A DETERMINATION OF THE INFLUENCE OF

POLISHING PROCEDURES ON THE SURFACE PROPERTIES

OF DENTAL AMALGAM

by

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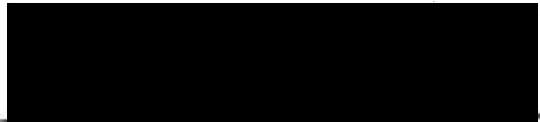A Thesis

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INTRODUCTION

The polishing of a restoration has become accepted as a beneficial finishing phase of operative dentistry. The presence of a sizable quantity of heat resulting from the polishing of certain restorative metals can be appreciated by anyone having vigorously polished a gold crown while holding it in his hand. The phenomenon of heat production in dental amalgam is not as readily noticeable clinically, but has been observed to some degree experimentally (1).

The practice of overheating amalgam by polishing is considered to result in the freeing of mercury from the mass and undesirable alterations of the physical properties of the material (2-6). Such changes are believed to render the restoration less resistant to marginal fracture and corrosion (4-5). The release of mercury at the amalgam surface is felt to be reflected in changes of surface finish (5) or the production of surface voids following the rebinding of freed mercury (3).

The release of mercury from the mass of hardened amalgam has been stated to occur between the temperatures of 65-100° C. (7-13). The specifications by which a given polishing procedure is to effect the release of this mercury have not been published, although the comparative ease of its commission has been cited (4). Gray (9) has demonstrated the possibility of immediate weakening of the restoration, as manifested by a decrease in compressive strength of samples, at temperatures above the transition temperature of amalgam. Also, the inability of the restoration to maintain its polished lustre has been recorded as an

aftermath of thermogenic polishing of amalgam (5). Nevertheless, only limited clinical or laboratory evidence is available to support the above contentions of delayed structural weakening (e.g., after 24 hours) and decreased resistance to corrosion of thermogenically polished amalgam surfaces.

It is the purpose of this investigation to seek specific information regarding the effect of thermogenic polishing procedures upon the surface properties of dental amalgam. The investigative approach used to accomplish this purpose will involve the use of certain in vitro test measurements of the surface in an attempt to demonstrate the existence of these effects of structural weakening and decreased resistance to corrosion attributed to thermogenic polishing of amalgam.

The mode of polishing selected offers conditions which are experimentally convenient and possible in the mouth. Since the effects of this polishing are most likely to be found near the surface, it seems reasonable to employ those tests which either directly or indirectly assess the integrity of this region. The tests chosen for this purpose were Knoop micro-indentation hardness, transverse strength, and observation of corrosion in electrolyte solution. The properties or qualities measured by these tests will be considered here only briefly, and the reader is directed to Appendix II for a more detailed exposition justifying their use, as well as that of the polishing procedures employed.

Indentation hardness represents the resistance of the surface to penetration by an indenter acting under a given load. The depth of penetration or observation beneath the surface is influenced by the load employed. Although this resistance is not clearly defined as a unique property (14-17), it has been offered to reflect a quality like strength

or resistance to wear or deformation (14, 16-17). More practically, hardness has been mentioned as having a bearing on the clinical performance of amalgam (17-18).

The limiting resistance of freely supported material to flexural rupture by a centrally applied load can be taken as a prospective measure of the property of transverse strength (4-p. 23). This property embodies the resistance to stresses which are felt to be important in marginal failure (19) and to offer a better indication of amalgam failure than the more widely used index of compressive strength (20).

The process of amalgam corrosion is not clearly understood. The progress of this reaction presents a picture of deterioration of the surface and, to some extent, the body of this material (21-22). The factors of surface roughness and fluid environment of the restoration are believed to influence this corrosion reaction (21). Other conditions of the metallic restoration, such as stress concentration, surface impurities, and other heterogeneities of the metal, are also felt to be of importance in this reaction (4-p. 345-8).

The observation of this in vitro reaction between amalgam and its electrolyte environment does not offer sufficient information to predict the behavior of this polished material in the mouth. In lieu of a test which provides such information (23), the purpose of this observation is to discern differences in the overall extent of surface reaction under conditions reported to allow discrimination (21).

From the foregoing, it can be appreciated that these tests may not presently measure properties known to predict the clinical behavior of amalgam alloy. However, some evidence of their usefulness in evaluating certain changes measured has and/or will be presented. In addition, they

appear to be among the best of the tests available by which an _in vitro_ study such as this might be undertaken.

BACKGROUND INFORMATION

The reader is referred to Appendix I and III for details of litera-
ture review of the topics of polishing, mercury release and corrosion
and for preliminary temperature measurements of amalgam. The conclu-
sions drawn from these preliminary observations of the effect of
temperature on the release of mercury from set amalgam are: (1) that
release occurs at a surface temperature of amalgam near 177° F.
(80.5° C.); (2) that polishing in a thermally abusive manner can result
in temperature increases at the amalgam surface in excess of 177° F.;
(3) that polishing with a prophylaxis cup can be controlled, for specified
conditions of time, load and speed of application, to effect release;
and (4) that the amalgam surface, having been heated or polished to
release mercury, and then allowed to cool, evidenced a frosty appearance
different from the original.

METHODS AND MATERIALS

This experiment was divided into two parts, each of which was intended to contribute to our understanding of the amalgam surface after thermogenic polishing.

The first and major portion to be presented had as its objective an evaluation of the amalgam surface polished by procedures P_1, P_2, and P_3 (to be defined) and measured by the tests of Knoop hardness, transverse strength, and the observation of corrosion. The basic plan for this evaluation is outlined in Fig. 1. The sequence of presentation of the factors outlined will be that of polishing procedures followed by the surface tests, in the order listed. Because of the difference in experimental designs employed, a detailed account of each design will be deferred until the introduction of the appropriate test.

The minor portion will be presented last under the heading of "Other Tests". This part was intended to supply additional information about: (1) changes in amalgam hardness pertaining to its mercury composition and clinical behavior; and (2) changes in the amalgam surface that relate to time and polishing.

Polishing Procedures

Initial Surface Preparation. The amalgam surface preparation to be described was common to all amalgam surfaces employed in this study. Any polishing procedure to be examined was superimposed over this initially prepared surface.

Figure I.

TESTS EMPLOYED TO EVALUATE
SURFACE EFFECTS OF POLISHING AMALGAM

TEST	POLISHING PROCEDURE		
	P_1	P_2	P_3
1	KNOOP HARDNESS		
2	TRANSVERSE STRENGTH		
3	OBSERVATION OF CORROSION		

Initial surface abrasion and rough polishing was accomplished by using a sequence of 600-A carbide, 4/0 emery, and micro-cut[1] papers, followed by slurries of XXX-silex and amalgloss powders applied to the micro-cut paper backing. The specimen was drawn back and forth (one stroke) across these agents, in the order presented, and in an increasing numerical pattern of 3, 4, 5, 6, and 7 strokes, respectively. Following each of these specified number of strokes, the specimen was rotated 90° clockwise until a complete cycle with each abrasive was obtained. The terminal strokes of abrasion and initial polishing were arranged to be at right angles to the major axis of Knoop indentations and along this axis of the transverse strength specimen. All conditions of preliminary preparation, save that of emery paper, were conducted in the wet state. The application of emery paper was carried out with considerable caution to minimize the production of heat and surface distortion (24). Each specimen was washed with detergent, rinsed with room temperature tap water, and examined under 10 X magnification between each abrasive and polishing stage.

Standard Polishing (P_1). Following the initial surface preparation, all specimens received what is here to be defined as a "standard polish" of wet amalgloss applied with a prophylaxis cup. This was considered to be the control polish.

