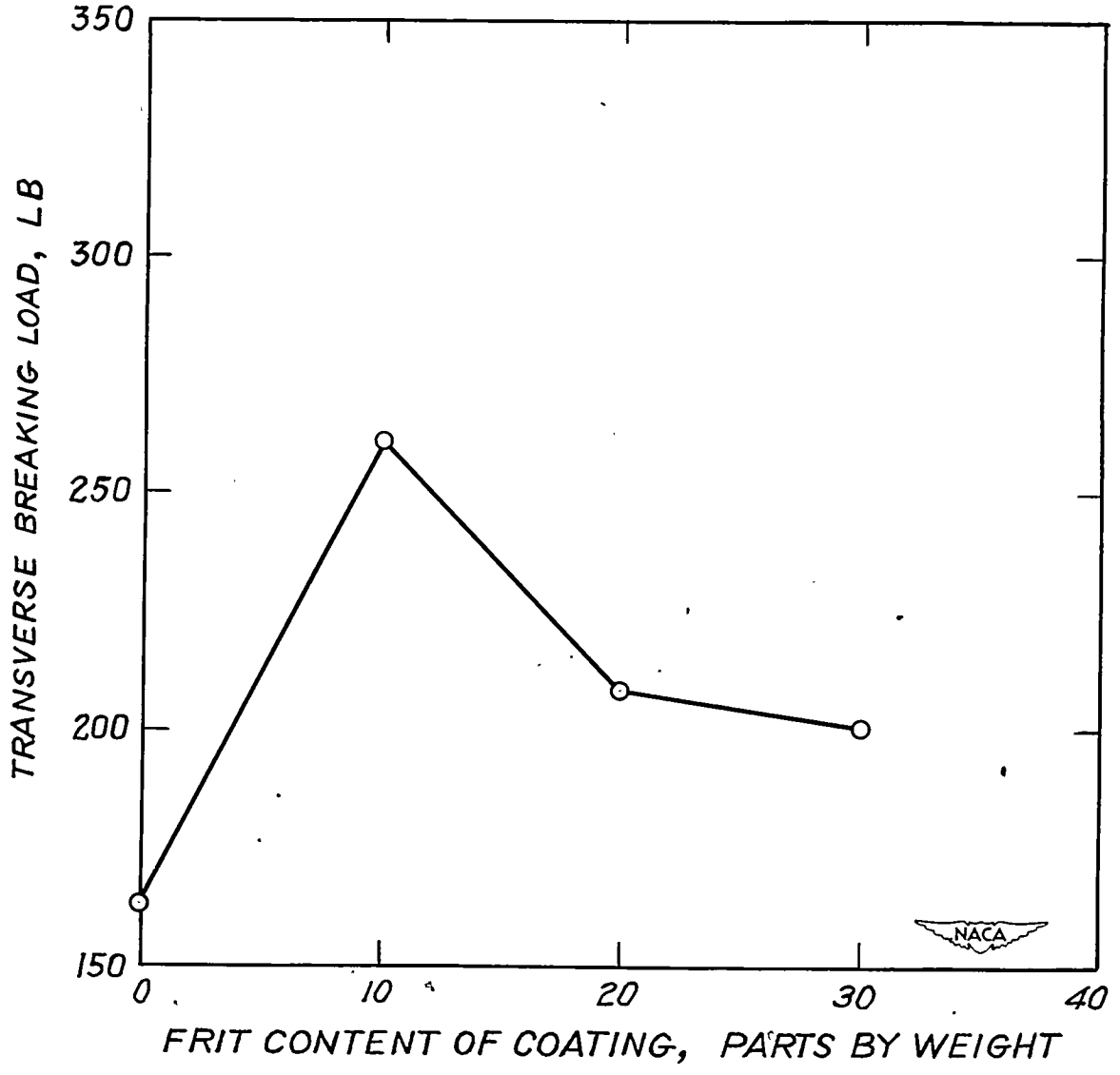


Figure 5.- Effect of frit content of coating on transverse load required to break K-138 ceramal specimens after 50 hours' heating in air at 2200° F.

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