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NATIONAL BUREAU OF STANDARDS REPORT

10382

Progress Report

July 1 through October 31, 1970

**DEVELOPMENT OF METHODS OF TEST
FOR QUALITY CONTROL OF PORCELAIN ENAMELS**

Porcelain Enamel Institute Research Associateship



U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS

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DEVELOPMENT OF METHODS OF TEST FOR QUALITY CONTROL OF PORCELAIN ENAMELS

by

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Sponsored

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PORCELAIN ENAMEL INSTITUTE RESEARCH ASSOCIATE SHIP

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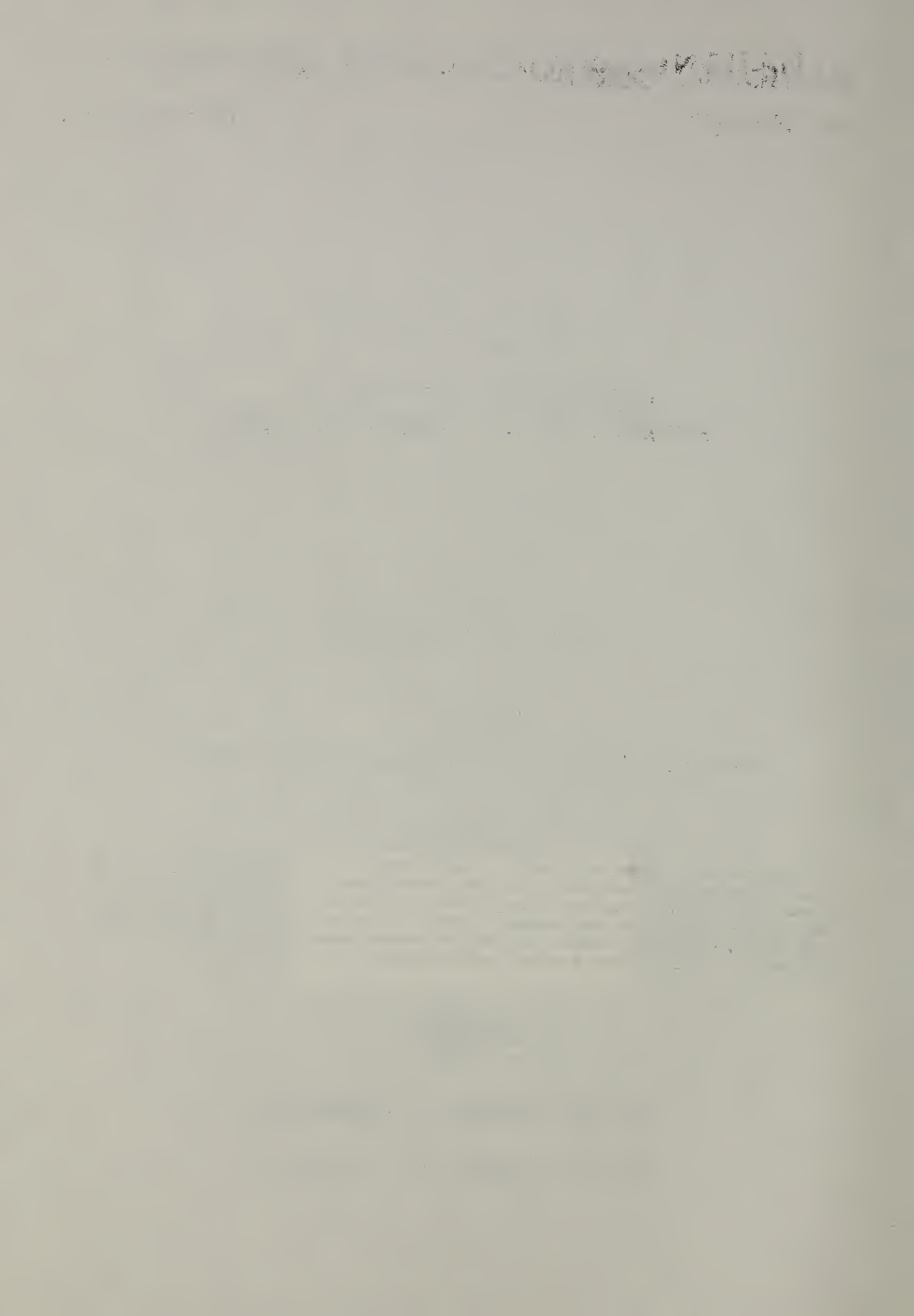
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I N T R O D U C T I O N

Three types of test procedures aimed at the evaluation of adherence quality particularly of cover coats of porcelain enamel direct-to-steel, are discussed in this report. The first is tensile testing with an adhesion tester. A description of the device used and preliminary results obtained therewith were described in the previous report of this series. Additional experience with this test device is described and a conclusion reached with regard to its use in adherence measurement.

The PEI deforming press together with an adherence meter to evaluate deformed specimens was developed in the late 1940's and has been limited in use since that time. Experience in the use of these devices has shown an insensitivity to distinguish between porcelain enamels in the "good" to "excellent" range of adherence quality. The drop weight method of deforming porcelain enameled ware is widely used but with a multiplicity of ball and die sizes, and impact energies. Many users of the drop weight device attain a more severe deformation treatment than is obtained with the PEI hydraulic deforming press and, as a result, make it difficult to distinguish between the middle and lower grades. Several modifications of the hydraulic deforming press are described and results of their use are given.

An introductory survey of users of the drop weight adherence test was made and the results are described.

A. Tension Tests with Adhesion Tester.

(1) The adhesion tester, described and illustrated in the previous report was used for additional tensile tests in an attempt to numericalize adherence of direct-on cover coats. A specially prepared series of specimens was used in which the same enamel was applied to substrates of 20, 18 and 14 gage. The results given in Table 1 show that the failure stresses for specimens on 14 gage metal were nearly double those of specimens prepared with 20 gage metal. It is thought that the heavy metal greatly reduced strain in the substrate and delayed failure due to microcracking around the circumference of the button until the stress level was greater than in the 20 gage specimens. The lower part of Table 1 shows the same increased failure stresses when applied to heavy gage specimens, through the use of buttons of materially reduced diameter (3/8-inch dia). The left side of Figure 1 schematically illustrates the localized strain when the large sized buttons were employed. Because of the close fitting hold-down plate the strain was concentrated in a small area around the button circumference. The use of the smaller diameter buttons, with the same hold-down plate as before, is shown in the center of Figure 1 at the same stress level as the left hand figure. The small button geometry allows

the substrate strain to be distributed over a much larger area and microcracks at the button circumference are delayed until a much greater stress has developed as shown at the right side of Figure 1.

(2) Figure 2 illustrates the effect of heavy gage substrates on the failure stress in button testing. In all cases shown the failure stresses were greater as the substrate thickness was increased whether the buttons were large or small. Specimens which had only isolated discs of enamel applied were prepared to show the effect of the presence or absence of a shearing stress in removing the discs. The third block diagram in Figure 2 shows that greater stress was required to shear out a button-sized disc from completely coated specimens than was required to overcome the adherence when shearing stress was absent in the isolated disc situation. These results could also result from greater rigidity of the continuously coated specimens.

Table 3 allows the comparison of substrate rigidity to be carried one step further by giving the failure stress of buttons removed from a composite specimen system in which two 20 gage completely coated specimens were joined back-to-back with epoxy. The thickness of metal in this sandwich of two 20 gage specimens corresponded roughly to that in a 14 gage specimen. It can be seen that the 1640 psi failure stress for the composite sandwiches was materially greater than when a 14 gage substrate was completely coated and tested to tensile failure.

(3) A summary of tensile tests illustrating the effect of substrate rigidity on failure stresses is given in Table 3. It can be seen that, from the least rigid geometry at the top to the most rigid at the bottom, the failure stresses increase. These data lead to a conclusion regarding the unsuitability of the adhesion tester for measuring adherence in direct-on porcelain enamel systems. The numerical failure stress in this type of tensile testing appears to be influenced, to a large extent, by the rigidity of the substrate and for this reason does not reflect the adherence of the enamel coating to the substrate.

B. Modifications of the PEI Hydraulic Deforming Press.

(1) The PEI deforming press employs a one-inch ball indenter attached to the fixed frame of the press, and is used for 20 gage metal with a die of 0.156 inch depth of appropriate diameter and a 1/8-inch radius rounded edge. Additional indenter balls of 1/2 and 3/4-inch diameters were made to attach to the press frame in the same way as the present one-inch indenter. Die dimensions for use with the modified indenters are given in Figure 3.

(2) A preliminary experimental test pattern was designed to observe the operation of the ball indenters and associated die designs, with various hydraulic pressures on duplicate specimens of both "good" and "poor" adherence properties. The results of this program are given in Tables 4 and 5. The water immersion treatment gave significantly different results only in tests on

the enamel with "poor" adherence. Specimens deformed with a 500-pound pressure were not significantly different from those which employed greater pressures. The three indenter sizes gave indentations which reflected marked differences in severity.

(3) The various DOAAC grades 1 through 6, in which adherence quality was influenced by controlled nickel deposits, were tested with the three indenter sizes, using 2,000 pounds pressure, both dry after water immersion. The values of meter counts given in Table 6 are the means for four indentations in the six variable combinations given.

It can be noted that the one-inch ball size fails to distinguish between the best adherence grades 2, 3, 4 and 5. This is the situation that this series of modifications was hoped to rectify, by creating more severe deformations. The one-half inch die resulted in deformations, for this specimen series, from about 30 to 80 counts per indentation. The deformations from the 3/4-inch ball size and its die, although somewhat more variable, covered a much wider range of about zero to 132 counts.