The force and speed of abrasive application was 4 ounces or less at a rotary speed of 1,000 to 1,500 r.p.m.. The duration of polishing was 15 seconds. The force of application of the prophylaxis cup was indicated by the flexion of the rubber cup during rotation. The rotary

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[1] Scientific Supply Company, Division of Van Waters and Rogers, Inc., Portland, Oregon

speed of the applicator was checked periodically with a Jagabi tachome-
ter[1]. This polishing time was the same for all polishing procedures,
and represented a useful period within which the abrasive remained in
the rubber cup during polishing.

Abusive Polishing Procedure (P₂). The thermogenic polishing proce-
dure which was defined as abusive employed the application of a dry
prophylaxis cup to the amalgam surface to release mercury. This release
was achieved by applying the prophylaxis in a rotary manner at a load of
application of 6 to 8 ounces and a speed of 10,500 ± 500 r.p.m. for a
duration of 15 seconds. The method used to control the rotary speed of
the rubber cup was the same as that employed with standard polishing.
The specimen being polished was thermally insulated in a supporting
cradle and was in contact at two lateral surface edges with small crys-
tals of tempilaq[2] paint (175° F.).

The control of this procedure has been represented as the most con-
sistent compromise between the factors contributing to force and heat.
The use of the tempilaq crystals was included as a precaution to assure
that no specimen surface should have failed to exceed 175° F.. The
monitoring of the load of application of the prophylaxis cup was accom-
plished using the loading platform described elsewhere (25). The
specimen subjected to this type of polishing procedure was observed to
assume a uniformly frosty appearance, after a few seconds of cooling,
unlike the original polished surface.

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[1] James G. Biddle Company, Philadelphia, Pennsylvania.

[2] Tempil Corporation, New York, New York.

<u>Abusive Polish Followed by Standard Repolishing Procedures</u> (P$_3$).
This procedure differed from P$_2$ by the addition of a standard repolishing procedure after a momentary delay. All specifications described for the standard polish were employed for this procedure.

Physical Property Tests

<u>Knoop Hardness Test</u>. This experiment employed a factorial design which included the factors of polishing procedure and indenter load on the hardness of amalgam. The polishing treatments used were those defined as P$_1$, P$_2$, and P$_3$. Five indenter loads of 10, 25, 50, 100, and 300 grams were used to determine surface hardness. Fig. 2 illustrates the basic plan of usage for the different polishes and indenter loads.

Fifteen amalgam specimens were fabricated, mounted in acrylic, and their surfaces prepared as described. After suitable randomization, these specimens were divided into three equal groups, according to the polishing treatment to be administered. Twenty four hours after polishing, each specimen surface was indented. In order to balance the distribution of indenter loads over the specimen, each amalgam surface was divided into 5 equally spaced, vertical zones, as shown in Fig. 3. The five indenter loads (L$_1$.....L$_5$) were applied to 5 specimens according to a 5 x 5 Latin square shown in Fig. 4. In this way, indentation at a given load and in a given zone appeared only once for any polishing treatment. Twenty indentations were obtained for each zone of each specimen tested. A total of 1,500 observations was obtained.

The indentation test was performed on a Kentron micro-hardness tester[1] using a duration of indenter descent and dwell for all indenter

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[1] Torsion Balance Company, Clifton, New Jersey.

Figure 2. SCHEMATIC DIAGRAM OF EXPERIMENTAL DESIGN

		POLISHING PROCEDURE		
		P_1	P_2	P_3
INDENTATION LOAD IN GRAMS	10	SPECIMEN 1 * ⋮ 5		
	25	1 * ⋮ 5		
	50	↑		
	100	↑		
	300	↑		

* SAME SPECIMEN

↑ SAME CONDITIONS AS ABOVE

Figure 3.

INDENTATION ZONES OF
AMALGAM SPECIMEN
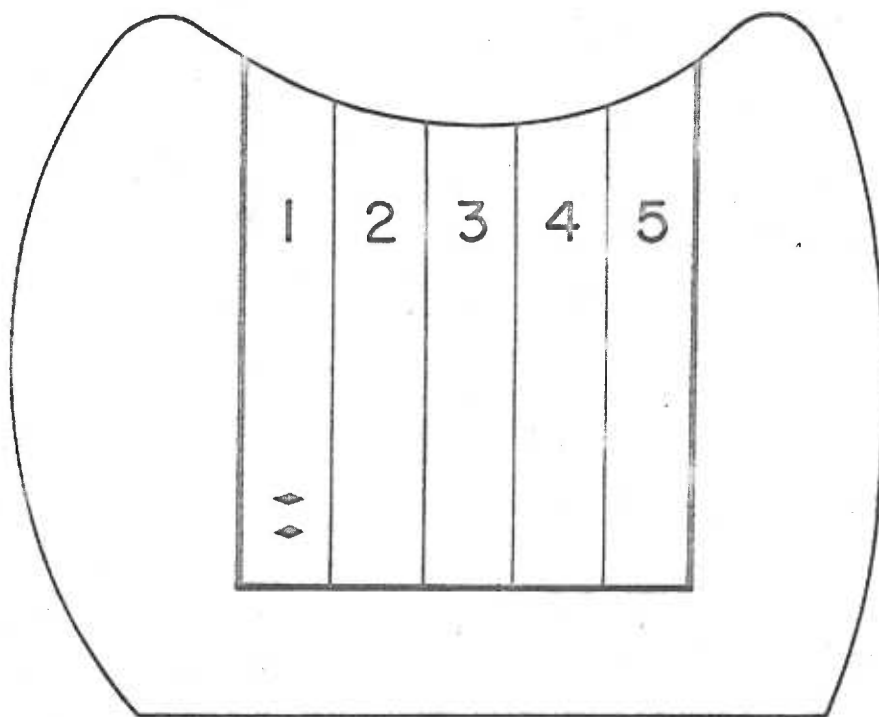
Figure 4.

APPLICATION OF INDENTER LOAD
ACCORDING TO LATIN SQUARE

ZONE OF INDENTATION

		1	2	3	4	5
NUMBER OF SPECIMEN WITH THE SAME POLISH	1	L_1	L_2	L_3	L_4	L_5
	2	L_2	L_1	L_5	L_3	L_4
	3	L_3	L_4	L_1	L_5	L_2
	4	L_4	L_5	L_2	L_1	L_3
	5	L_5	L_4	L_3	L_2	L_1

loads of 20 and 7 seconds, respectively. The surface of the mounted amalgam specimen was leveled on the specimen stage, oriented in the optical field, and subdivided into rectangular zones by producing a scratch using a 10 gram indenter load, as illustrated in Fig. 3. The commencement and spacing of indentations within any zone, irrespective of indenter load, was controlled by the micrometer travel of the specimen stage. No indentations were made closer than twice the diagonal length of an indentation to any edge of the specimen surface, or more than one diagonal length for each other. The criterion for rejection of any indentation was that of indistinct diagonal terminals, local or general geometric assymetry, discontinuity within the indentation, or interference with test surface discontinuities. Unless otherwise specified, all indenting procedures were undertaken 24 hours after the final polishing procedures were applied.

Knoop indentation hardness numbers were calculated according to the expression: $KHN = L/d^2 C_p$, where L represents the indenter load in kilograms, C_p represents the indenter constant, and d denotes the length of the diagonal of the rhomboid indentation in millimeters. The length d is expressed as the product of filar length and an appropriate optical magnification factor specific for the testing system. The particular indenter constant employed with this instrument allowed a −0.3% correction for all hardness values calculated on the basis of $C_p = 7.028 \times 10^{-2}$.

