NATIONAL BUREAU OF STANDARDS REPORT

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Progress Report

April 1 through June 31, 1970

DEVELOPMENT OF METHODS OF TEST FOR QUALITY CONTROL OF PORCELAIN ENAMELS

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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Progress Report

April 1 through June 30, 1970

DEVELOPMENT OF METHODS OF TEST FOR QUALITY CONTROL OF PORCELAIN ENAMELS

By

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U.S. DEPARTMENT OF COMMERCE

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INTRODUCTION

The general subject of our current work is to provide a technique to measure the quality of adherence of cover coat porcelain enamels applied direct-to-steel. The long-range goal is to develop a non-destructive method to estimate the quality of adherence in these systems. A more easily attainable goal is to find a test method to give a better estimate of enamel adherence than is obtained with the PEI adherence test even though it may involve specimen damage. Results obtained with one possible device to assist in direct-on adherence measurement, a commercially available adhesion tester, are described in this report.

A round-robin test on carefully prepared specimens covering a wide range of adherence values is described. The suggested modification of current practice is immediately available and is shown to result in more reliable and repeatable estimates of adherence in production than those obtained from trained observers.

RESULTS AND DISCUSSION

A. Preparation of a Series of Cover Coats Direct-to-Steel with a Wide Range of Adherence

Direct-on specimens previously used were prepared by the several frit companies using various decarburized steels, various pickling and cleaning procedures and porcelain enamels in white, coppertone and avocado. For a more careful and extensive study of direct-on adherence the Direct-on Adherence Advisory Committee (DOAAC) recommended that a new series of specimens be obtained. Companies represented on the advisory committee volunteered a single uniform steel supply, and the preparation of a series of specimens in which the adherence quality was controlled by various amounts of nickel deposition, with the same porcelain enamel on all specimens.

Preliminary tests with this new specimen series showed a good inverse correlation between bare metal exposed after drop weight deformation and the amount of nickel added before enamel application.

The widely accepted concept of adherence measurement is that when adherence to the metal is good, much enamel will be retained within a deformed area, but when poorer adherence is attained, little or no enamel will be retained after a similar deformation. Assuming that this is a valid criteria of adherence quality, it will be noted from Table 1 and Figure 1 that the new specimen series covered a wide range of quite uniform adherence grades.

B. Direct-on Adherence Round Robin Phase II

A series of tests was described in the previous report to determine industry agreement on adherence evaluation by visual rating of drop-weight deformed specimens.

The comparative tests described in the previous report were repeated with the controlled nickel deposit, DOAAC specimens.

Two specimens of each of the five DOAAC grades were deformed in a drop-weight device using a one-half inch ball indenter and a five pound weight dropped through a distance of 16 inches. Two indentations were made on each specimen. One was over a 3/4-inch bottomless die while the other was over a 5/8-inch die. Each observer made his visual rating, according to his laboratory practice, before sending the specimens to the next cooperating laboratory. The number of meter counts for each indentation was measured both before and after circulation among the industry observers. The specimens were identified by code letters so that the observers did not know the grade numbers.

Comparison of the results given in Table 2 with similar results described in the previous report, shows that a much improved agreement was obtained among the various observers in Phase II. The better agreement may be attributed to the observers rating the same test-pieces, rather than companion specimens, or to the use of only one deforming device, or to both of these. A plot of the visual ratings as a function of PEI meter counts is shown in Figure 2. The equations of the least squares lines through the points of Figure 2 are:

For the 3/4-inch die: $y = 1.625 + 0.056\ x$
For the 5/8-inch die: $y = 1.058 + 0.063\ x$

where y = the visual ratings of experienced industry observers, and
x = PEI Adherence Meter counts.

Figure 3 allows a comparison of the visual adherence ratings of the various observers with an instrumental rating based on an industry consensus. Perfect correlation lines are shown on these plots. Observer B appears to assign ratings that agree well with the industry average. The ratings assigned to Observer D appear to be more severe than the instrumental ratings. The bias of Observer A appears to be toward less severe ratings than either Observer D or the industry consensus.

C. The Instrumental Rating of Direct-on Adherence

Phase II of the Round Robin adherence test suggests the following instrumental method for the rating of cover coats direct-to-steel.

1) Specimen deformation - A drop-weight device using a) a one-half inch diameter hemispherical indentor (or tup), b) an 80 inch-pound impact energy (such as 5 pounds from 16 inches), and c) a 5/8-inch diameter bottomless die, should be used to deform the specimen with a single blow.

2) Estimation of the area of bare metal - The PEI Adherence Meter may be used to obtain the number of counts on the deformed specimen or part.

3) Enter Table with the number of counts (not the adherence index) and read directly the instrumental rating of a basis of 1 to 5 in which 1 represents the best and 5 the poorest adherence.

D. Tensile Tests with Adhesion Tester

1) One rather obvious and straightforward way to measure adherence of a cover coat to its substrate is by tensile tesing. Through the use of high strength adhesives tensile fixtures can be attached to the top surface of the steel sheet. The stress required to strip the enamel layer from the substrate should be a measure of the adherence quality. Experiments with a commercially available adhesion tester have been made in an attempt to evaluate the adherence of cover coats porcelain enamel direct-to-steel. The device used is illustrated in Figure 4. An aluminum alloy spool (or button), in the foreground of the horizontal specimen is attached to the glass layer by an epoxy adhesive. The jaw of the device, engaged under the top flange of the button, pulls vertically when the hand wheel at the top is turned and the stress required to cause failure is indicated on the vertical barrel scale. One type of failure is shown by the specimen on the left where failure occurred near the interface between the glass and the black oxide-rich layer.

2) A device similar to that shown in Figure 4, but with a 2000 psi capacity, was adapted to operate with a constant speed motor so that load could be applied continuously and at a uniform rate. The stress scale marked at 250 psi intervals was difficult to read with much precision. Additional scale marks were scribed onto the barrel, under a microscope, assuming linearity between the 250 psi marks. The stress-time curve shown in Figure 5 allowed failure stress to be esimated rather precisely by measuring loading time to failure.

3) A summary of average failure stresses obtained in multiple specimen tests is given in Table 4 together with nickel flash data and average PEI meter counts in the button holes. It can be seen that the amount of metal bared at failure is proportional to the nickel deposited during specimen preparation. The individual values for the 16 grade 5 specimens given in the lower part of Table 4 illustrate the scatter of failure stresses (from 550 to 834 psi) and also show that, within this group of specimens, the amounts of bare metal reflected by the meter counts in the button holes occur randomly over the scattered failure stresses.

The results in Table 4 reflect elements of both encouragement and discouragement. The amount of bare metal by meter counting in the button holes places the six DOAAC grades in the same order as measurements on drop-weight deformed specimens. The failure stresses, on the other hand, fail to place the different grades with the possible exception of grades 1, 2, 3 in the order which we have assumed is valid.