In order to make a more valid comparison of different indenter sizes the meter counts given in Table 6 were converted to the number of counts relative to their areas, and are given in Table 7. From this table it can be seen that the 3/4-inch die produced deformations which were rated by relative meter counts from 0 to 100 for those specimens immersed in water before measurement. The "dry" treatment gave results which were some-

what more variable than those specimens which were water immersed and oven dried before measurement. The relative counts on specimens deformed with the 1/2-inch die covered a somewhat narrower range, from 40 to 100, whether measured dry or after water immersion. A graphical representation of the preceding results is shown in Figure 4 which shows the relation of relative counts to the amount of deposited nickel. The results shown in Figure 4 and given in Table 7 lead to the selection of the following as a desirable deforming and measurement procedure:

- Indenter size, 3/4-inch diameter,
- Die configuration shown in Figure 3 for use with 3/4-inch indenter,
- Deforming pressure, 2,000 pounds
- Measurement of deformed specimens after water immersion with a PEI meter.

(4) The work described in the previous paragraphs involved a water immersion step for one minute and oven drying for 30 minutes before meter counting. An experiment was performed which showed that towel drying could replace oven drying without serious loss of precision. The results of this experiment are given in Table 8. The mean number of counts measured on 20 indentations was the same whether oven dried or towel dried after water immersion.

(5) An attempt was made to observe the degree of correlation between the severity of deformations obtained with a hydraulic press and with a drop weight device. Figure 5 shows the correlations

of relative meter counts for the six DOAAC adherence grades between specimens hydraulically deformed with 1/2- and 3/4-inch indenters on the one hand, and specimens drop weight deformed with 9/16-inch indenter, 3/4-inch die at 70, 80 and 90 inch-pounds on the other hand. A good correlation was found for specimens hydraulically deformed with a 1/2-inch indenter and the drop weight deformed specimens for impact energies in the 70 to 90 inch-pound range.

An upcoming program of drop weight testing of all DOAAC grades of adherence will seek out drop weight test parameters which will provide a severity of deformation comparable to that obtained hydraulically with a 3/4-inch indenter discussed in paragraph 3 above.

C. A Survey of Industry Practice in Adherence Estimation by Drop Weight Testing.

The use of drop weight devices for deforming porcelain enamels, both of direct-on and ground coated systems, is wide spread. It was known that many different parameters were used in this quick-type evaluation of porcelain enamel adherence. A questionnaire was circulated within the industry to determine the diversity of test parameters in use. A summary of the data reported in the 20 responses to this inquiry is given in Table 9.

If one were concerned with the standardization of this test method, a selection of test parameters might be based on frequency

of current use. This particular set of parameters might be expected to require the least amount of equipment modification for the greatest number of users. A different selection of test conditions might be made to achieve a degree of deformation severity which would lead to a optimum separation of adherence grades. The test conditions most frequently used in drop weight devices for adherence evaluation given in Table 9 are:

Indenter ball size - - - - - 1/2-inch diameter

Die size - - - - - 3/4-inch diameter

Impact energy - - - - - 70 to 80 inch-pounds

Further study is planned to determine the extent or degree of substrate strain most effective in distinguishing between grades of adherence quality, particularly for cover coats direct-to-steel.

W E A T H E R I N G S T U D I E S

INTRODUCTION

The Porcelain Enamel Institute has been co-sponsoring weathering studies with the National Bureau of Standards for the past thirty years. During this report period, the enamels in two of these weathering programs have completed their selected periods of exposure.

These two sets of enamels are: 1) The enamels exposed at Washington in the 1939 Exposure Test of Porcelain Enamels on Steel and 2) the enamels exposed at Gaithersburg in the 1966 Exposure Test of Nature Tone Enamels on Steel. The former completed 30 years' exposure, the latter 3 years' exposure.

Upon completion of the selected periods of exposure, the enamels were returned to the laboratory at NBS where they were cleaned by scouring 30 strokes with a sponge that had been sprinkled with calcium carbonate and moistened with a one percent solution of trisodium phosphate and rinsed successively with tap water, distilled water, and alcohol. Once the specimens had been cleaned they were observed visually for signs of rust or other degradation and then measured for changes in gloss and color.

RESULTS & DISCUSSION

1) 1939 Exposure Test of Porcelain Enamels on Steel.

Since it has been 15 years since any data pertaining to this test has been published, the initial data are presented in Table 10 together with a weather resistance rating based on the visual observation of these enamels. These data indicate that the enamels with good acid resistance, (AA and A), generally appeared to change very little after 30 years' exposure.

The color retention and percentage gloss retained data for these enamels are presented in Table 11 and shown graphically in Figures 6 & 7. Again, it can be seen that the enamels with good acid resistance have good weather resistance.

2) 1966 Exposure Test of Nature Tone Enamels on Steel.

The visual examination of the nature tone enamels on steel exposed at Gaithersburg revealed that all the enamels had good color and gloss retention, but some of the enamels showed signs of rust around pinhole-type defects as indicated in Table 12. This tendency to rust was not markedly reduced by testing for continuity of coating before exposure. However, extremely low test overvoltages were used. Had higher overvoltages been selected, these differences would probably be more pronounced.

The color retention and percentage gloss retained data are given in Table 13 and Figure 8. These data indicate that the nature tone enamels have excellent color and gloss retention after three years' exposure.

PLANS FOR NEXT REPORT PERIOD

During the next report period, it is planned to prepare comprehensive reports for publication in the Building Science Series on the enamels included in these two exposure tests.

Table 1. TENSILE TESTS OF A SINGLE PORCELAIN ENAMEL ON
SUBSTRATES OF VARIOUS THICKNESSES

NORMAL BUTTON SIZE (Area = 0.482 sq. in.)

Specimens Coated Both Sides					Specimens Coated One Side				
<u>Metal Gage</u>	<u>Failure in:</u>				<u>Metal Gage</u>	<u>Failure in:</u>			
	<u>n</u>	<u>Enamel</u>	<u>n</u>	<u>Epoxy</u>		<u>n</u>	<u>Enamel</u>	<u>n</u>	<u>Epoxy</u>
		psi		psi			psi		psi
20	12	560	8	400	20	6	750	0	---
18	20	700	0	---	18	12	970	0	---
14	16	1250	4	1310	14	10	1300	0	---

0.375 in. DIAM BUTTON SIZE (Area = 0.114 sq. in.)

Specimens Coated Both Sides					Specimens Coated One Side				
<u>Metal Gage</u>	<u>Failure in:</u>				<u>Metal Gage</u>	<u>Failure in:</u>			
	<u>n</u>	<u>Enamel</u>	<u>n</u>	<u>Epoxy</u>		<u>n</u>	<u>Enamel</u>	<u>n</u>	<u>Epoxy</u>
		psi		psi			psi		psi
20	9	1570	4	1350	20	4	1585	2	1210
18	5	1710	7	1990	18	3	1640	13	1645
14	0	---	8	1850	14	2	2210	8	1870

Table 2. TENSILE TESTS OF DIRECT-ON PORCELAIN ENAMELS WITH AN ADHESION TESTER, EMPLOYING VARIOUS ARRANGEMENTS TO INFLUENCE SPECIMEN RIGIDITY.

Regular Buttons on Completely Coated Specimen 1/				Regular Buttons on Isolated Discs With Bare Surround 2/				Regular Buttons on Completely Coated Specimen With 20 Gage Back-up Plate 3/			
Metal Gage	n	Mean Failure Stress	psi	Metal Gage	n	Mean Failure Stress	psi	Metal Gage	n	Mean Failure Stress	psi
20	6	750		20	20	430		20	4	1640	
18	12	970		18	12	700					
14	10	1300		14	12	780					

- 1/ The buttons were attached to specimens coated on one side only.
- 2/ The disc area was equal to the contact area of the buttons. The area surrounding the discs was not enamelled.
- 3/ An enamelled 20 gage back-up plate was firmly attached to the back side of the 20 gage test specimen with epoxy.

Table 3. SUMMARY OF TENSILE TEST RESULTS OBTAINED WITH ADHESION TESTER ILLUSTRATING THE EFFECT OF SUBSTRATE RIGIDITY.

<u>Test Variable</u>	<u>Grade 4</u>	<u>Grade X</u>
	<u>psi</u>	<u>psi</u>
After removal of enamel surrounding button by sandblasting.	545	---
Isolated enamel discs removed from 20 gage	---	430
Continuously coated specimens: 20 gage	680	750
" 18 gage	---	970
" 14 gage	---	1300
20 gage specimen with 20 gage back-up plate	---	1640
Battelle-type tensile test; specimen area 1.56 square inches. <u>1/</u>	> 4840	---

1/ Nature of Adherence of Porcelain Enamels to Metal, King, Tripp and Duckworth, Jour. Am. Ceram. Soc., 42, 504 (1959)

Table 4. The Effect of Various Parameters on the Meter Count Response Following Deformation of Direct-On Cover Coats in a Modified PEI Hydraulic Press

Indenter Ball Size - Die Configuration
Loading Pressure
Water Immersion

		Measured Meter Counts							
		<u>AN "Good"</u>				<u>DN "Poor"</u>			
Indenter Size	Loading Pressure pounds	<u>Dry*</u>		<u>Wet*</u>		<u>Dry*</u>		<u>Wet*</u>	
		A	B**	A	B**	A	B**	A	B**
1/2"	500	25	32	35	20	72	73	71	77
	1000	35	40	46	35	76	74	70	83
	1500	31	44	46	35	74	74	72	77
	2000	35	45	44	33	68	77	73	73
3/4"	500	3	0	4	10	86	90	99	87
	1000	11	7	15	30	111	84	117	103
	1500	6	4	24	25	96	94	123	100
	2000	7	5	13	28	99	87	112	97
1"	500	0	0	0	0	52	57	50	83
	1000	0	0	0	0	82	53	81	110
	1500	0	0	0	0	64	51	81	119
	2000	0	0	0	0	47	44	88	141

* "Dry" specimens were measured directly after deformation; "Wet" specimens were immersed in water, for one minute, after deformation and oven dried before measurement.