Transverse Strength Test. This was a randomized block experiment with three replications. The effect of replication was considered to be the day-to-day variation in the preparation of amalgam specimens used in this test. Sixty three specimens of amalgam were obtained in a manner to be described. Twenty one specimens were prepared on each of three

successive days and the daily output was assigned, at random, to receive one of the polishing treatments (P_1, P_2, and P_3). Each specimen was stored in physiologic saline at 38° C. during the 24 hour interval between the application of polishing and transverse fracture.

The method used for securing transverse fracture of the specimen has been described by Mahler, et al. (25). The fracturing procedure, from immersion bath to fracture, was conducted as rapidly as conditions would permit in order to allow for a reasonable consistency in the temperature change of the specimen prior to fracture. The immersed specimen was blotted dry and held between the fingers prior to transfer into the testing apparatus. The polished surface of the specimen was placed in the testing apparatus so as to fracture in tension. Transverse fracture was effected in an Instron[1] testing machine at a headspeed of .05 inches/minute. The load at fracture was utilized in determining the transverse strength of the specimen according to the equation:

$S = 3 \ WL/2bh^2$, where;

$\quad$ S = Transverse strength,

$\quad$ W = Load at fracture,

$\quad$ L = Distance between supports,

$\quad$ b = Width of specimen,

$\quad$ h = Thickness of specimen.

The measurement of the variables of b and h was obtained using a micrometer ($\nleq 2 \times 10^{-4}$ inch) as the mean of two readings for each of the fractured ends of each specimen.

Corrosion. Thirty acceptable specimens of buccolingual profile

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[1] Instron Engineering Corporation, Canton, Massachusetts.

Figure 5.

MOUNTED AMALGAM
SPECIMEN

ACRYLIC
CASEMENT

DIVISIONS OF SURFACE
OF MOUNTED AMALGAM
SPECIMEN.
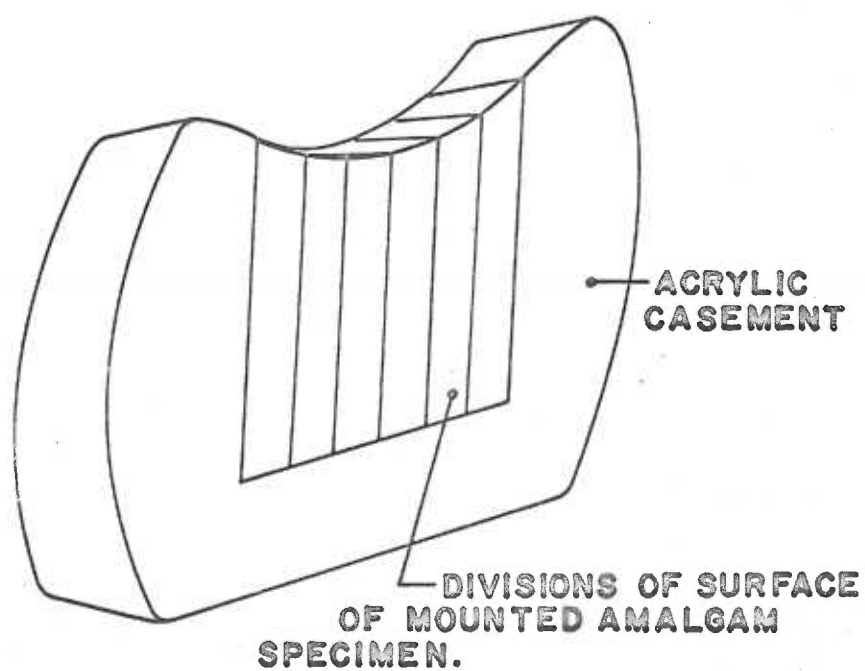
were sealed in polyurethane, mounted in acrylic, as illustrated in
Fig. 5, and divided into six groups by systematic randomization. Fur-
ther assignment to one of three polishing treatments and two immersion
baths was accomplished by a similar random process. The immersion media
consisted of 50 ml. of physiologic saline and molar equivalent Na_2S
solutions placed in capped Erlenmeyer flasks. The temperature of these
solutions was kept at 38° C., except for short periods of observation.

Gross observation at 1-10 X magnification was accomplished under
conditions of immersion through the neck of the container by oblique
illumination. Microscopic examination was performed on an auxiliary
series of amalgam specimens passing all criteria of selection for this
test. The 125, 250, and 625 X magnification and vertical lighting
facilities of the Kentron optical system were employed for the above
examination. Each specimen surface was washed in laboratory glassware
detergent, rinsed in distilled water, and patted dry with paper tissue
prior to each microscopic examination. The frequency of observation was
daily for the first week and then weekly for a period of 65 days.

Every effort was exerted to maintain the moistened status of these
amalgam surfaces during handling or polishing prior to immersion. Just
prior to immersion, each specimen was washed, rinsed, and examined at
10 X magnification to eliminate the carry-over of any visible abrasive
residuum from previous polishing procedures into the immersion bath.
New immersion solutions were prepared weekly from reagent grade NaCl
and $Na_2S \cdot 9H_2O$. The solvent employed was distilled water at laboratory
temperature. In the case of the sulfide solution, it was necessary to
dissolve the sulfide crystals in approximately one sixth of the total
solution volume of heated distilled water. This concentrate was

subsequently cooled to room temperature prior to transfer and dilution in a volumetric flask. Oxygen replenishment and solution agitation were limited to a daily swirling of five revolutions, with the container cap off, followed by 5 container inversions, with the cap on. No specimen was allowed to remain in the solution with its polished surface touching the flask.

Amalgam Specimen Preparation

Specimens of amalgam were prepared from S. S. White New True Dentalloy (batch No. 1716288) using a precondensation mercury alloy ratio of 1.11/1. The appropriate amounts of alloy filing were weighed using a Crandall balance and then incorporated with the required volume of mercury. The mixture was triturated for 9 seconds in a Wig-L-Bug[1]. No precondensation expression of mercury was attempted. Hand condensation of small increments at a pressure of 2,000 $\neq$ 500 p.s.i. was employed for this proportioning of alloy and mercury. The method of monitoring and recording condensation pressure has been described elsewhere (25). Two types of specimens were obtained. Specimens used for transverse strength testing have been described (25). Specimens for the observation of corrosion and micro-indentation hardness were obtained from a chrome-plated mold in the shape of a buccolingual proximal profile as shown in Figs. 6 and 7. The total time of condensation for transverse strength and proximal profile specimens were 3.0 to 3.5 minutes and 6.0 to 6.5 minutes, respectively. The profile sections shown in Fig. 3 required two mixes of amalgam to fill the mold.

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[1] Crescent Dental Manufacturing Company, Chicago, Illinois.

Figure 6.