Two hypotheses are suggested to explain the lack of significance of the failure stresses in the button tests:

HYP I. Two things occur, either in rapid sequence or simultaneously when failure occurs: a) a disc of enamel is sheared out and b) the enamel disc is parted at the vertical location of failure in the enamel system. This hypothesis suggests that both the stress required to shear out the disc and the stress required to remove the sheared disc from the substrate are combined in the total stress at failure. If the stress component required to shear the disc could be separated from the adherence stress the latter might serve to evaluate adherence. Experiments to test this hypothesis are described in a later paragraph.

HYP II. During the removal of buttons by tensile stress, a critical strain within the metal initiates micro-cracking at the button circumference. Additional stress causes crack growth resulting in catastrophic failure.

Experiments with an altered hold-down plate to avoid circumferential micro-cracking are underway. Enameled specimens on heavier gage metal will be used to test the micro-cracking theory. Other changes in the button geometry may be helpful in testing the validity of this hypothesis.

4) Sand Blast Series. Two possible approaches are visualized to eliminate or avoid the shear stress referred to in hypothesis I of the previous paragraph.

a) remove the enamel layer except under the button by sandblasting. This should avoid the restraint of the continous enamel layer and the failure stress might consist only of that required to overcome adherence.

Table 5 gives results of the sand blast experiment. Two buttons were attached to each specimen. One was pulled off before and the second was pulled off after sandblasting. The failure stresses after the sandblast treatment were always about 30 percent less than that required before cutting away the enamel surrounding the button. This appears to be evidence that a shear stress is involved in the normal failure mechanism.

One must not overlook the fact that a porcelain enamel coating on a metal substrate tends to give the system more rigidity than bare metal. The greater flexibility after enamel removal may enhance circumferential micro-cracking by allowing greater deflections and thus lead to a lower failure stress.

b) the second approach to avoiding a shear contribution to failure stress is through the testing of button sized discs of enamel fired onto substrates of various thickness. Specimens have been received but have not yet been tested.

5) Relation Between Button Contact Area and Failure Stress. Two series of tests have been made to study the relationship between the contact area of the buttons and the observed failure strengths. In the hollow button series the contact area was reduced in steps by removing the central part of the contact end of the buttons. The outside diameter of these buttons was unchanged. The solid button series was made by successively reducing the diameter of the buttons to achieve smaller contact areas. Both of these series of modified buttons are schematically illustrated at the bottom of Figure 6 together with the reduced contact areas. An understanding of the results obtained in the hollow and solid series (Table 6) has not yet been obtained and will be sought in future work.

E. Tensile Tests with Specimens of Large Contact Area

In 1959 adherence tests of porcelain enamels were made at Battelle [1/] and tensile values of about 6000 psi were reported. Values obtained with the adhesion tester in our research have yielded values of the order of 600 psi, where the contact area was about 0.5 square inch.

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

Preliminary tests have been made using the same specimen size and method used in the older Battelle tests. The specimens were cut in squares 1.25-inches on a side and epoxied between appropriate hardware to pull apart in a conventional testing machine. Values obtained so far are given in Table 7. It can be seen that the two poorest grades, 6 and 1, have values of 2980 and 3620 psi. Failures in the epoxy at between 4000 and 4800 have prevented obtaining tensile stresses for the better grades of DOAAC specimens. The epoxy used at Battelle which limited their values to 6000 psi seems to be superior to that used in these preliminary tests.

PLANS FOR THE NEXT REPORT PERIOD

Work described above failed to show a suitable relationship between adherence quality and the tensile test results obtained with the adhesion tester. It is believed that failure in the button tests result from micro-cracks initiated at some point on the button circumference. This cracking tendency, a forerunner of failure, would be enhanced by bending moments within the pulling device or by deflection of the specimen under stress. Experiments are planned to avoid or minimize specimen deflection through the use of 1) a heavy back-up plate, or 2) specimens prepared on heavier than 20 gage metal. Specimens are also available on which button-sized discs of enamel were fired onto metal of various gages in the usual way. These specimens might avoid shearing stresses and thus pinpoint an inadequacy of the button tests as currently practiced. A suitable hold-down ring will be used with the series of reduced diameters buttons in an attempt to obtain results in better agreement with theory.

WEATHERING STUDIES

In 1966 an exposure test was initiated which consisted solely of nature-tone enamels on steel. The enamels exposed at Kure Beach and South Florida completed three-years' exposure during this report period. These enamels were returned to the laboratory at the National Bureau of Standards for their three-year inspection.

After the enamels arrived at the laboratory, they were cleaned by scouring thirty strokes with a cellulose sponge that had been moistened with a one-percent solution of trisodium phosphate and sprinkled with calcium carbonate followed by successive rinses with tap water, distilled water and alcohol. After cleaning the specimens were measured for changes in gloss is reported as percentage gloss retained. The gloss and color retention values are presented in Table 8 and Figure 7. These data indicate that these enamels have good color and gloss retention after three years' exposure. After the gloss and color measurements were completed, the enamels were carefully examined for signs of rust. A summary of enamels with either rust spots or irridescent stains (the usual forerunners of obvious rust) is presented in Table 9. These data indicate that almost all the enamels exposed at Kure Beach and half of the enamels exposed at South Florida had signs of rust occurring on them. This was true whether they were tested for continuity of coating before exposure or not. However, these enamels were not tested at high overvoltages so differences between the tested and untested enamels might not be realized. When these enamels were inspected to note only those specimens with obvious rust on them, the results presented in Table 10 were obtained. Thus, it can be seen that while many of the specimens appear to have the beginning of rust, most of the small rust spots are noticeable at this time only by extremely close observation.

The enamels exposed at Gaithersburg, the final site in this test, will complete their three-year exposure during the next report period. A complete report of the three-year inspection of these enamels will be prepared following the completion of this inspection.