** A and B were duplicate specimens.

Table 5 Summary of the Effects of Various Parameters
in Terms of Average Meter Counts

<u>Parameters</u>	AN "Good"	DN "Poor"
Wet	19	91
Dry	14	74
500	11	75
1000	18	87
1500	18	85
2000	18	84
1/2"	36 (45%)*	74 (92%)*
3/4"	12 (9%)	99 (75%)
1"	0 (0%)	75 (38%)
A	16	82
B	16	84

Conclusions from statistical
analyses by "t" tests.

	<u>AN</u>	<u>DN</u>
Is the wet treatment more severe than the dry?	No	Yes
Does the 500 pound load differ from the others?	No	No
Do the results obtained with different ball sizes differ?	Yes, all sizes differ	Yes, all sizes differ.
Do the duplicate specimens give statistically different results?	No	No

* The three ball sizes result in widely different deformed areas. The percentages shown were obtained by normalizing the measured number of counts on the nominal deformed area (or its equivalent number of counts), to obtain a valid comparison. The above conclusion regarding ball sizes is based on the percentages so obtained.

T.ble 6.All DOAAC Adherence Grades Deformed in a Modified P.H. Hydraulic Press

Meter Counts per Indentation

Ball Size	Indentation:		Measured Dry						Measured after Water Immersion & Oven Drying								
	Area Sq. In.	No of Counts*	Grades:					Grades:									
			6	1	2	3	4	5	Mean	6	1	2	3	4	5	Mean	
1/2"	0.601	80															
		mean	79	79	66	42	37	30	56	77	73	54	63	39	36	57	
		s	4	3	6	6	2	7	5	4	4	4	9	6	7	6	
		V, %	5	4	9	13	7	24	10	6	5	7	14	17	20	12	
3/4"	0.994	132															
		mean	84	112	28	2.6	0.5	2.3	38	132	118	37	43	14	0.4	57	
		s	28	5	26	4	0.6	1.6	11	2	10	9	34	3	0.4	10	
		V, %	33	4	91	145	115	68	76	2	9	25	79	22	106	40	
1"	1.485	197															
		mean	27	101	0.25	0	0	0	21	157	49	0	0	0	0	34	
		s	32	27	0.5	0	0	0	10	3	39	0	0	0	0	7	
		V, %	117	26	200	0	0	0	57	2	80	0	0	0	0	14	

* The number of counts equivalent to the deformed area

Table 7. All DDMC Adherence Grades Deformed in a Modified PSI Hydraulic Press
The Percentage of the Deformed Area Found to be Bare Metal

Ball Size	Indentation:		Measured dry					Measured after Water Immersion & Oven Drying							
	Area. Sq. In	No. of Counts	1	2	3	4	5	Mean	6	1	2	3	4	5	Mean
1/2"	0.601	80*													
		mean	99	83	52	46	38		96	92	68	78	48	44	
		s.d.	4	6	6	3	9		6	5	5	12	8	9	
		V, %	4	8	12	6	24	10	7	5	7	15	17	20	12
3/4"	0.994	132*													
		mean	84	22	2	0	2		100	89	28	32	10	00	
		s.d.	1	20	3	1	1		17	8	7	26	2	0.5	
		V, %	1	91	141	110	72	75	17	9	24	80	20	207	59
1"	1.485	197*													
		mean	52	0	0	0	0		80	25	0	0	0	0	
		s.d.	14	0.5	0	0	0		1	20	0	0	0	0	
		V, %	26	207				58	2	80					14

* The number of counts equivalent to the deformed area

Table 8. The Effect of Water Immersion Following Deformation of a Direct-On Cover Coat in a Hydraulic Press Equipped With a 3/4" Ball Indenter

	<u>Measured Directly after Deformation</u>	<u>After water Immersion Towel Dried</u>	<u>After water Immersion Oven Dried</u>
n*	12	20	20
Range of Values, Counts	94-116	93-122	97-122
Mean Value, Counts	104	110	110
Coef. of Variation **	7%	7%	4%

* Number of indentations measured

** Coefficient of variation is the standard deviation expressed as a percentage of the mean value.

Table 9 An Analysis of Responses to Drop Weight Questionnaire.

<u>Indenter Sizes</u> <u>Used</u>	<u>Number</u> <u>Reported</u>	<u>Die Sizes</u> <u>Used</u>	<u>Number</u> <u>Reported</u>	<u>Impact</u> <u>Energies</u>	<u>Number</u> <u>Reported</u>
Inch		Inch		Inch- pounds	
1/2	8 **	1/2	1	20 to 40	1
9/16	3	5/8	3	41 to 60	2
5/8	2	41/64	1	61 to 80	7 **
3/4	2	3/4	5 **	81 to 100	4
1	2	7/8	1	101 to 120	1
		1	2	121 to 140	0
		1 1/16	1	141 to 160	0
		1 1/4	1	161 to 180	1

Of the eight using the $\frac{1}{2}$ inch ball
the following die sizes were used:

$\frac{1}{2}$	1
5/8	1
3/4	3 **
7/8	1

Of the 7 using between
61 and 80 inch-pounds

66	1
70	3 **
74	1
80	2

PEI Meter: Seven out of 17 used the meter.

Visual Rating: Twelve out of 17 used a visual rating scale.

Those most commonly used were

<u>Scale</u>	<u>Number Reported</u>
1 to 3	3
1 to 4	1
1 to 5	8 **

** Used with the greatest frequency.

Table 10. Summary of Initial Data and Weather Resistance Ratings for Enamels in 1939 Exposure Test of Porcelain Enamels on Steel.

Enamel	Fabricator of Specimens	Frit Supplier	Average Initial Specular Gloss	Acid Re- sistance (PEI Test)	Visual Weather- ing Rating ^{a/}
A-1,2	a	1	57.0	AA	A
A-11,12	b	1	57.4	AA	A
A-21,22	a	2	60.4	AA	B
A-31,32	b	2	60.1	AA	B
A-41,42	a	3	58.5	AA	B
A-51,52	b	3	58.1	AA	B
A-61,62	a	4	62.0	AA	C
A-71,72	b	4	60.1	AA	C
B-1,2	b	1	52.6	C	C
B-21,22	b	2	54.1	C	C
B-41,42	b	3	51.6	D	D
B-61,62	b	4	53.2	C	C
C-11,12	c	1	40.6	C	C
C-31,32	c	2	52.4	A	C
C-51,52	c	3	51.6	A	B
C-71,72	c	4	53.2	A	C
D-1,2	c	1	----	-	D
D-21,22	c	2	56.9	D	C
D-41,42	c	3	55.1	D	D
D-61,62	c	4	----	-	D
E-11,12	d	1	50.9	B	B-
E-31,32	d	2	54.3	AA	B-
E-51,52	d	3	54.4	AA	A
E-71,72	d	4	53.5	AA	A
F-1,2	d	1	51.5	D	D
F-11,12	e	1	48.7	D	D
F-21,22	d	2	50.2	C	D
F-31,32	e	2	55.6	D	D
F-41,42	d	3	43.1	D	D
F-51,52	e	3	56.6	D	D
F-61,62	d	4	46.4	D	D
F-71,72	e	4	52.6	D	D
H-1,2	e	1	39.6	A	B
H-11,12	f	1	48.1	A	C
H-21,22	e	2	56.5	A	B
H-31,32	f	2	54.5	A	B

Table 1c (Cont.)

Enamel	Fabricator of Specimens	Frit Supplier	Average Initial Specular Gloss	Acid Re- sistance (PEI Test)	Visual Weathering Rating ^a
H-41,42	e	3	47.4	AA	A
H-51,52	f	3	48.5	AA	A
H-61,62	e	4	55.1	AA	A
H-71,72	f	4	55.4	AA	A
K-1,2	f	1	----	----	D
K-11,12	g	1	----	--	D
K-21,22	f	2	54.1	D	C
K-31,32	g	2	53.7	D	C
K-41,42	f	3	52.0	D	C
K-51,52	g	3	53.5	D	D
K-61,62	f	4	----	-	D
K-71,72	g	4	----	-	C
L-1,2	g	1	55.8	AA	A
L-11,12	h	1	56.1	AA	A
L-21,22	g	2	54.2	B	A
L-31,32	h	2	56.4	A	A
L-41,42	g	3	55.8	AA	B
L-51,52	h	3	52.3	A	B-
L-61,62	g	4	53.0	A	B
L-71,72	h	4	41.8	AA	A
N-1,2	h	1	51.7	C	C
N-11,12	k	1	50.5	C	C
N-21,22	h	2	51.2	D	D
N-31,32	k	2	45.5	D	C
N-41,42	h	3	51.7	C	C
N-51,52	k	3	46.8	C	D
N-61,62	h	4	47.1	D	D
N-71,72	k	4	54.4	D	D
P-1,2	k	1	55.4	AA	B
P-11,12	l	1	----	--	D
P-21,22	k	2	----	--	-
P-31,32	l	2	39.9	C	D
P-41,42	k	3	49.5	A	A
P-51,52	l	3	33.4	A	D
P-61,62	k	4	49.7	AA	A
P-71,72	l	4	55.4	AA	A

Table 10 (Cont.)