CHROME MOLD
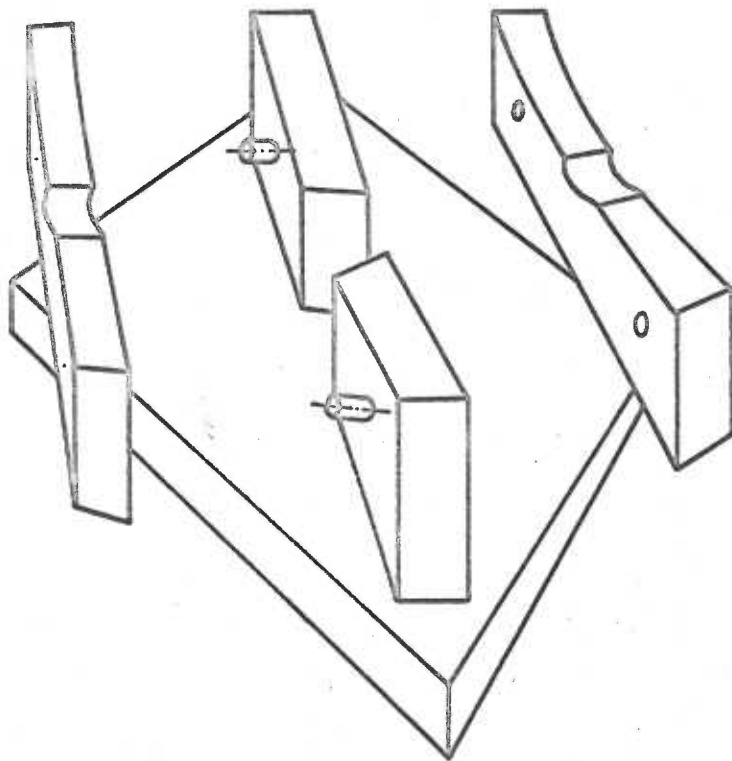
Figure 7.

UNMOUNTED AMALGAM
SPECIMEN

6.5mm

3.0mm

3.0mm

7.0mm
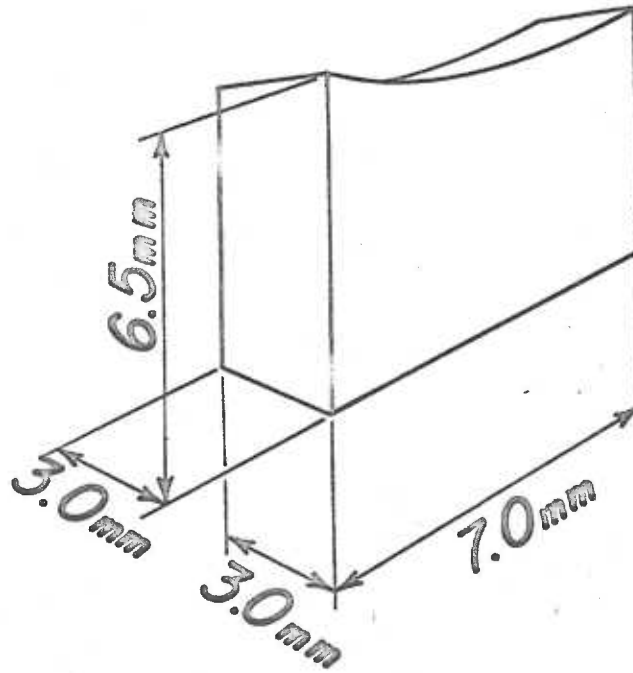
Specimens of amalgam for tests other than for transverse strength were coated with a single application of a polyurethane resin prior to mounting in quick-curing acrylic resin. The urethane resin was applied to mask all but the test surface of amalgam. The completed specimen is illustrated in Fig. 5. The initial polymerization of the acrylic casement about the amalgam specimen was retarded by cooling to avoid heating of the specimen and to minimize surface porosity of the polymerized acrylic, which might otherwise collect abrasive particles capable of affecting the corrosion reaction. Unless otherwise specified, the age of all specimens at the time of polishing and testing was 7 and 8 days, respectively. During the 24 hour interval between these two operations, each specimen was stored at 38° C..

The level of condensation pressure was the criterion for selecting specimens for all tests. The additional criterion for selecting corrosion specimens was that no specimen possessed any major surface flaws or fissures and that no more than 5 pits of discernible size at 10 X magnification (roughly 50-75μ in diameter) were visible on the finished surface.

RESULTS AND DISCUSSION

Knoop Hardness

The analysis of variance[1] of hardness data is shown in Table 1 of Appendix IV. The conclusions drawn from this analysis were:

(1) There was no significant difference among hardness values obtained over the range of indenter loads employed.

(2) There was no significant interaction between the factors of indenter load and polishing procedure in the determination of this surface property.

(3) There was a significant effect of polishing on amalgam hardness. There was a significant difference in hardness following abusive polishing. No significant difference in hardness resulted from the repolishing of abusively polished amalgam.

The mean Knoop hardness values obtained for each of the polishing treatments are listed in Fig. 8. This figure indicates that a small but significant increase in surface hardness resulted from abusive polishing of amalgam and that, in spite of careful repolishing afterwards, the hardness of the amalgam did not change or revert to its initial value. Each mean value presented in Fig. 8 was derived from the pooling of all hardness values obtained at each indenter load.

The observations recorded were individual Knoop hardness numbers.

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[1] Analysis performed through the courtesy of Western Data Processing Center; Los Angeles, California.

Figure 8.

MEAN MICRO-INDENTATION HARDNESS (KHN) OF DENTAL AMALGAM

POLISHING PROCEDURE APPLIED TO AMALGAM		
P_1	P_2	P_3
112.17	115.06	117.35

This procedure is common to previous reports (26-31) concerning this property. However, another measurement, indentation length in filar units, has been used as the observed data in a study (32) of indenter load dependency in Knoop hardness determinations.

The rationale for using indentation filar length is not at all clear; particularly when this measurement, per se, has little meaning in a comparison of indentations obtained from differing indenter loads. In addition, those (32) suggesting the use of this measurement have not demonstrated any advantage in describing or understanding the resistance of a material to penetration by an indenter. When such an analysis is conducted, as described (32), it cannot routinely deal with individual hardness numbers unless a proportionality between indenter load and indentation area is assumed to exist. Lacking this assumption, the choice of indentation filar length manifests no obvious merit over some other measurement, e.g., indentation length or possibly the power to which this length is raised.

From the foregoing, it is believed that the choice of hardness numbers as the basic observation in this study was best suited to examine the affect of polishing and indenter load on the determined property of amalgam hardness.

The method of dividing the specimen surface into areas or regions was used to control the variations in the condensing procedure which might be reflected in hardness variations of amalgam between buccal and lingual regions of this proximal section of amalgam represented in Fig. 3. The occlusal-gingival pattern of indentation was employed to include any hardness variations which, as suggested by preliminary examination, might attend the addition of increments of plastic amalgam

into the mold or proximal portion of larger class II restorations (35).

No indenter load dependency was observed in the determination of amalgam hardness. This finding is not in agreement with that of Ryge, et al. (32). The source of such a disparity may arise from the different approaches to the test of load dependency. To facilitate our understanding in this area, a brief description of these approaches is in order.

In the present study, hardness values (K.H.N.) were determined according to the expression $L = (K.H.N.) \cdot C_p \cdot d^2$, in which $\underline{L}$, $\underline{C}_p$, and $\underline{d}$ are specified. Essentially, the test of load dependency is to show that the proportionality between indenter load and indentation area, of which hardness is a factor, is constant. In the "Meyer Analysis" employed by Ryge, et al. (32), this same test examines departures of the exponent $\underline{n}$, in the expression $L = ad^n$, from the value of 2. This examination requires two determinations, i.e., the exponent $\underline{n}$ from the constant $\underline{a}$. Thus, it can be generalized that both approaches question the proportionality of indenter load and indentation area, but only the former deals directly with the value of hardness irrespective of load dependency. The latter approach deals with the value of hardness only when load dependency is assumed to be absent.

There is further evidence that the working hypotheses of these tests differ; a condition which may contribute to this lack of agreement. The difference which seems important is the prospect that the decision as to the extinction of n-2 for all indenter loads is influenced by the magnitude of $\underline{n}$ and consequently by $\underline{a}$. Since there is no indication of the simultaneous determination of $\underline{a}$ and $\underline{n}$ for each indentation, the determination of $\underline{a}$, either by graphic or analytic projection, appears subject to

the inaccuracies of extrapolation beyond the limits of observed data. This unsatisfactory aspect of the graphic determination of a, with subsequent low values of n, has been demonstrated (32).