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TABLE 1. DOAAC Specimens; Drop-Weight Deformed Using 3/4 and 5/8 in. dies and 70, 80 and 90 in-lbs - Impact Energy

Die	Enamel No.	Individual Values of PEI Meter Counts				Ave.	S.D.
				70 in-lbs			
3/4"	1	59.7	56.7	58.0	66.7	60.3	4.46
	2	48.0	52.7	41.7	43.3	46.4	4.96
	3	21 7	41.3	33.7	25.7	30.6	8.70
	4	19.0	28.7	16.0	19.0	20.7	5.53
	5	23.0	9.3	11.7	14.7	14.7	5.97
						mean	5.92
5/8"	1	85.0	55.7	76.0	65.7	70.6	12.68
	2	7.7	18.0	42.7	27.3	25.2	27.02
	3	9.3	28.7	17.3	2.7	14.5	11.19
	4	11.7	24.3	11.7	12.3	15.0	6.21
	5	9.0	5.3	6.0	1.0	5.3	3.30
						mean	12.1
				80 in-lbs			
3/4"	1	62.0	65.7	58.3	54.0	60.0	5.01
	2	56.3	48.0	48.3	49.7	50 6	3.89
	3	38.7	47.0	34.7	40.7	40.2	5.13
	4	28.7	27.3	17.0	27.7	25.2	5.48
	5	20.7	13.7	24.0	17.3	18.9	4.43
						mean	4.79
5/8"	1	79.0	55.7	65.3	75.3	68.8	10.49
	2	51.3	43.3	10.3	44.3	37.3	18.35
	3	16.3	14.3	23.0	33.3	21 7	8.57
	4	10.0	17.7	11.7	8.3	11.9	4.09
	5	5.7	5.7	4.7	7.3	5.8	1.08
						mean	8.52
				90 in-lbs			
3/4"	1	38.7	60.7	54.0	55.0	52.1	9.41
	2	48.0	45.7	46.3	47.0	46.8	0.99
	3	61.7	21.7	41.7	33.7	39.7	16.81
	4	34.7	27.7	43.0	24.3	32.4	8.27
	5	23.7	18.7	19.7	17.7	20.0	2.63
						mean	7.62
5/8"	1	88.3	60.3	68.7	70.3	71.9	11.78
	2	27.0	20.7	22.3	24.7	23.7	2.76
	3	66.3	21.7	20.7	24.3	33.2	22.09
	4	7.7	12.0	11.7	15.7	11.8	3.27
	5	13.0	9.3	9.3	12.3	11.0	1.96
						mean	8.37

TABLE 2. DIRECT-ON ADHERENCE ROUND ROBIN TEST, PHASE II.

DOAAC Grade	Specimen Data 1/ PEI Adherence Meter Counts Before Circulation	After Circulation	Rating by Industry Observers 2/ A	B	C	D	E	Instrumental Rating 3/
			3/4 inch die indentations					
1	58	64	5	5	5	5	5	4.7
2	56	61	5	5	5	5	5	4.7
3	47	49	4	4	5	5	4	4.2
4	17	31	2	3	3	3	3	2.6
5	21	25	2	3	3	3	3	2.8
1	66	66	5	5	5	5	5	5.2
2	48	55	4	4	5	5	4	4.3
3	39	43	3	3	4	4	4	3.8
4	28	33	2	3	3	3	3	3.2
5	21	22	2	3	3	3	3	2.8
			5/8 inch die indentations					
1	65	68	5	5	5	5	5	5.2
2	51	53	4	4	5	5	4	4.3
3	14	16	2	2	2	3	3	1.9
4	12	12	1	1	2	2	1	1.8
5	6	11	1	1	1.5	2	1	1.4
1	56	59	5	5	5	5	5	4.6
2	51	53	3	4	4	4	4	4.3
3	16	21	2	2	2	3	2	2.1
4	8	12	1	1	2	2	1	1.6
5	6	8	1	1	2	2	2	1.4

1/ Each specimen had two indentations; one over a 3/4 inch die and the other over a 5/8 inch die.

2/ The same deformed specimens were circulated to the laboratories in sequence. Five trained observers assigned visual ratings on a scale of 1 to 5. (1 is best).

3/ The instrumental rating is based on PEI Adherence Meter counts before circulation, see text and Table 3.

Table 3. A Guide For Rating Adherence of Porcelain Enamel Cover Coats Direct-to-Steel.

PEI Meter Counts	Adherence Rating 5/8" Die	Adherence Rating 3/4" Die	PEI Meter Counts	Adherence Rating 5/8" Die	Adherence Rating 3/4" Die
1	1.1	1.7	36	3.3	3.6
2	1.2	1.7	37	3.4	3.7
3	1.2	1.8	38	3.4	3.7
4	1.3	1.8	39	3.5	3.8
5	1.4	1.9	40	3.6	3.9
6	1.4	2.0	41	3.6	3.9
7	1.5	2.0	42	3.7	4.0
8	1.6	2.1	43	3.8	4.0
9	1.6	2.1	44	3.8	4.1
10	1.7	2.2	45	3.9	4.1
11	1.7	2.2	46	4.0	4.2
12	1.8	2.3	47	4.0	4.2
13	1.9	2.3	48	4.1	4.3
14	1.9	2.4	49	4.1	4.4
15	2.0	2.5	50	4.2	4.4
16	2.1	2.5	51	4.3	4.5
17	2.1	2.6	52	4.3	4.5
18	2.2	2.6	53	4.4	4.6
19	2.3	2.7	54	4.5	4.6
20	2.3	2.7	55	4.5	4.7
21	2.4	2.8	56	4.6	4.7
22	2.4	2.9	57	4.6	4.8
23	2.5	2.9	58	4.7	4.9
24	2.6	3.0	59	4.8	4.9
25	2.6	3.0	60	4.8	5.0
26	2.7	3.1	61	4.9	5.0
27	2.8	3.1	62	5.0	5.1
28	2.8	3.2	63	5.0	5.1
29	2.9	3.2	64	5.1	5.2
30	2.9	3.3	65	5.2	5.3
31	3.0	3.4	66	5.2	5.3
32	3.1	3.4			
33	3.1	3.5			
34	3.2	3.5			
35	3.3	3.6			

TABLE 4 TENSILE BUTTON TEST ON DOAAC COVER COATS DIRECT-TO-STEEL

Grade	Nickel Flash	Failure Stress	PEI Meter Counts	Number of Specimens
	mg/sq. ft.	psi		
6	0.0	636	54	16
1	0.029	566	53	16
2	0.05	592	42	22
3	0.065	648	21	8
4	0.09	612	12	16
5	0.158	649	18	16

Grade 5 individual determinations arranged in order:

	of determination: psi	of determination: meter counts	of increasing stress at failure: psi	of increasing stress at failure: meter counts
	598	11	550	19
	645	25	564	17
	834	19	564	22
	550	19	577	17
	679	14	591	21
	679	20	598	11
	679	19	598	14
	577	17	645	19
mean	655	17.8	585	17.5
	807	13	645	25
	598	14	672	17
	699	15	679	14
	564	22	679	20
	672	17	679	19
	591	21	699	15
	564	17	807	13
	645	19	834	19
mean	642	17.2	712	17.5

TABLE 5 SUMMARY OF SAND BLAST SERIES

	Before Sandblasting			After Sandblasting			
Grade	n	Failure psi	V, %	n	Failure psi	V, %	Shear Stress 1/
6	7	655±110	18	8	435±110	31	34
1	12	630 70	18	7	495 65	14	22
2	6	670 140	20	8	485 140	35	28
3	8	660 40	7	8	360 74	25	46
4	6	680 120	17	8	545 80	18	20
5	5	765 150	19	8	465 90	24	39
	Mean		16.5			24.6	32

1/ Difference between failure stress before and after sandblasting is suggested as the shear stress. It is expressed here as a percentage of the failure stress before sandblasting.