Enamel	Fabricator of Specimens	Frit Supplier	Average Initial Specular Gloss	Acid Re- sistance (PEI Test)	Visual Weathering Rating ^{a/}
S-1,2	l	1	-----	-	
S-11,12	m	1	-----	-	D
S-21,22	l	2	45.1	B	C
S-31,32	m	2	45.1	C	B
S-41,42	l	3	35.0	A	B-
S-51,52	m	3	39.5	A	A
S-61,62	l	4	45.0	C	D
S-71,72	m	4	35.7	C	D
T-1,2	m	1	73.0	AA	B
T-11,12	n	1	66.0	AA	A
T-21,22	m	2	53.8	AA	A
T-31,32	n	2	55.5	AA	B-
T-41,42	m	3	60.9	AA	B
T-51,52	n	3	59.0	A	B
T-61,62	m	4	65.5	AA	B
T-71,72	n	4	65.6	AA	B
V-1,2	n	1	55.2	C	D
V-11,12	a	1	56.2	C	D
V-21,22	n	2	57.6	C	D
V-31,32	a	2	56.0	C	C
V-41,42	n	3	53.0	C	D
V-51,52	a	3	54.9	C	D
V-61,62	n	4	46.7	C	D
V-71,72	a	4	54.5	C	D

^{a/} A rating means no change readily apparent

B rating means beginning to change, but not objectionable

C rating means moderate changes have taken place, somewhat objectionable

D rating means severe changes have taken place, very objectionable.

Table 11 Summary of Gloss Retention and Color Change Data for Enamels in 1939 Exposure Test of Porcelain Enamels on Steel

<u>Enamel</u>	<u>Percentage Gloss Retained after:</u>				<u>Color Change after:</u>	
	<u>30 yrs</u>	<u>15 yrs</u>	<u>7 yrs</u>	<u>1 yr</u>	<u>30 yrs</u>	<u>15 yrs</u>
White, Glossy, Acid-Resistant Enamel						
A-1,2	71.8	83.1	81.4	90.8	0.8	0.7
A-11,12	71.1	86.9	83.2	95.4	0.9	1.0
A-21,22	70.3	84.2	79.8	89.7	0.8	0.6
A-31,32	67.1	76.6	74.8	90.0	0.8	0.5
A-41,42	72.5	85.0	84.1	92.1	1.8	3.0
A-51,52	69.3	85.4	77.5	90.2	1.5	1.2
A-61,62	46.2	83.3	74.6	96.8	3.8	1.1
A-71,72	52.0	73.4	70.5	91.0	3.1	1.7
Average	65.0	82.2	78.2	92.0	1.7	1.2
White, Glossy, Nonacid-Resistant Enamel						
B-1,2	12.4	18.0	28.9	69.8	2.10	2.9
B-21,22	11.5	15.0	34.8	69.2	-	4.8
B-41,42	12.5	15.0	26.5	71.8	1.0	2.7
B-61,62	11.3	16.4	34.8	71.1	0.9	3.1
Average	11.9	16.1	31.2	70.5	1.4	3.4
White, Semimat, Acid-Resistant Enamel						
C-11,12	18.7	30.3	51.8	84.0	0.9	1.5
C-31,32	25.4	42.1	61.5	85.8	1.9	2.5
C-51,52	58.8	76.7	76.8	91.5	2.4	2.2
C-71,72	42.0	54.8	73.1	95.7	1.9	1.3
Average	36.2	51.0	65.8	91.0	1.7	1.8
White, Semimat, Nonacid-Resistant Enamel						
D-1,2	-	-	-	-	3.0	1.0
D-21,22	10.9	15.0	43.2	70.5	3.2	3.3
D-41,42	10.9	15.0	38.0	74.0	2.4	3.3
D-61,62	-	-	-	-	5.2	3.6
Average	10.9	15.0	40.6	72.2	3.5	3.4

Table 11 (Cont.)

Enamel	Percentage Gloss Retained After:				Color Change After:	
	30 yrs	15 yrs	7 yrs	1 yr	30 yrs	15 yrs
Buff, Glossy, Acid-Resistant Enamel						
E-11,12	52.0	60.2	68.2	83.8	1.9	1.4
E-31,32	38.5	54.8	64.2	90.8	1.5	1.8
E-51,52	72.8	90.0	74.0	91.6	1.0	0.4
E-71,72	63.2	76.0	77.7	96.1	1.5	1.6
Average	56.6	70.2	71.0	92.8	1.5	1.3
Buff, Glossy, Nonacid Resistant Enamel						
F-1,2	18.7	24.3	43.0	72.1	13.0	7.8
F-11,12	23.9	35.6	61.2	90.2	2.8	4.4
F-21,22	24.5	33.2	52.9	72.1	5.7	3.8
F-31,32	32.8	44.3	48.8	69.0	5.3	2.9
F-41,42	20.0	26.2	37.3	71.3	21.0	13.4
F-51,52	16.3	27.6	33.7	74.1	8.3	5.3
F-61,62	13.7	24.4	22.5	61.2	15.6	12.0
F-71,72	13.2	23.7	28.0	62.6	16.0	12.1
Average	20.4	29.9	40.9	71.6	11.0	7.7
Buff, Semimat, Acid-Resistant Enamel						
H-1,2	59.2	64.3	60.7	81.7	3.0	2.0
H-11,12	51.6	52.1	57.6	71.3	1.9	0.9
H-21,22	62.1	77.0	70.7	88.8	0.7	0.8
H-31,32	62.7	74.3	71.2	87.8	0.6	0.5
H-41,42	68.6	87.5	75.7	93.0	0.8	0.6
H-51,52	63.2	76.8	72.5	95.8	1.3	1.2
H-61,62	60.3	83.9	74.4	93.2	1.8	4.3
H-71,72	63.2	83.5	76.4	92.2	4.2	2.0
Average	61.4	74.9	69.9	91.8	1.8	1.5
Buff, Semimat, Nonacid-Resistant Enamel						
K-1,2	-	-	-	-	9.2	7.4
K-11,12	-	-	-	-	11.5	7.2
K-21,22	14.7	21.3	49.0	70.7	5.7	4.1
K-31,32	13.7	18.8	36.4	65.9	9.5	7.2
K-41,42	47.2	63.8	63.5	79.6	2.8	1.7
K-51,52	11.0	16.2	36.5	69.9	15.2	9.2
K-61,62	-	-	-	-	19.7	6.9
K-71,72	-	-	-	-	7.4	14.4
Average	21.7	30.0	46.4	71.5	10.1	7.3

Table 11 (Cont.)

<u>Enamel</u>	<u>Percentage Gloss Retained After:</u>				<u>Color Change After:</u>	
	<u>30 yrs</u>	<u>15 yrs</u>	<u>7 yrs</u>	<u>1 yr</u>	<u>30 yrs</u>	<u>15 yrs</u>

Black, Glossy, Acid-Resistant Enamel

T-1,2	39.4	57.9	56.1	72.6	1.1	0.8
T-11,12	56.8	73.4	64.2	93.7	1.1	0.4
T-21,22	66.7	87.8	80.6	92.0	0.6	1.4
T-31,32	66.1	79.1	70.0	93.0	1.0	0.8
T-41,42	52.3	64.2	70.7	91.0	2.0	0.0
T-51,52	64.9	85.4	68.9	92.3	1.5	1.5
T-61,62	45.9	66.7	59.8	86.8	1.6	1.1
T-71,72	48.7	57.6	54.9	78.5	1.5	0.8
Average	55.1	71.5	65.6	87.5	1.3	0.8

Black, Glossy, Nonacid-Resistant Enamel

V-1,2	58.9	61.5	63.6	76.0	26.4	19.9
V-11,12	35.0	34.5	57.0	74.0	49.1	41.3
V-21,22	56.2	64.0	58.8	68.8	2.1	1.4
V-31,32	36.3	41.1	40.1	65.8	5.1	4.0
V-41,42	56.5	66.3	59.7	75.5	8.2	4.6
V-51,52	53.5	58.2	58.0	71.9	12.0	12.3
V-61,62	44.4	41.1	75.9	83.2	18.1	13.4
V-71,72	61.6	61.7	73.9	78.6	10.2	6.4
Average	50.3	55.2	60.9	74.2	16.4	12.9

Table 11 (cont.)

Enamel	Percentage Gloss Retained After:				Color Change After	
	30 yrs	15 yrs	7 yrs	1 yr	30 yrs	15 yrs
Red, Glossy, Acid-Resistant Enamel						
L-1,2	65.1	85.5	76.4	85.6	6.4	3.8
L-11,12	69.2	80.4	74.7	83.7	1.2	0.8
L-21,22	66.4	82.1	78.1	85.4	3.9	2.2
L-31,32	68.7	82.0	79.7	86.4	2.6	3.1
L-41,42	63.3	77.0	75.7	85.1	1.6	3.0
L-51,52	59.8	68.5	67.6	86.9	8.7	0.9
L-61,62	66.4	80.8	73.2	88.0	3.4	2.3
L-71,72	77.4	91.9	74.0	87.8	0.7	1.3
Average	67.0	81.0	74.9	86.2	3.5	2.2
Red, Glossy, Nonacid-Resistant Enamel						
N-1,2	55.0	65.8	70.0	76.3	3.9	2.5
N-11,12	71.1	86.9	83.2	95.4	2.0	2.1
N-21,22	70.3	84.2	79.8	89.7	4.7	1.9
N-31,32	67.1	76.6	74.8	90.0	2.1	2.7
N-41,42	72.5	85.0	84.1	82.1	4.6	3.3
N-51,52	69.3	85.4	77.5	90.2	3.7	2.7
N-61,62	46.2	83.3	74.6	96.8	15.4	4.3
N-71,72	52.0	73.4	70.5	91.0	23.8	22.0
Average	65.0	82.2	78.2	92.0	7.5	5.2
Red, Semimat, Acid-Resistant Enamel						
P-1,2	66.3	86.3	70.3	86.6	4.4	3.8
P-11,12	-	-	-	-	22.2	24.7
P-21,22	-	-	-	-	-	38.0
P-31,32	40.7	48.6	64.4	88.5	5.0	6.2
P-41,42	62.5	70.6	69.2	93.5	1.6	2.6
P-51,52	91.3	84.2	73.8	92.5	8.8	5.9
P-61,62	63.2	83.5	76.2	90.6	2.2	2.7
P-71,72	66.3	89.7	81.0	92.0	3.0	5.3
Average	65.1	77.1	71.1	91.0	6.8	11.1
Red, Semimat, Nonacid-Resistant Enamel						
S-1,2	--	-	-	-	31.8	24.3
S-11,12	-	-	-	-	29.4	15.6
S-21,22	63.2	76.9	65.8	78.2	16.6	2.9
S-31,32	59.0	63.8	57.6	70.2	7.2	6.7
S-41,42	55.8	61.9	58.0	80.4	3.2	2.6
S-51,52	84.8	89.4	84.1	93.9	1.5	1.0
S-61,62	26.4	38.8	35.2	64.7	18.7	14.3
S-71,72	41.2	50.0	70.3	93.4	24.9	18.6
Average	55.1	63.5	61.8	80.1	16.7	10.7