Perhaps there are factors whose influence on this lack of agreement is more easily recognized. One of these may be the different experimental conditions, such as a shorter length or span of applied indenter loads. Aside from the statistical implications of such an observation, the current choice of indenter loads tends to avoid the largest reported (32) region of disproportionality between load and indentation area. Examination of the graphic presentation of n (32) does not indicate any notable change in n for the large majority of loads used in this study. One factor not described by Ryge, et al. (32), but discussed earlier, is the control of the possible effect of hardness variations over the surface of the amalgam indented. Finally, a factor which may influence these differing results is the possibility of an effect of amalgam composition upon load dependency, as suggested from the results of other tests to be given.

The lack of significant interaction of indenter loads and polishing procedures indicates that this particular indenting process was not sensitive to the presence of a superficial, polished layer of homogenous or deformed amalgam. Due to the depth of the surface examined or penetrated by this process, the preceding conclusion would not be expected to discredit the observation of this type of layer following etching attack (34-36).

Transverse Strength

The analysis of variance of transverse fracture data is given in Table 2 of Appendix IV. The conclusions drawn from this analysis were

as follows:

(1) There was a significant effect of polishing upon the transverse strength of amalgam.

(2) There was a significant difference in this strength of amalgam subject to abusive polishing.

(3) There was no significant difference in this strength due to careful repolishing of abusively polished amalgam.

Figure 9 shows the mean values of transverse strength for the polishing treatments employed in this test. Here there is an indication that a small but significant decrease in transverse strength resulted from the abusive polishing of amalgam and that careful repolishing of this . . not effect a restoration of this strength. The mean strength of the standard polished amalgam specimens was consistent with previous determinations of this property under similar conditions of specimen preparation (20).

Following the polishing by abusive methods, the observance of a small but significant increase or decrease in hardness and transverse strength, respectively, may reflect an important surface characteristic of this material. No attempt will be made, on present evidence, to explain this divergence of material response. Furthermore, these results do not provide sufficient basis to examine the proposition that surface alterations have initiated early transverse fracture. Further tests may prove helpful in this regard. The most important conclusion to be noted is that little change in either hardness or transverse strength results from these abusive polishing procedures when compared with the standard method.

Figure 9.

MEAN TRANSVERSE STRENGTH (PSI) OF POLISHED DENTAL AMALGAM.

POLISHING PROCEDURE APPLIED TO AMALGAM		
P_1	P_2	P_3
19,449	18,556	18,414

Corrosion

The observations derived from amalgam corrosion are to be presented in the form of a qualitative comparison of: (1) the progress of corrosion among the three polishing procedures imposed on the amalgam surface; and (2) the characteristics of this reaction in the immersion media employed. This comparison will be presented according to the type of observation used, with an appropriate consideration given to the factor of observation time, where possible.

Macroscopic Observation

1. Effect of Polish on Corrosion. The application of polishing procedures P_2 and P_3 resulted in a frosty-appearing amalgam surface after 24 hours immersion. The specimens receiving these treatments yielded an apparent reflectivity to oblique illumination of some magnitude less than surfaces polished by procedure P_1. For this reason, a gross comparison of the initial corrosion reaction, as influenced by the polish, was not possible with any expected degree of accuracy. Much of this distinction was lost by prolonged immersion exposure.

2. Effect of Immersion Media on Corrosion. The most striking single observation of the corrosion of the amalgam surface was the rapid initiation of this reaction in the saline bath. The progress of this reaction was gauged by the presence and number of interference color patterns on the washed and dried or immersed amalgam surface. Under available laboratory illumination, many specimens evidenced localized color patterns embracing as many as two complete orders of color change in the first 12 to 24 hours of saline immersion. The first order would be represented by a series of colored bands from yellow or gold to red-violet, to blue, and, finally, to gold. The distribution of these bands

appeared to simulate the topographical contour lines about a hilly region of a map. At low magnification, higher order green-colored bands were frequently observed near many local regions of active, surface build-up. No visible interference orders were noted on any specimens under sulfide immersion. Under similar conditions of oblique illumination, these specimens revealed a generalized golden color reflection from the metallic surface.

The progress of the reaction in saline was characterized by an edge involvement of some specimens or by a generalized confusion in color change. These events were not observed in the sulfide medium. A comparison of the two corrosion processes suggested that the saline reaction demonstrated a rapid onset and a pronounced tendency toward a localization of attack, with or without the presence of a general deterioration of the surface. In contrast, the sulfide reaction was best described as slowly initiated attack of the whole surface. The overall appearance of surface deterioration seemed to be more advanced on the specimens in saline than in the sulfide solution at the termination of the experiment.

Microscopic Observations

1. Effect of Polish on Corrosion. After the first week of immersion, the surfaces receiving the three polishing treatments were quite readily distinguishable from one another on the basis of what appeared, at 625 X magnification, to be unreacted alloy particles (Ag_3Sn) and surrounding matrix. For the purpose of further description, these structures were tentatively identified as unreacted alloy particles (U.A.P.) and matrix, as previously demonstrated (34). Further clarification of this identification will be considered.

The amalgam surface receiving polishing procedure P_1 evidenced

clearly defined and, for the most part, apparently unaffected U.A.P.. The surface polished by procedure P_2 revealed U.A.P., which appeared to be different from those polished by procedure P_1. For lack of a more knowledgeable reference, these particles for conditions of procedure P_2 were described as appearing "eroded". This characteristic took the form of multiple peripheral invaginations, internal sinusoid tracts, or rather extensive internal granularity or pitting. The latter form was fairly characteristic of the surface polished by procedure P_3. In some regions, this intra-particular granularity was so advanced that the faint outline of the particles could scarcely be distinguished from the granularity of the matrix structure.

After several weeks and up until the termination of observation, distinctions in the appearance of corrosion among polishes could not readily be made. The surface receiving the standard polish evidenced few recognizable U.A.P. and these were generally associated with extensive transparent granularity. Those surfaces receiving polishing treatments P_2 and P_3 did not present any important number of recognizable surface structure.

2. Effect of Immersion Media on Corrosion. The amalgam subjected to immersion appeared to demonstrate, at least initially, the results of an etching type of reaction. The U.A.P. and surrounding matrix became evident after one week immersion. The progress of corrosion of the surface, with regard to these particles, was one of an apparent deterioration within the particles themselves. Small, circular, transparent granules were seen to develop within the boundaries of these U.A.P.. On occasion, criss-cross scratches were visible on these particles and, in other instances, these small intra-particle granules would simulate

scratches by their chain-like orientation. In the sulfide medium, these granular spots inside the U.A.P., as well as throughout the matrix, would evidence a reddish-pink cast suggestive of mercuric sulfide formation at these sites.

After one week of saline immersion a frequent, although inconsistent, observation was that of an apparent advanced reaction of the matrix beyond that of the U.A.P.. This distinction was based upon the color differences of red and blue matrix as compared with the golden color of the U.A.P.. Much of the inconsistency of this observation appeared to be due to general color changes which transcended all structural   .ts within a given region. The distinction between these two surface components, under conditions of initial sulfide immersion, was attributed to the darkness of the matrix and a golden coloring of U.A.P..

3. Discussion of All Observations. The rate and extent of corrosive deterioration of the amalgam surface observed in this study appeared to be somewhat retarded from that described in other experiments (21). Certainly, no categorization of degrees of corrosive attack, as proposed in the previous citation, could have been used with this immersion reaction.