TABLE 6 THE EFFECT OF BUTTON GEOMETRY ON FAILURE STRESS 1/

HOLLOW BUTTON SERIES

Contact area, square inches	0.172	0.289	0.424
Mean failure stress, psi	1320	1015	590
Number of Determinations	10	5	6
Coefficient of Variation, %	30	10	19
95 Percent Confidence limits	290	120	115

SOLID BUTTON SERIES

Contact area, square inches	0.114	0.223	0.361	0.482
Mean failure stress, psi	1225	680	645	680
Number of Determinations	7	6	6	6
Coefficient of Variation, %	14	19	21	17
95 Percent Confidence limits	155	133	142	119

1/ All determinations on DOAAC Grade 4 specimens

TABLE 7 PRELIMINARY TENSILE TEST VALUES

Specimen Data			Tensile Failure Stresses Specimen Area:			
DOAAC Grade	PEI Meter Counts	Instrumental Rating 1/	1.56 sq. in 2/ n	1.56 sq. in 2/ mean psi	0.11 sq. in. 3/ n	0.11 sq. in. 3/ mean psi
6	--	--	2	2980	7	1090
1	69	5.0	2	3620	2	1250
2	37	3.5	2	(4360)4/	2	1120
3	22	2.6	2	(4470)4/	2	1130
4	12	1.8	2	(4840)4/	7	1220
5	6	1.1	2	(4030)4/		----

1/ See TABLE 3.

2/ Loading rate 10 psi per second.

3/ As in Solid Button Series, TABLE 6, loading rate 28 psi/second

4/ Failure in the epoxy adhesive.

Table 8. Summary of Gloss and Color Data for Nature-Tone Enamels Exposed for Three Years

Enamel	Percentage Gloss Retained Kure Beach	South Florida	Storage	Color Retention Kure Beach	South Florida
101	78.1	89.7	99.9	98.5	99.3
102	73.9	87.8	100.4	98.5	99.2
103	72.5	89.8	99.2	98.9	99.3
104	64.2	90.2	99.0	99.0	99.6
105	79.9	89.2	98.0	98.9	99.4
106	64.0	90.1	96.2	96.5	98.5
107	78.0	90.3	96.7	99.2	99.3
108	92.5	91.2	98.3	99.5	99.2
109	97.2	87.1	97.2	99.6	99.3
110	101.2	86.5	95.6	99.7	99.7
111	82.9	91.0	97.3	98.2	97.1
112	94.3	95.0	98.6	99.4	99.6
113	93.9	103.1	104.7	99.7	99.3
114	76.2	91.5	96.6	99.0	99.7
115	86.5	97.6	100.6	99.3	99.4
116	79.8	93.3	95.9	98.4	99.5
117	74.7	84.1	97.0	97.9	99.4
118	72.4	85.3	92.6	99.2	98.8
119	70.5	85.1	93.4	99.0	99.6
120	74.8	90.5	93.1	98.7	99.6
1	74.8	83.8	93.9	98.9	99.7
3	70.1	84.3	95.1	98.8	98.6
4	71.6	92.6	89.1	97.9	99.3
6	158.6	177.7	181.9	99.4	99.6
7	139.9	149.8	146.9	99.8	99.4
Average	84.9	95.5	102.3	98.9	99.3

Table 9. . Summary of Enamels with Rust appearing on the Surface after 3-Years' Exposure

Enamel	Specimens Rusted at Kure Beach						Specimens Rusted at South Florida					
	Selected by High-Voltage Probe			Randomly Selected			Selected by High-Voltage Probe			Randomly Selected		
	6-mos.	1-yr	3-yr	6-mos	1-yr	3-yr	6-mos.	1-yr	3-yr	6-mos	1-yr	3-yr
101	0	0	3	1	1	3	0	0	1	1	1	3
102	1	1	1	1	1	2	0	0	0	1	1	2
103	3	3	3	3	3	3	0	0	2	0	0	0
104	0	0	3	1	1	3	0	0	1	2	2	2
105	1	1	2	2	2	2	0	1	3	0	1	3
106	0	0	2	0	0	2	0	0	3	1	1	3
107	1	1	3	2	2	3	0	0	0	0	0	1
108	0	0	2	0	0	1	0	0	0	0	0	0
109	0	0	1	0	0	2	0	0	2	0	0	0
110	0	0	2	0	0	0	0	0	1	0	0	0
111	1	1	3	1	1	3	0	0	1	0	0	2
112	0	0	3	0	0	3	0	1	2	0	1	1
113	0	0	2	0	0	2	0	2	2	0	0	1
114	0	0	3	0	0	3	0	0	1	0	0	1
115	0	0	3	0	0	3	0	0	2	0	0	1
116	0	0	3	0	0	3	0	0	1	0	1	2
117	0	0	3	0	0	2	0	0	0	1	1	3
118	0	0	3	0	0	3	0	0	1	0	0	3
119	0	0	2	0	0	1	0	0	3	0	1	3
120	0	0	3	0	0	3	0	0	0	0	0	0
1	0	1	3	0	0	3	0	0	0	0	0	3
3	0	0	2	0	0	3	0	0	1	0	0	1
4	0	0	3	0	0	3	0	0	2	0	0	3
6	0	0	3	1	2	3	0	0	3	1	1	3
7	0	0	3	0	1	3	0	0	3	0	0	3
Total	7	8	64	12	14	62	0	4	35	7	11	44

Table 10. Summary of Enamels with obviously Rusted Surfaces after Three Years' Exposure

Enamel	Specimens Rusted at Kure Beach		Specimens Rusted at South Florida	
	Selected by High-Voltage Probe	Randomly Selected	Selected by High-Voltage Probe	Randomly Selected
101	1	1	0	0
102	1	1	0	0
103	3	3	0	0
104	0	1	0	0
105	0	0	0	1
106	0	1	0	0
107	0	0	0	0
108	1	0	0	0
109	0	0	0	0
110	0	0	0	0
111	0	0	0	0
112	0	0	0	0
113	0	0	0	0
114	0	0	0	0
115	0	0	0	0
116	0	0	0	0
117	0	0	0	0
118	0	1	1	0
119	0	0	0	0
120	0	0	0	0
1	1	1	0	0
3	0	0	0	0
4	0	0	0	0
6	0	1	1	0
7	0	0	2	1
Total	7	10	4	2