Table 13. Summary of Gloss and Color Data for Nature-Tone Enamels Exposed for Three Years

Enamel	Percentage Gloss Retained			Color Retention		
	Kure Beach	South Florida	Gaithersburg	Kure Beach	South Florida	Gaithersburg
101	78.1	89.7	76.0	98.5	99.3	98.5
102	73.9	87.8	71.1	98.5	99.2	98.5
103	72.5	89.8	74.3	98.9	99.3	99.3
104	64.2	90.2	73.1	99.0	99.6	99.0
105	79.9	89.2	74.6	98.9	99.4	99.1
106	64.0	80.1	64.7	96.5	98.5	97.4
107	78.0	90.3	73.8	99.2	99.3	99.1
108	92.5	91.2	86.8	99.5	99.2	99.5
109	97.2	87.1	90.2	99.6	99.3	99.7
110	101.2	86.5	88.6	99.7	99.7	99.6
111	82.9	91.0	80.9	98.2	99.1	99.1
112	94.3	95.0	89.6	99.4	99.6	99.5
113	93.9	103.1	92.4	99.7	99.3	99.4
114	76.2	91.5	76.3	99.0	99.7	99.2
115	86.5	97.6	85.2	99.3	99.4	99.6
116	79.8	93.3	78.1	98.4	99.5	99.4
117	74.3	84.1	72.7	97.9	99.4	98.6
118	72.4	85.3	80.3	99.2	98.8	99.4
119	70.5	85.1	67.8	99.0	99.6	99.9
120	74.8	90.5	72.5	98.7	99.6	99.0
1	74.8	83.8	71.2	98.9	99.7	99.2
3	70.1	84.3	69.2	98.8	98.6	99.4
4	71.6	92.6	79.8	97.9	99.3	98.7
6	158.6	177.7	192.2	99.4	99.6	99.6
7	139.9	149.8	142.4	99.8	99.4	99.5
Average	84.9	95.5	84.9	98.9	99.3	99.1

Table 12. Summary of Enamels with obviously Rusted Surfaces after Three Years' Exposure

Enamel	Specimens Rusted at:					
	Kure Beach		South Florida		Gaithersburg	
	Tested*	Untested*	Tested	Untested	Tested	Untested
101	1	1	0	0	1	1
102	1	1	0	0	0	1
103	3	3	0	0	1	1
104	0	1	0	0	0	0
105	0	0	0	1	0	0
106	0	1	0	0	0	0
107	0	0	0	0	0	0
108	1	0	0	0	0	0
109	0	0	0	0	0	0
110	0	0	0	0	0	1
111	0	0	0	0	0	0
112	0	0	0	0	0	0
113	0	0	0	0	0	0
114	0	0	0	0	0	0
115	0	0	0	0	0	0
116	0	0	0	0	0	0
117	0	0	0	0	0	0
118	0	1	1	0	0	1
119	0	0	0	0	0	0
120	0	0	0	0	0	0
1	1	1	0	0	1	1
3	0	0	0	0	0	0
4	0	0	0	0	0	0
6	0	1	1	0	1	1
7	0	0	2	1	1	1
Total	7	10	4	2	5	8

*The tested specimens were probed with the high voltage continuity of coating probe at overvoltages of 0.1 to 1.6 KV with no defects being located before the specimens were exposed. The untested specimens were not tested for continuity of coating before exposure.

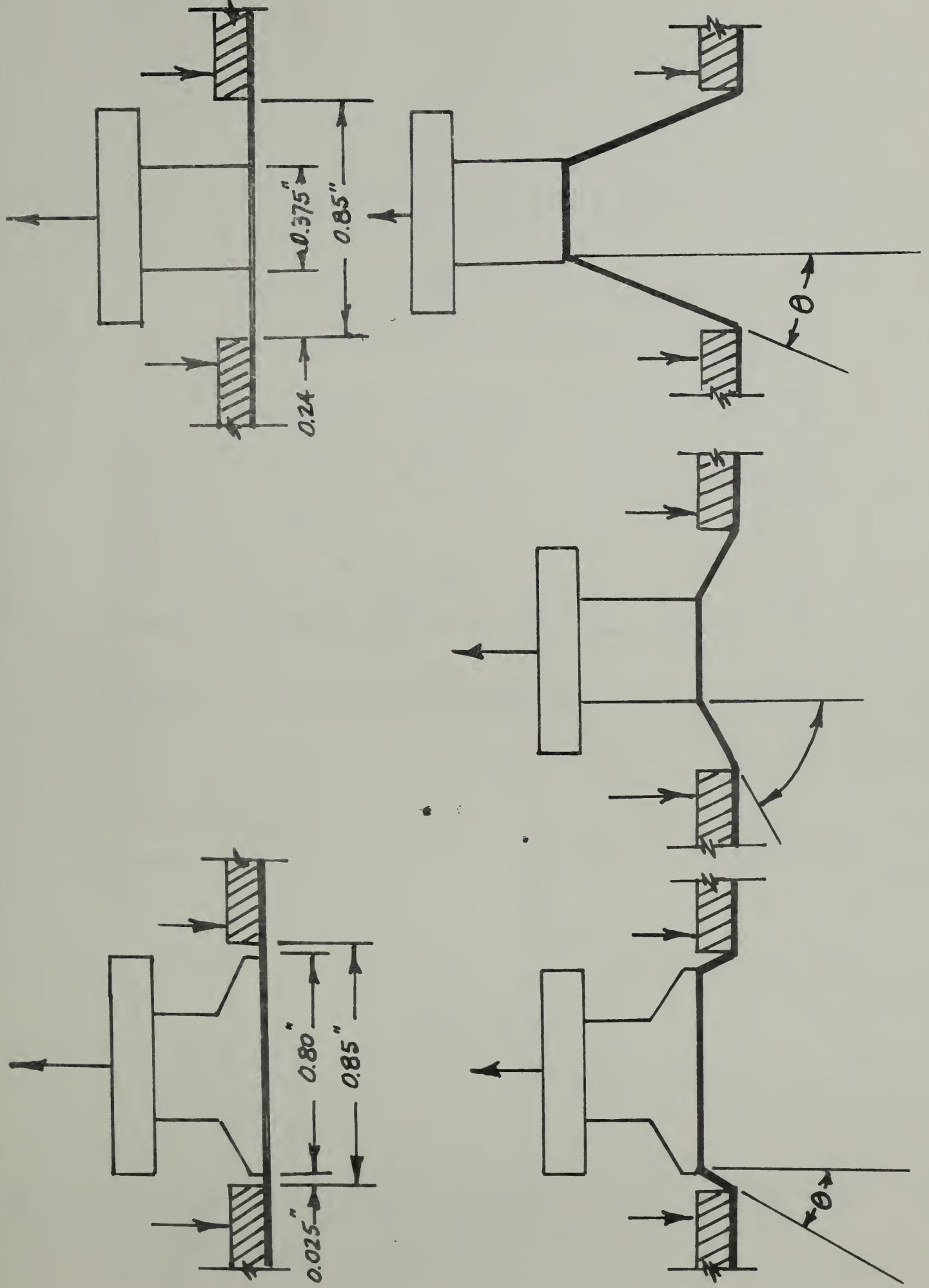


Figure 1. Substrate conditions which initiate and control button circumference.