The understanding of whether amalgam corrosion, as evidenced in the time and corrosive media employed, represent additive or subtractive reactions at the surface is incomplete. Evidence from chemical analysis (22) and x-ray diffraction analysis (21) suggests that an additive reaction upon the surface is most noteworthy following certain fluid exposures. However, neither examination excludes the possibility of a subtractive surface reaction and, in the instance of corrosive loss at the surface or within the body of the material (22), there is an

indication of possible amalgam solution in the corrosive medium. Also, there appears to be little evidence which refutes the possibility that this loss could have occurred in conjunction with an additive or film growth type of reaction. Thus, the corrosion reactions observed in this study may have been additive or subtractive and may have occurred either singly or together.

Microscopic examination of amalgams from saline immersion revealed local regions of addition or accumulation of corrosion products upon the surface, as indicated by Schoonover and Souder (22). This would not be inconsistent with observed interference bands requiring odd quarter wavelength differences of surface film for polychromatic light sources (37). Similarly, the consistency of surface color reflectance after sulfide immersion would not indicate any noticeable breakdown or repair of film growth (21).

These reactions would not be characterized as involving gross pitting attack of the surface in either media. This does not agree with other observations of this reaction but the duration of corrosive exposure, in this instance, is much shorter (22). Another factor which may effect this agreement involves the recognition of this type of attack. One problem is to distinguish between pits which are due to corrosive loss and those which belong to the original surface. As indicated later in the discussion of surface flow, some of these voids may exist at the surface but be concealed by surface deformation during preliminary or subsequent polishing. In attempting to minimize this problem, a further criterion to limit the number and size of surface voids was imposed upon specimen selection at a preliminary polishing stage most likely to reveal these voids. It was also reasoned that the presence of these voids

tended to concentrate stress and invite electrochemical dissimilarities which might mask any general reaction in the vicinity.

Two details of corrosion observation require further clarification. Following an examination of colored, crystalline compounds which might be formed between amalgam and sodium sulfide, the most reasonable conclusion regarding the observed reddish granules was that mercury within the U.A.P. became available to form mercuric sulfide. This is consistent with a previous identification (21).

The appearance of U.A.P. after corrosion was verified by acid etching using 30% nitric acid as suggested by Smith, et al. (34). This was accomplished while observing the etching reaction under 125 and 250 X magnification. The large U.A.P. defined by corrosion were found to be continuous with the underlying amalgam structure. Using the same procedure but with frequent interuptions to identify, the smaller, irregular or granular particles were identified. In many instances these particles could not be followed to the end of the etching process, thus indicating some degree of discontinuity of these smaller particles with the underlying subsurface structure.

Based upon these observations which indicate that initial corrosion behaves something like etching, some conjecture regarding structural changes of the amalgam surface due to polishing may warrant mention. The application of polish P_1 did not appear to result in any gross disruption of the unreacted alloy particles. The application of an abusive polishing treatment, releasing bound mercury from the surface, resulted in alterations of the observable unreacted particles. These observations suggested a further reaction or solution of this unreacted phase with the available unbound mercury in the area. This argument would not

be inconsistent with the following: (1) the absence of phase distinction above 80° C., as demonstrated by Schmitt and quoted by Ryge (38); (2) the recognized capacity of solution of mercury in Ag_3Sn; (3) the implied presence of mercury within the boundaries of unreacted alloy particles, by the formation of an identified (21) mercury compound; and (4) the recognition of the presence of erosion of this unreacted phase in amalgam containing increased amounts of mercury (34).

Perhaps another term might be applied to the surface effects of abusive polishing. A surface layer of homogenous amalgam with increased resistance to corrosion is thought (4-p. 398) to result from polishing. The application of the term homogenization to the effect of polishing the amalgam surface is somewhat vague, but the results of its use would appear to represent, essentially, a heat treatment. From previous statements about the heating of set amalgam and after considering the temperatures which might be used to homogenize the original Ag_3Sn ingot (4-p. 360), the most reasonable interpretation of amalgam homogenization from polishing would be represented as a further solution of mercury in the unreacted Ag_3Sn phase. If this is the connotation given to the term homogenization, then the observations of surfaces polished by P_2 and P_3, in the early stages of corrosion, might indicate a partial homogenization by decrease in phase distinction. Whether or not a degree of homogenization is related to the phenomenon of surface flow of amalgam can only be suspected from existing evidence.

These observations of amalgam surface deterioration, following prolonged immersion in these media, do not directly offer any promising method of quantitatively measuring the extent to which this reaction might be expected to proceed in a given time interval. The reaction

rate, as based on weight loss or gain and measured during immersion, could not be expected to reveal the severity of corrosive attack in local areas versus that of the entire surface. Perhaps electrochemical methods, i.e., anodic and cathodic polarization curves, polarization-break, and polarization resistance (39), might prove to be of value in the problem of corrosion rate determination. It is evident that further study should be directed toward a consideration of what features of the polished restoration surface make it more able to resist corrosive attack than the unpolished surface. Also, what practical limits of smoothness and the area of inclusion exist in the clinical polishing of a restoration which allow for the most efficient conduct of this practice?

4. Summary of All Observations. From an overall point of view, little, if any, gross distinction can be made regarding differences in the extent of corrosion of polished amalgam surfaces. However, certain microscopic differences between polished surfaces are appreciated. A much more clearly defined difference was observed between media than between polishing treatments. This would support the conclusion drawn by Schwartz, et al. (21), that the judgment of the effect of corrosive attack upon dental restorative materials would best be made in several corroding media. Such a conclusion would follow directly from the supposition that more than one discrete reaction is possible for a given material subject to several environment conditions.

Clinical Implications of Tests

The similarity in instance of change in the resistance to indentation and transverse loading of polished amalgam is summarized in Fig. 10. While these changes did not occur in the same direction, the

Figure 10.

SUMMARY OF THE EFFECTS OF ABUSIVE POLISHING

TEST OBSERVED		POLISHING TREATMENT RENDERED		
		P_1	P_2	P_3
1	Knoop Hardness	control	increase	increase
2	Transverse Strength	control	decrease	decrease
3	Corrosion – Gross Surface Appearance			
	Initial	shiny	dull	shiny
	After 24 hours	shiny	no change	dull
	After 2 months	dull	no change	no change

magnitude of change was not large. A feature common to both measurements is the lack of significant change exhibited by amalgam after having been repolished in procedure P_3.

It is difficult to state directly the practical importance of hardness changes shown in Fig. 8. Some evidence is required which portrays these changes, or associated factors, within the limits of detectable clinical performance. A recent study of the frequency of clinical amalgam failure (40) employed a criteria of failure which included marginal fracture, surface roughening, surface wear, tarnish and corrosion, and gross fracture of the restoration. The determination of increasing failure was made on restorations possessing compositions of 48, 58, and 62% residual mercury. Two factors appear to have a common bearing upon the increase in clinical failure reported. These are; residual mercury content and the manipulative technics used to secure these compositional levels of mercury in amalgam.

On examination of the measurements in Fig. 13, a considerable difference in hardness might be attributed to amalgam compositions closely resembling those identified with increasing clinical failure (40). The magnitude and direction of these composition differences in hardness provides some insight into the importance of a small increase in hardness resulting from abusive polishing, particularly, if hardness is used to reflect a resistance to surface roughening and wear (40). A desirable upper limit of amalgam hardness has not been established. From this, it is felt that serious impairment of the performance of this amalgam would not be expected from abusive polishing.

The associated effects of manipulative factors and residual mercury contents upon the transverse strength of amalgam has been studied (20).