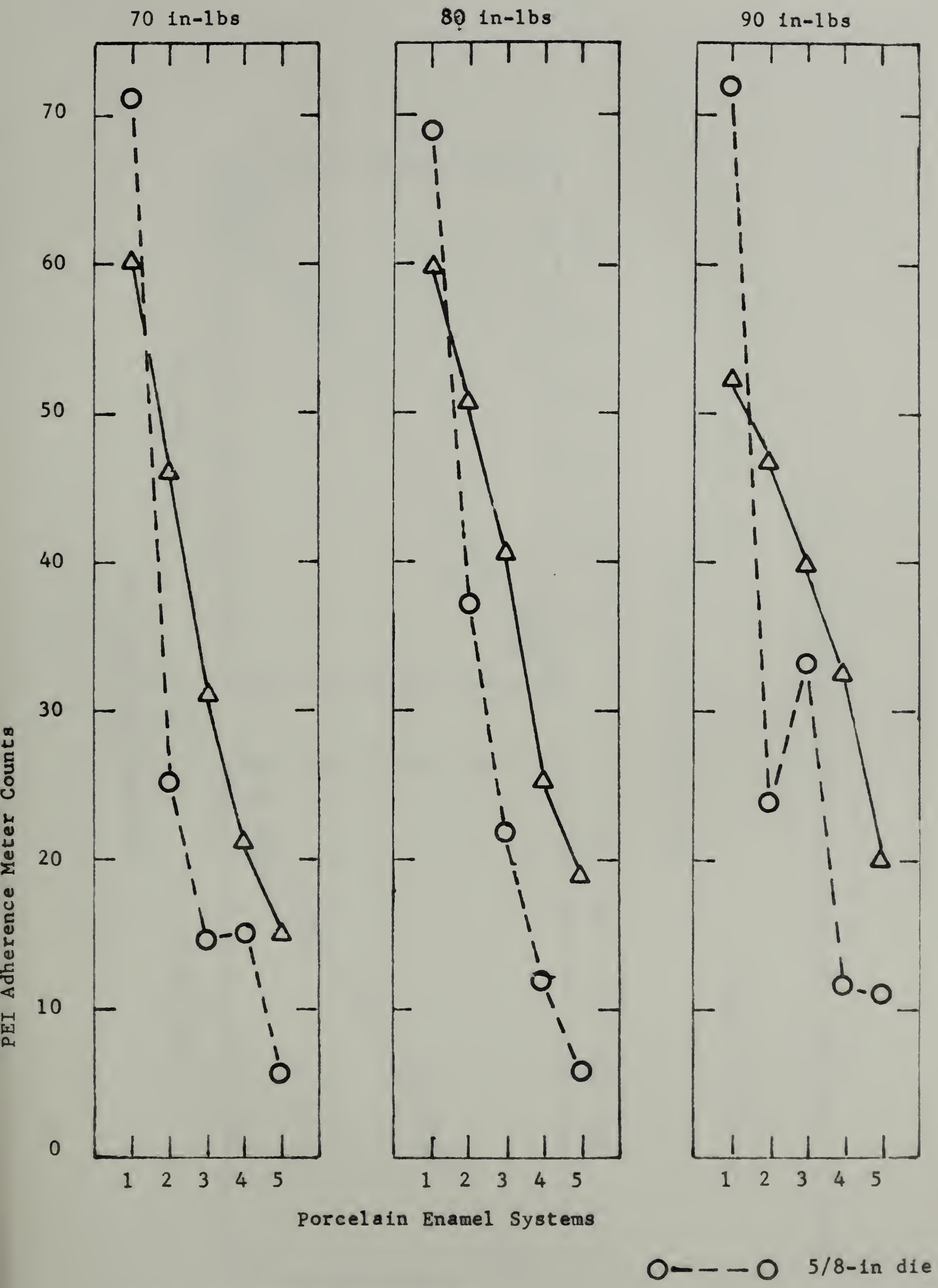
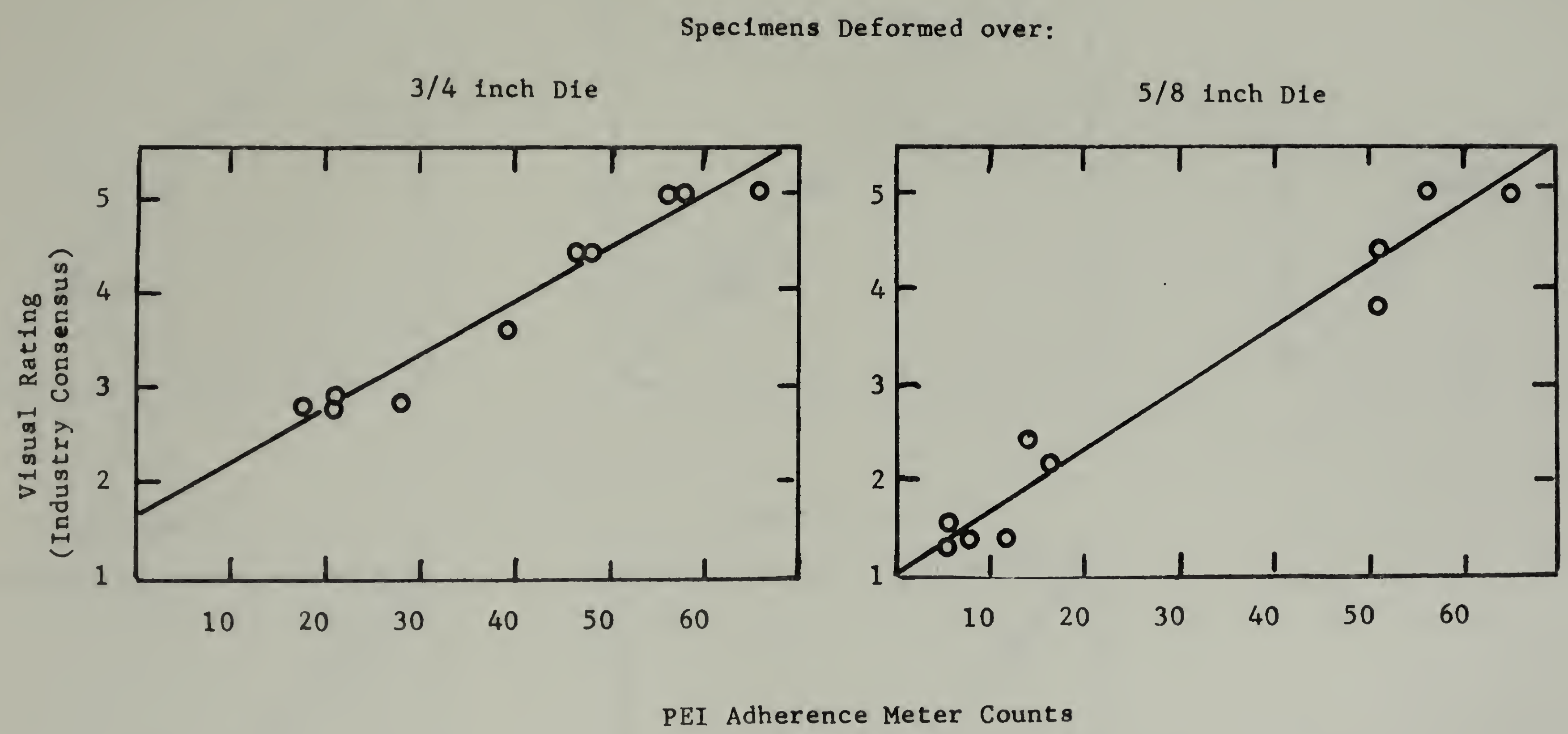


FIGURE 1. Effect of Die Size and Impact Energy on PEI Adherence Meter Response.