REGULAR BUTTONS

3/8" DIAM. BUTTONS

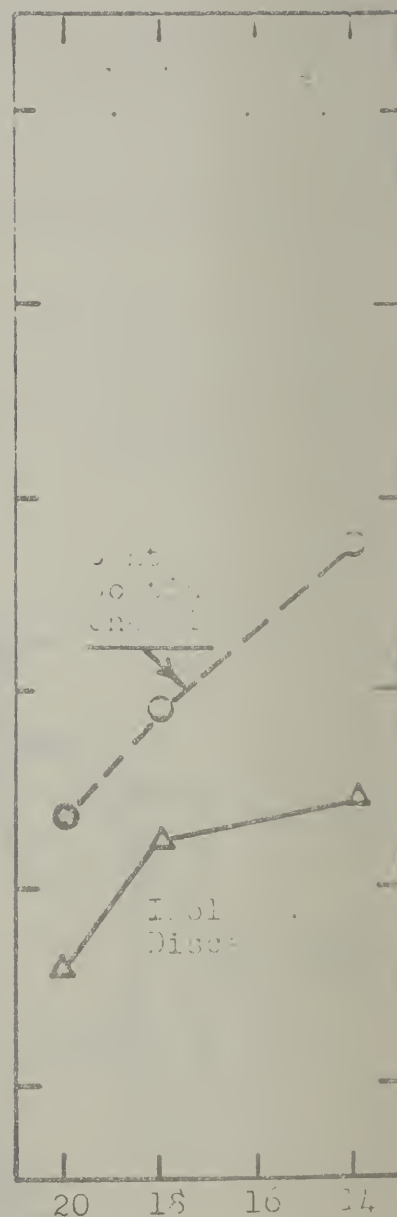
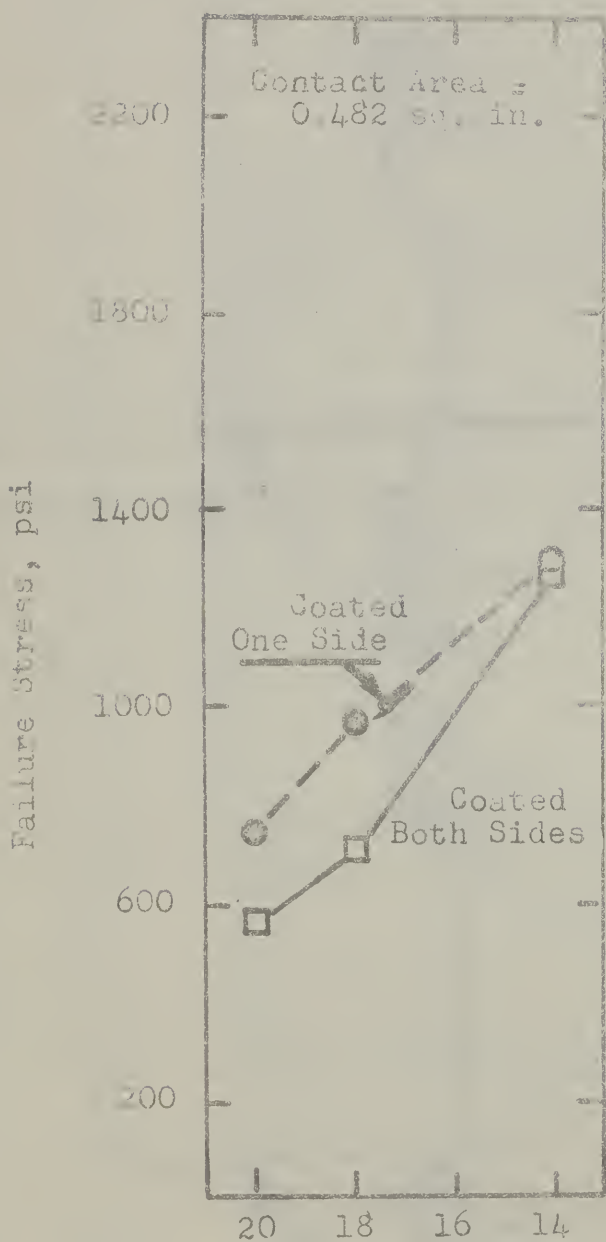
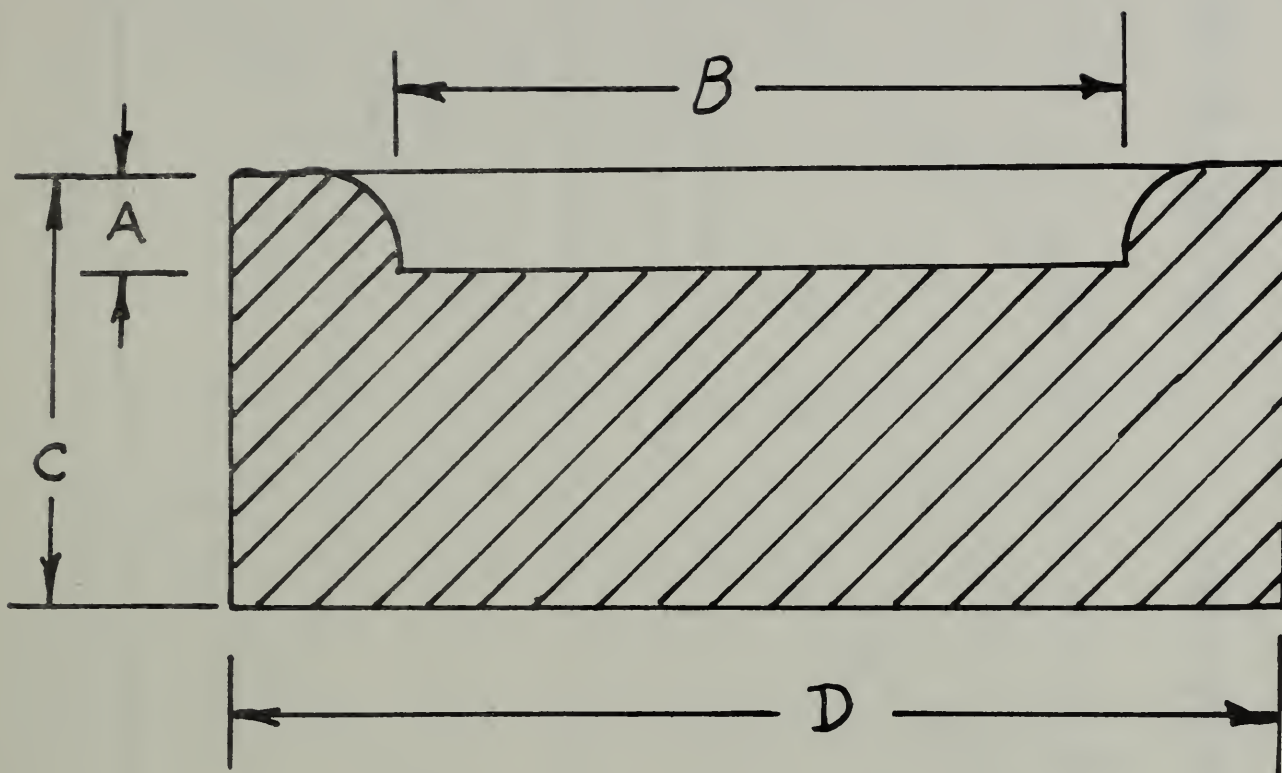


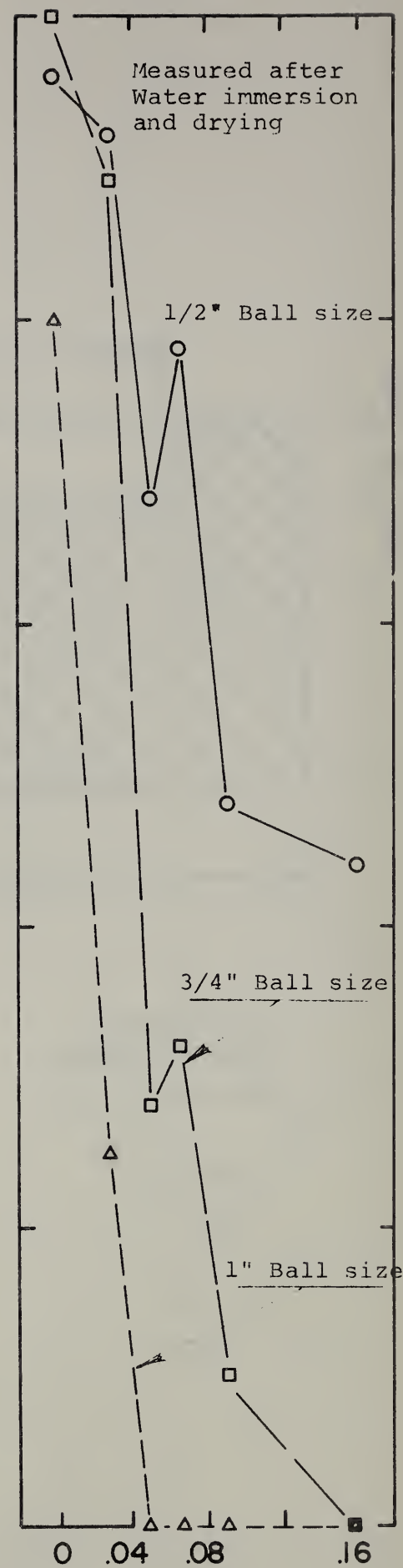
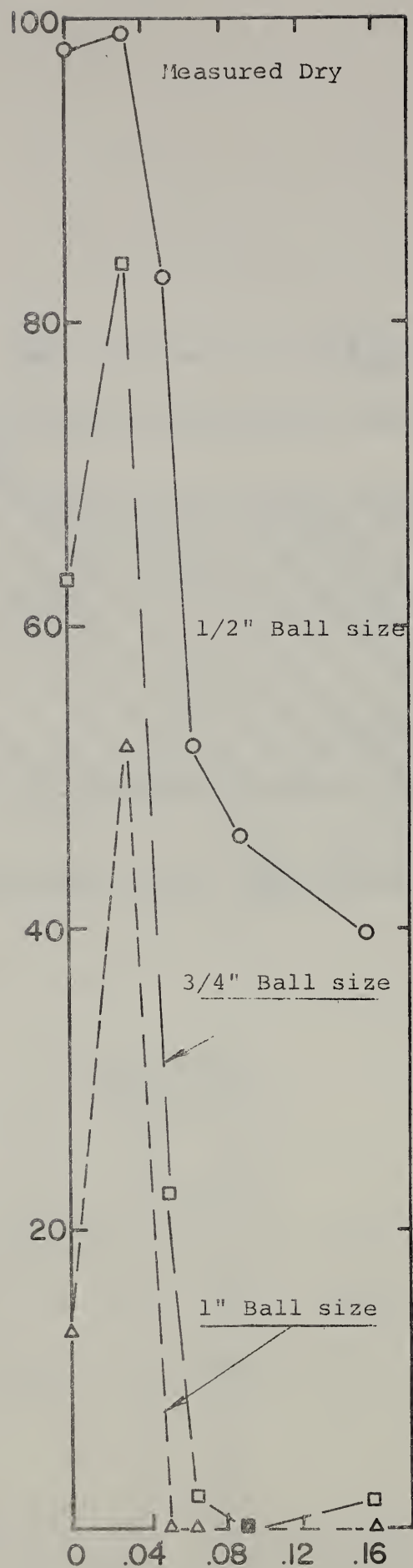
Figure 2. TENSILE STRESS AT FAILURE WITH ADHESION TESTER



Indenter Ball Diameter	Dimension			
	A	B	C	D
inches		inches		
1	0.156	1.125	0.5	1.625
3/4	0.156	0.875	0.5	1.625
1/2	0.156	0.625	0.5	1.625

Figure 3 Modified Deforming Dies for use with PEI Press

Relative Counts, percent, after Deformation in Hydraulic Press



Amount of Nickel Deposited, mg/sq. ft.