Within a general compositional range previously identified with increased clinical failure, a considerably greater decrease in transverse strength has been reported (20, 25) than is shown in Fig. 9 to result from abusive polishing. Much larger changes in transverse strength than shown in Fig. 9 might also be associated with rather wide variations in residual mercury content attributed to the clinical handling of amalgam (41-42). On the basis of these observations, the loss in flexural strength resulting from abusive polishing might not be expected to reflect a clinically important change in the structural behavior of this material.

Little clinical inference regarding the effect of abusive polishing can be drawn from the observation of corrosion, as outlined. What might be expected in the way of long-term stability of surface lustre after polishing is not known. The lack of this stability (dulling) following the repolishing procedure P_3 did not appear to depend upon the presence of the immersion bath. Surfaces polished similarly but allowed to remain in air demonstrated the same diminution of lustre. This would tend to support earlier observations (4-5). Surfaces repolished 24 hours after the application of P_2 evidenced a considerable reflective lustre in air storage after a period of three months. The implications drawn from these observations are twofold. Firstly, the apparent change in reflectivity of the repolished surface may coincide with the recombination of unbound mercury released during the polishing procedure. If this were true, one might deduce that this recombination in the mouth would proceed independent of the conditions of immersion (12, 43). Secondly, in attempting to gain the smoothest possible surface of more lasting lustre, repolishing after abusive polishing might best be deferred until a later

date. The support for this recommendation of delayed repolishing does not appear to differ greatly from that given to the polishing of incompletely set amalgam (4). The above surface qualities, which are sought by this delay, may facilitate cleaning and minimize the accumulation of food and bacteria upon the restoration (4-p. 590).

When the results of these tests are viewed as a composite, little change in the material can be ascribed to the application of abusive polishing. Furthermore, examination of polishing in Fig. 18 and an appreciation of the possibilities of temperature changes through heat conduction (1), impresses one with the sizable energies: (1) which are expended upon amalgam to achieve such small changes; and (2) which are available to structures adjacent to the restoration being polished. Hence, these impressions contribute to a degree of clinical perspective which specifies that the viable system necessarily adjoining the restoration may be more sensitive to the energy imposed upon the restoration than the restorative material itself. A major implication of this argument is that further study of the effects of this type of polishing might be more rewarding if directed to consider the response of the tooth.

OTHER TESTS

Because of our limited knowledge of amalgam and its surface hardness, other tests were performed in this study in order to provide: (1) a reference point for the behavior of amalgam whereby the practical importance of preceding test results might be appreciated, (2) additional information about the conduct of the indentation test, and (3) an increased understanding of polishing which might be afforded by this testing method.

Effect of Mercury Content and Repolishing of Heated Amalgam Surfaces on Knoop Hardness

Rationale. In order to associate the hardness index with mercury-containing amalgam shown to provide greater likelihood of clinical failure (40) and to gain some insight into the ability of the test to discriminate differences in surface mercury content which might have resulted from the freeing of mercury during thermogenic polishing, the following test was conducted.

Method and Materials. This test utilized a factorial design representing a four-way classification of the factors of mercury-alloy ratio, indenter load, occlusal-gingival areas of indentation, and polishing procedures. A schematic illustration of the application of this design on the Knoop hardness determination of the amalgam surface is offered in Fig. 11.

Twelve amalgam specimens, six regular and six mercury-rich, were obtained and their surfaces given preliminary polishing preparation as

Figure II.

SCHEMATIC DIAGRAM OF
EXPERIMENTAL DESIGN

INDENTATION LOAD IN GRAMS		AREA OF SPECIMEN INDENTATION		RECONDENSATION MERCURY-ALLOY-RATIO			
				REGULAR		MERCURY-RICH	
				POLISHING PROCEDURE			
				P_1	P_4	P_1	P_4
300			L	SPECIMEN 1 * ⋮ 3			
			C	1 * ⋮ 3			
			R	1 * ⋮ 3			
10			L	↑			
			C	↑			
			R	↑			

* SAME SPECIMEN

↑ SAME CONDITIONS AS ABOVE

previously described. All amalgam surfaces were given a standard pol-
ish. After suitable randomization, each collection of a given precon-
densation mercury-alloy ratio was divided in half. Half of the regular
and mercury-rich specimens were subjected to immersion heating followed
by standard repolishing (procedure P_4, to be defined). Each of the four
groups generated by this assignment according to mercury proportioning
and polishing contained three specimens. Each specimen was divided into
three occlusal-gingival areas of indentation, corresponding to the
buccal, center, and lingual surface of mold regions. In turn, each of
these areas was subdivided into two indenting zones to accommodate, at
random, indentation at loads of 10 and 300 grams. Fig. 12 demonstrates
this division and subdivision of the amalgam surface. Twenty indenta-
tions were obtained for each zone, in each specimen, for a total of
1,440 observations.

Following the indentation procedures, the above specimens were sub-
jected to an analysis of residual mercury content. The method followed
was that of Crawford and Larsen (44), modified by the substitution of
Argon for nitrogen gas.

The conduct and specifications of indentation testing were those
previously described for this test, with the exception of the area of
indentation. The division of the amalgam surface followed a similar
pattern as employed earlier, with the exception that the intent in the
present examination was one of measurement rather than control of varia-
tions in hardness from buccal to lingual of the specimen. The indenter
loads employed to assess surface hardness represent the extremes of
values of weight and penetration utilized earlier.

The polishing procedure used to heat amalgam represented a necessary

Figure 12.

INDENTATION AREAS OF
AMALGAM SPECIMEN
SUBDIVISION INTO ZONES
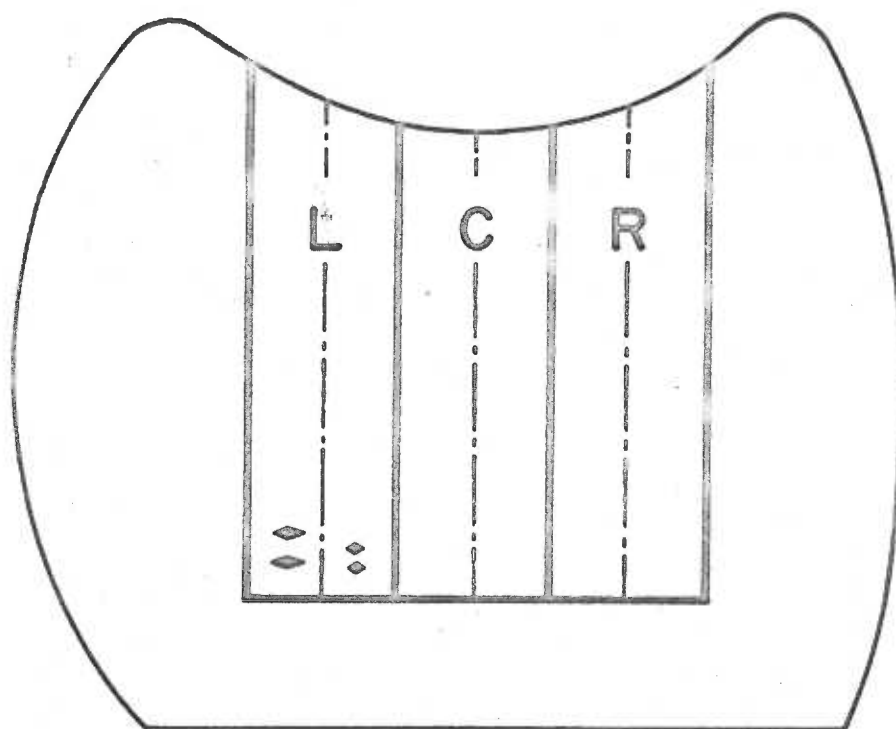
departure from those of the preceding experiment. Procedure P_2 was found, by preliminary examination, to be too deformative in its action on mercury-rich amalgam surfaces, particularly at low indenter loads. For this reason, a purely thermal immersion procedure was chosen which parallels, to a degree, time and temperature specifications of P_2. It is important to note that this immersion process should not be looked upon as representing the intact isolation of the caloric variable from P_2, since frictional components of this polish are absent.