$y = 1.625 + 0.056\ x$

$y = 1.058 + 0.063\ x$

FIGURE 2. The Visual Rating of Drop-Weight Deformed Specimens.

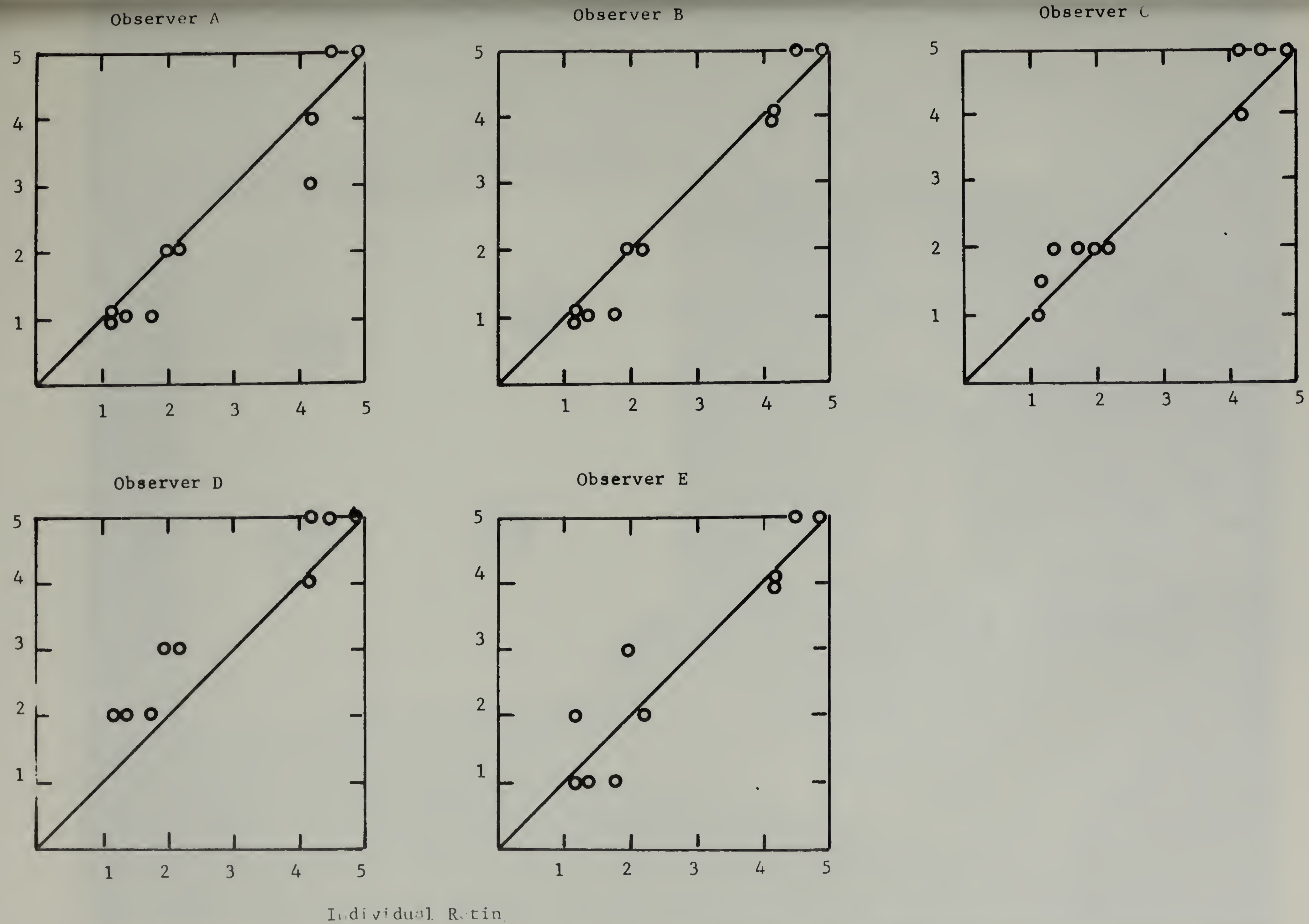


Figure 3. Comparison of Individual and Visual Ratin