Figure 4. Relative Counts of Six DOAAC Adherence Grades as Function of the Amount of Deposited Nickel

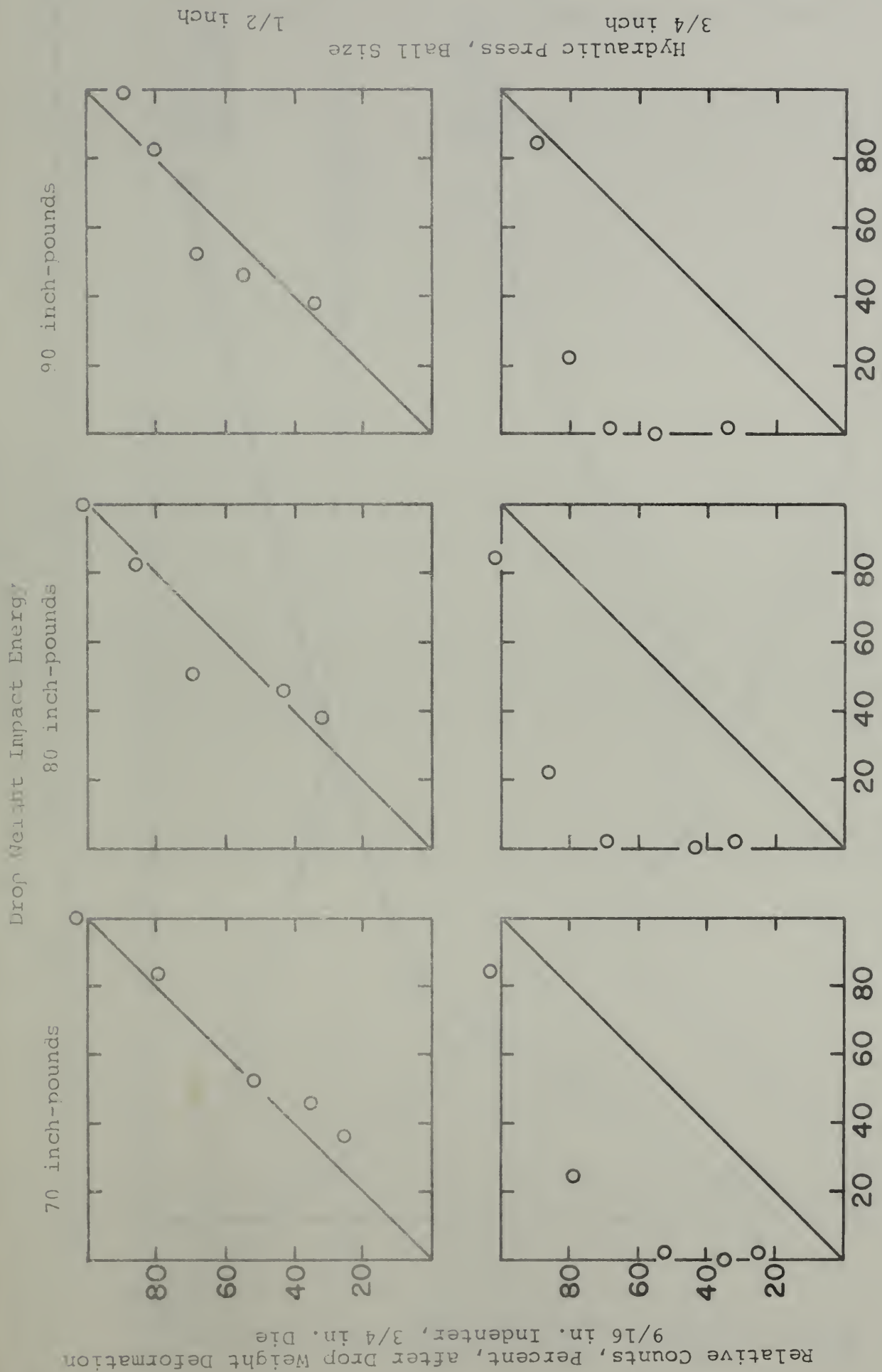


Figure 5 Correlation of the Severity of Deformations with a Hydraulic Press and with a Drop Weight Device

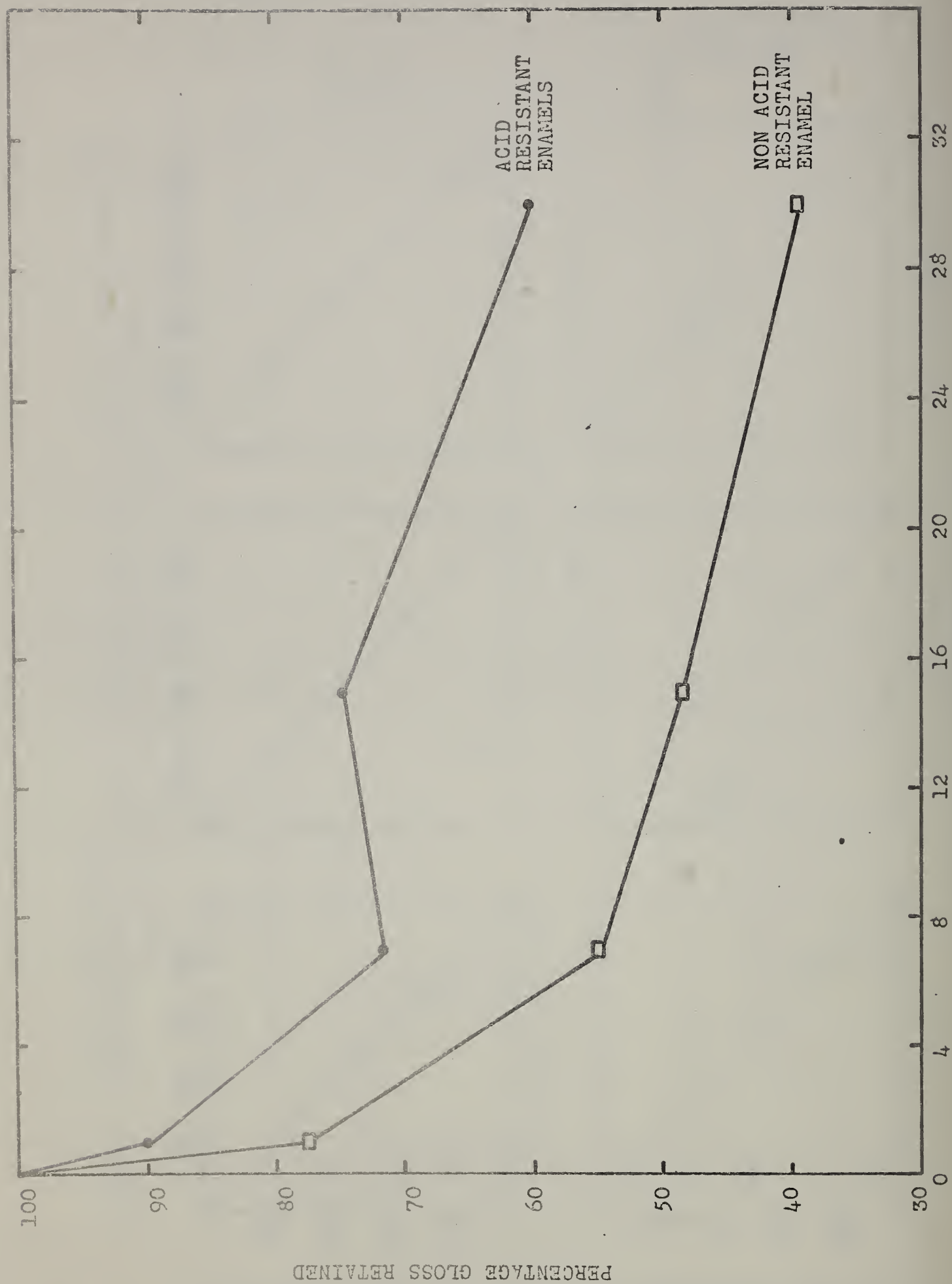


Figure 6. Effect of exposure time on the gloss retention of porcelain enamels on steel.