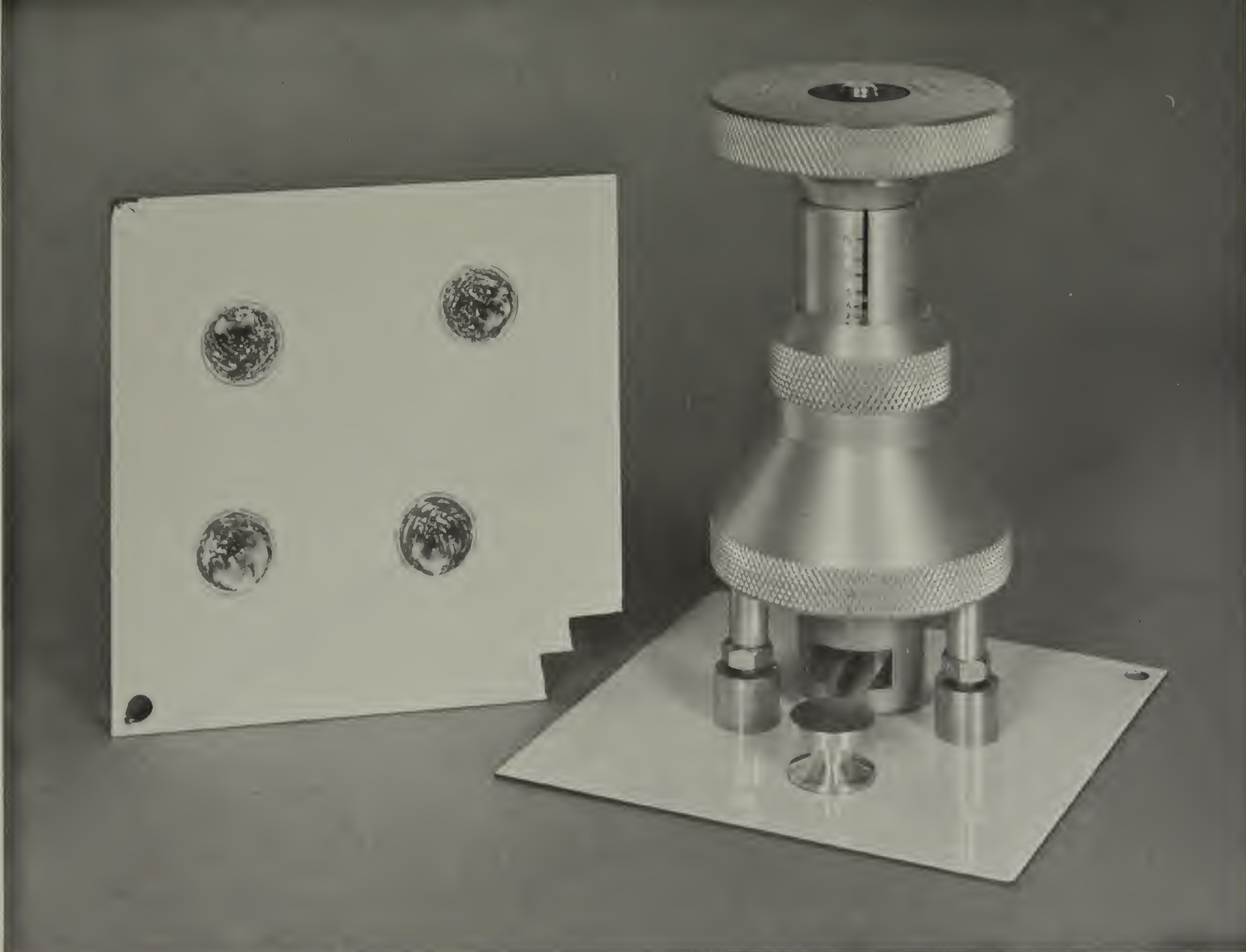


Figure 4

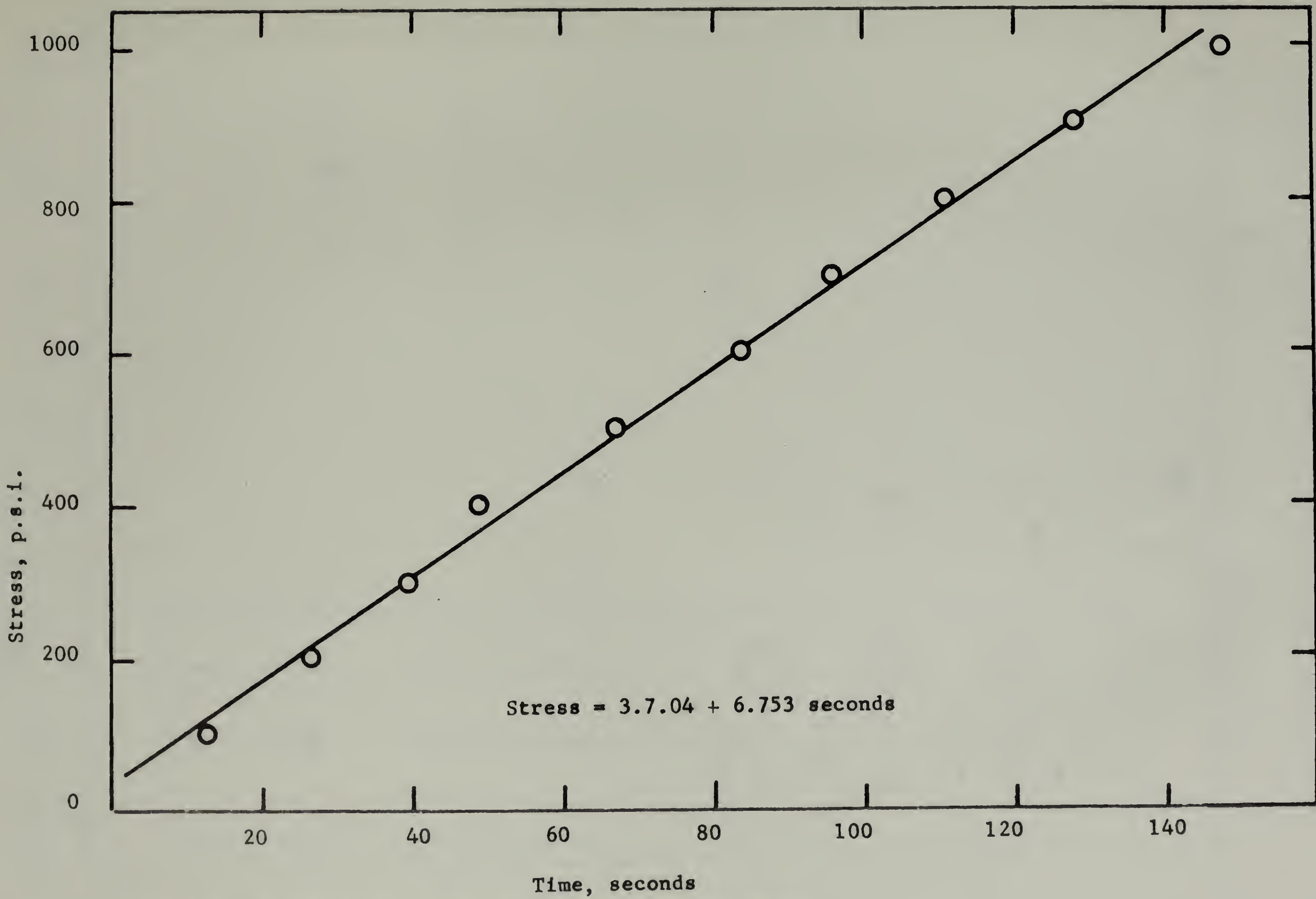


FIGURE 5. Stress-time Curve, Motorized Adhesion Tester

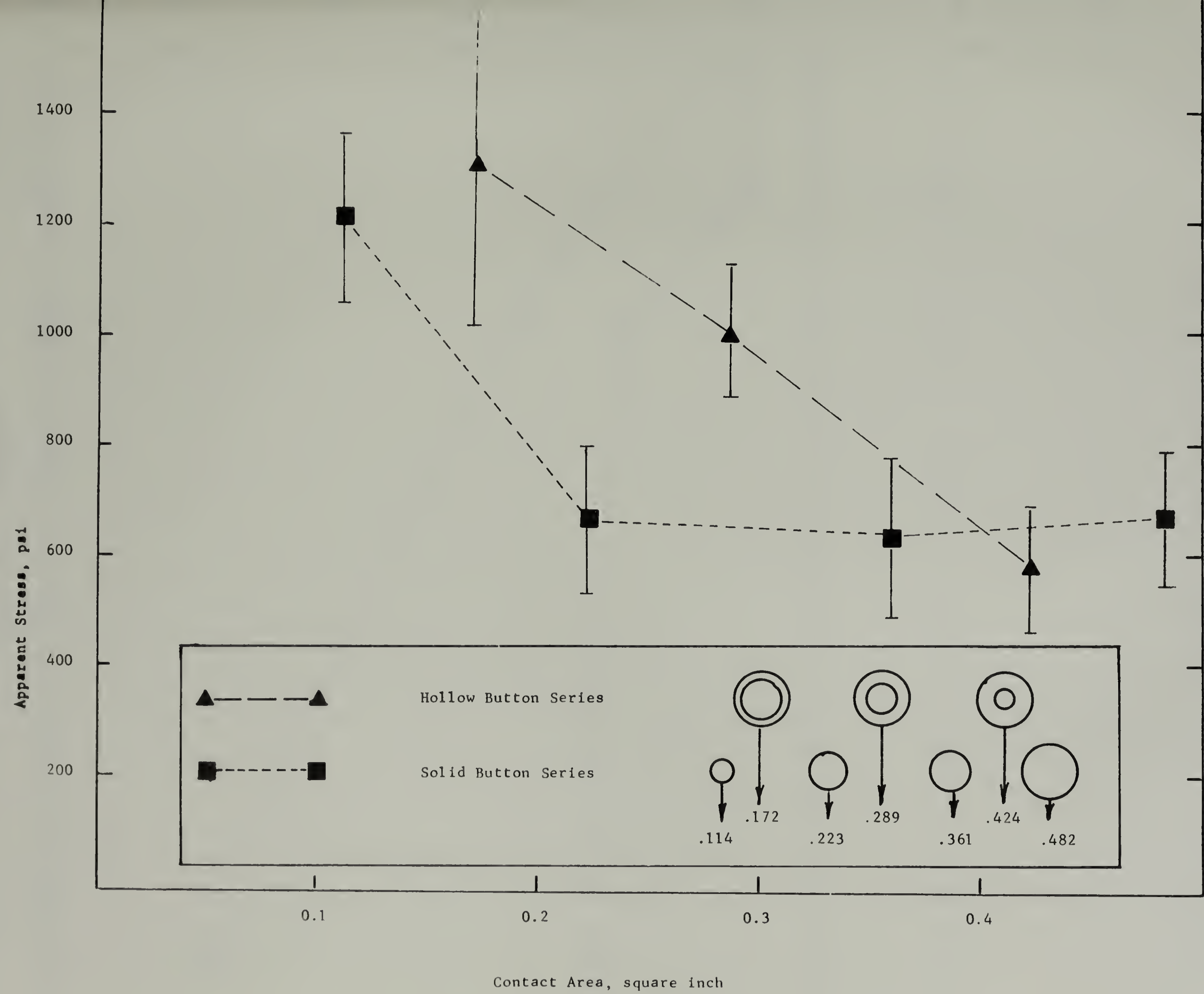


FIGURE 6. The relation between Apparent Strength and Contact Area