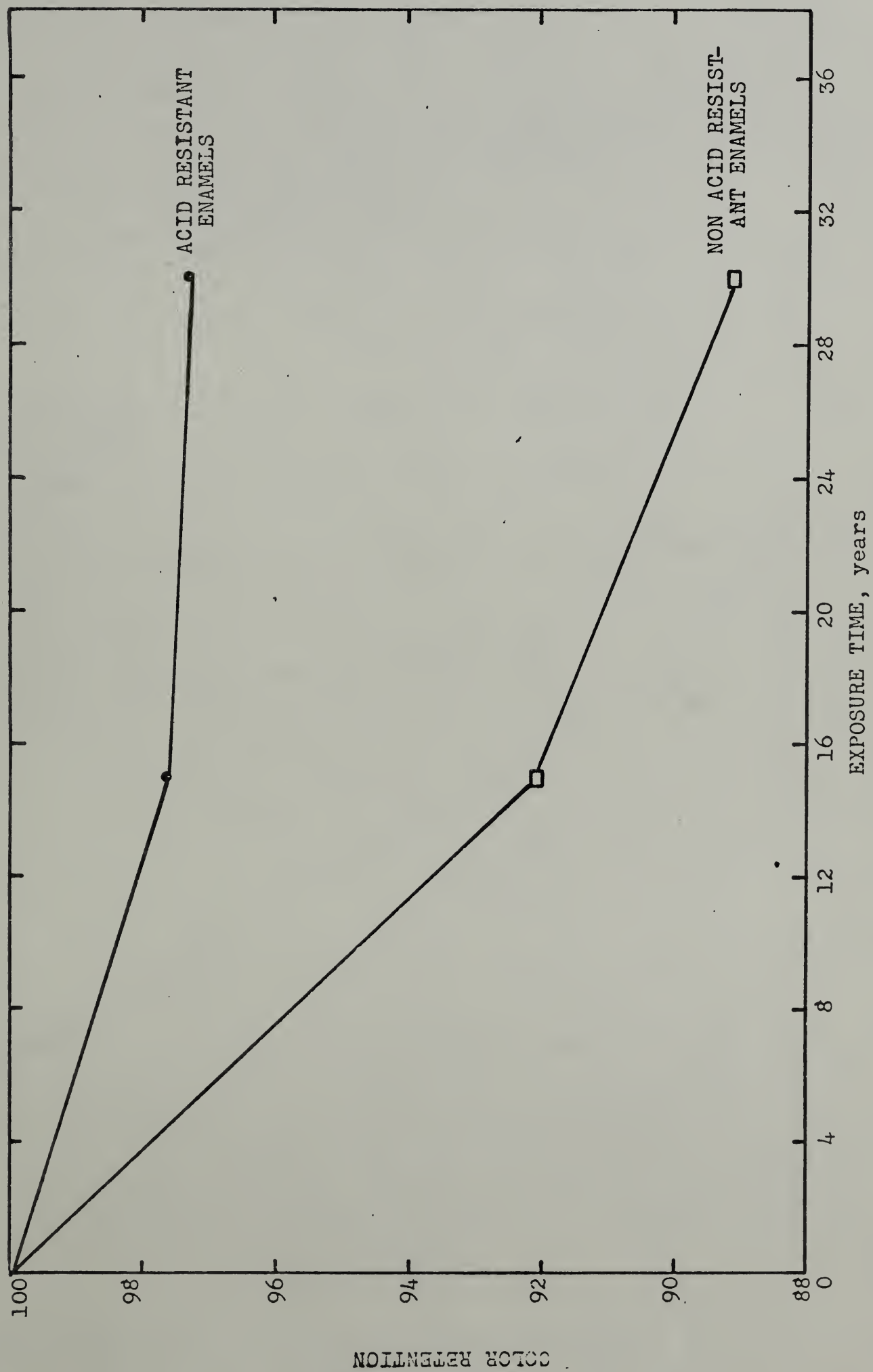


Figure 7. Effect of exposure time on the color retention of porcelain enamels on steel.

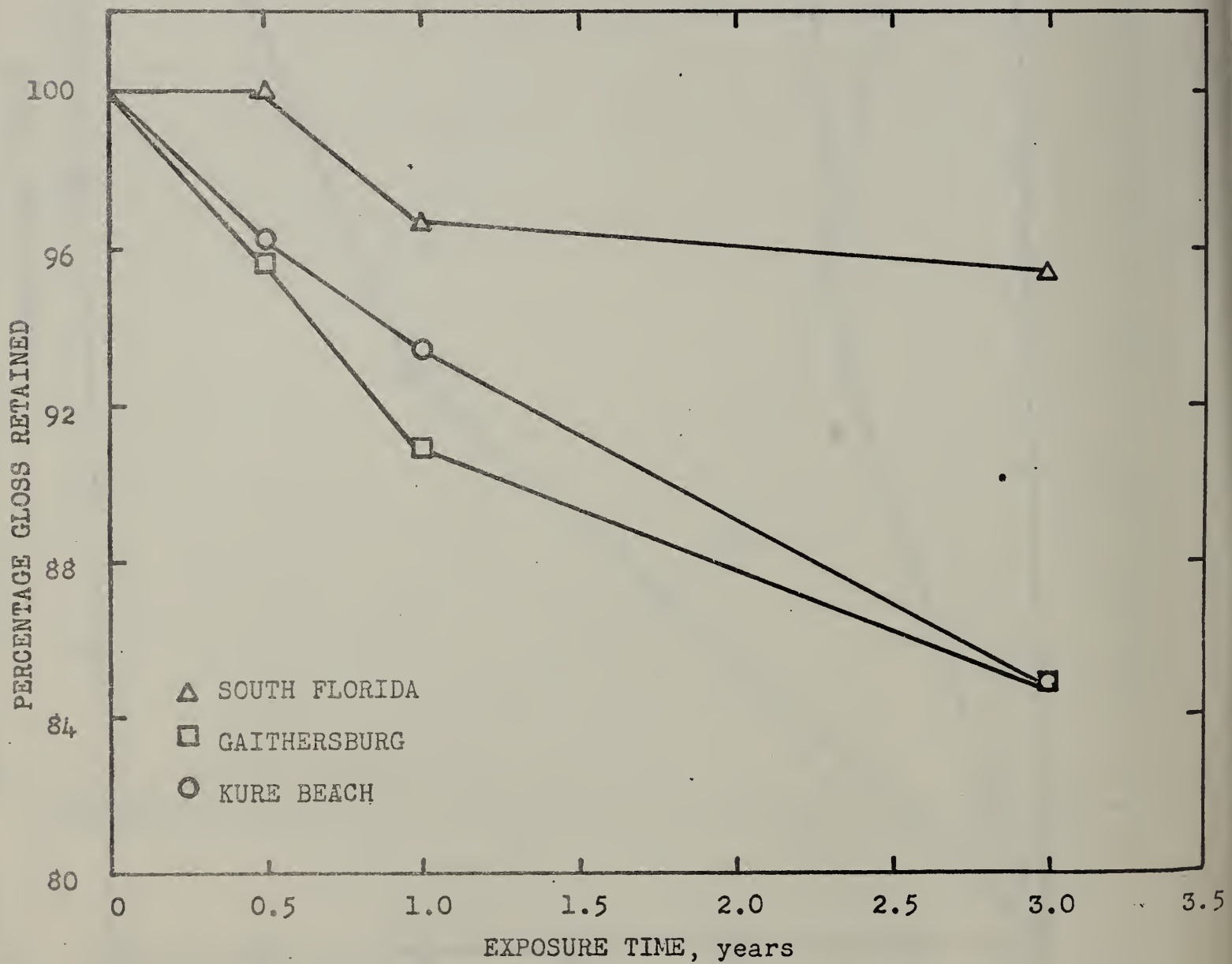
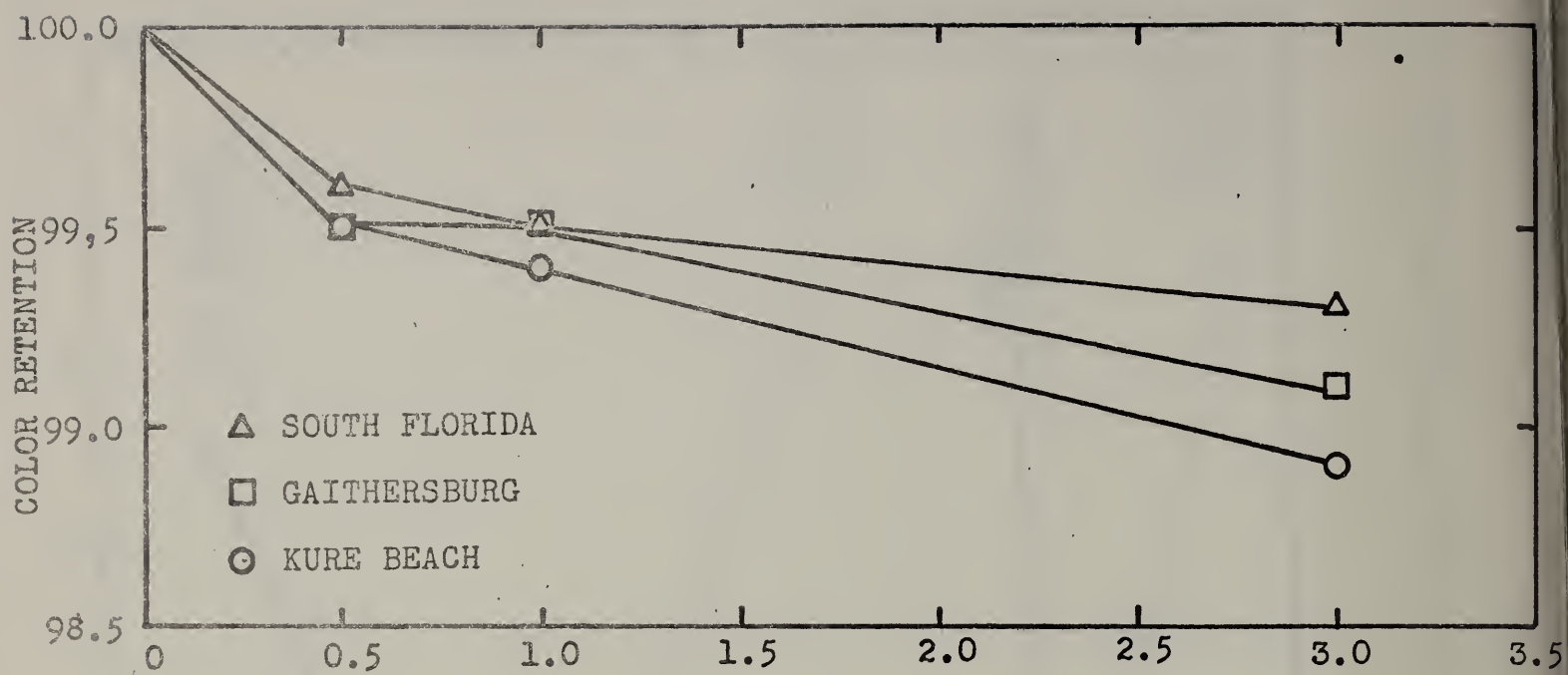


Figure 8. Effect of exposure time on gloss and color retention of nature-tone enamels on steel.