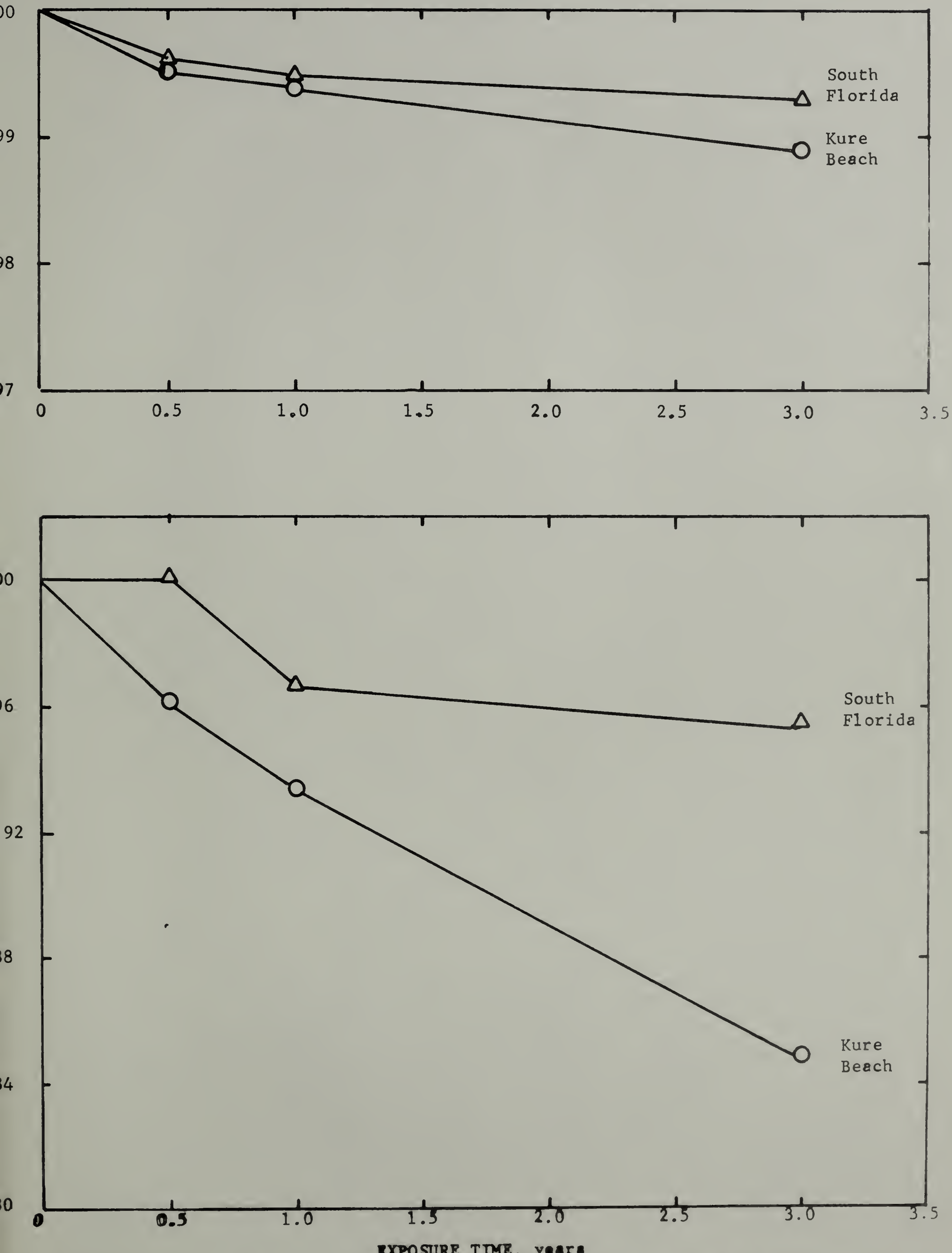


FIGURE 7. Effect of Exposure Time on Gloss and Color Retention of Nature-Tone Enamels on Steel

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