Repolishing was incorporated into this heating procedure to negate the effects of surface recrystallization which might tend to yield spurious recordings of hardness. The repolishing procedure used has already been specified as standard polishing P_1. This combination of immersion heating and repolishing bears a sequential similarity of application to P_3 but was identified as P_4. The immersion heating of amalgam destined to receive polishing procedure P_4 was accomplished by immersing the specimen in a vacuum flask containing water at $180 \neq 1^\circ$ F. for a period of 15 seconds.

Amalgam specimens were prepared using mercury-alloy ratios of 1.11/1 and 2.22/1. Those prepared from the ratio of 1.11/1 were obtained in the same manner described earlier and identified as "regular" amalgam specimens. Those possessing a mercury-alloy ratio of 2.22/1 were defined as "mercury-rich" amalgam specimens. The mercury-rich amalgam was hand condensed at a pressure of 200 p.s.i., or less, and represented an attempt to simply get the plastic amalgam into the mold without extracting too much mercury. Where applicable, all other conditions for handling amalgam, stipulated for regular amalgam specimens, were utilized for those regarded as mercury-rich.

The precondensation mercury-alloy ratios above were chosen to reflect low and high residual mercury contents of amalgam. These were employed to introduce a measure of extremes in this variable, while at the same time providing an amalgam surface containing more than the regular (as used here) amount of bound mercury. The assumption regarding mercury content, considered here, is based upon the absence of any manipulative procedure of specimen preparation which is known to effect appreciable distributional differences in mercury content of the set amalgam surface and subsurface. The practicability of this assumption is supported by the residual mercury analysis of bulk and marginal areas of clinical restorations (42, 45). It was felt, therefore, that a mercury determination of the entire specimen would serve to define a general estimate of preheated surface mercury content.

Results and Discussion. The analysis of variance of Knoop hardness values for conditions of different indenter loads, specimen test areas, precondensation mercury-alloy ratios, and polishing procedures is shown in Table 3 of Appendix IV. The conclusions of interest drawn from this analysis were:

(1) There was no significant effect among either vertical areas of amalgam surface, indenter loads, or polishing procedures, on hardness values. Furthermore, in the determination of this physical property, no significant interaction was observed between the factors of; (a) indenter load and polishing procedure, (b) indenter load and vertical area of amalgam surface, (c) vertical area and polishing procedure, (d) vertical area and precondensation mercury-alloy ratio, and (e) polishing procedure and mercury-alloy ratio.

(2) There was a significant difference in hardness of amalgam

prepared from different precondensation mercury-alloy ratios.

(3) There was a significant interaction of the factors of precondensation mercury-alloy ratio and indenter load on the determination of indentation hardness.

Figure 13 expresses the overall effect of precondensation mercury-alloy ratio on hardness values, along with the residual mercury content for each of the 12 amalgam specimens tested. From the previous assumption about the distribution of bound mercury, an increase in surface hardness of approximately 24 K.H.N. was observed with a decrease in mercury content of the surface region from about 60 to 44%.

Figure 13 also exhibits the hardness values for amalgams subjected to polishing procedures P_1 and P_4. In this instance the release and possible redistribution of mercury about the surface by heating and careful repolishing did not effect a significant change in hardness after 24 hours. The conditions of storage during the time between polishing and testing were arranged to minimize the amount of free mercury on the amalgam surface (12). The effect of much earlier indentation testing will be considered shortly.

The lack of a significant difference in surface hardness among the general areas of what might be considered an exterior proximal surface of the class II restoration does not add greatly to our knowledge of the clinical manipulation of amalgam. The lack of observed difference in the effect of these two indenter loads on Knoop hardness does not conflict with decisions made earlier.

The absence of significant interactions between the factors of indenter load and test area, indenter load and polish, test area and mercury-alloy ratio, test area and polish, and polish and mercury-alloy

Figure 13.

MICRO-INDENTATION HARDNESS FOR DIFFERENT MERCURY CONTENTS & POLISHING PROCEDURES

Polishing Procedure	PRECONDENSATION MERCURY CONTENT				Average KHN
	52.5%		68.84%		
	KHN °	RM% *	KHN	RM%	
P₁	107.84	43.52	83.74	60.43	
	107.01	43.28	85.12	62.23	
	108.48	43.94	84.87	61.96	96.18
P₄	103.75	44.41	83.52	58.17	
	116.16	43.17	87.79	59.92	
	113.06	44.17	90.56	60.93	99.14
Average	109.39	43.75	85.94	60.61	

● KNOOP HARDNESS NUMBER

* RESIDUAL MERCURY CONTENT

ratio would not be viewed with either surprise or expectancy based upon current knowledge of the mechanism of indentation hardness of dental amalgam.

The presence of a significant interaction between the factors of indenter load (depth of surface penetration) and precondensation mercury-alloy ratio may be illustrated in the following situation. A larger index of hardness was obtained for regular amalgam at a 300 gram load than for the lesser 10 gram indenter load on the mercury-rich amalgam surface.

A comparison of mean values of hardness for regular amalgam speci-mens (52.5% precondensation mercury content) in Fig. 13 with the same variety listed in Fig. 8 would denote some type of disparity for condi-tions of apparent identity. Although the effect of the various loads upon the hardness values of amalgam depicted in Fig. 8 was not signifi-cant, an examination of some of the higher hardness values recorded in the mid-range of indenter loads would appear to justify an overall increase due to pooling of these values for all indenter loads.

The Relationship of Post-Polishing Time and Knoop Hardness Determination on Mercury-Rich Amalgam Surfaces

Rationale. A question of some concern to the conduct of the indentation test was touched upon in the preceding discussion. The con-clusion regarding the effect of polishing procedure P_4 on the determina-tion of hardness rested upon the contention that the method of storage would have accounted for complete recombination of any free mercury resulting from polishing. This would suggest that a difference in hard-ness might exist on the surface of amalgams containing some measure of unbound mercury. If this were true, a difference in hardness might be

measured for the same amalgam at different times after polishing. Thus, it was decided to examine the hardness of mercury-rich amalgam within a short interval of time following the application of polishing procedure P_4.

Methods and Materials. A series of indentation measurements was obtained from a mercury-rich amalgam specimen, in the manner described for the previous test, using both 10 and 300 gram indenter loads. These measurements were acquired in such a way that indentations from a given indenter load were parallel but separated by at least two indentation diagonal lengths from those of similar description placed prior to heating. To accommodate any deterioration in conformation of these indentations, the indenting and measuring was done in pairs and in groups of 10 before changing to a different load. A total of 20 indentations was measured at each load. The mean hardness values so obtained are compared in Fig. 14 with those of prior measurement taken in the same area 24 hours after polishing. A time interval of 45 minutes after polishing was necessary to secure the required number of observations, allowing an initial two to three minutes for leveling and alignment of the specimen on the micrometer stage of the testing instrument. Twenty four hours after indenting, the tested zone was examined to discredit any of these indentation measurements which had violated the standard of proximity to the previous indentations in that zone. In this instance, the previous occupants of the indenting zone were apparent as diffuse outlines.

Results and Discussion. The comparison to be made here is an indirect one. What is actually being compared is the hardness values derived from the same specimen, in the same area of test, at the same indenter loads, but at 24 hours after standard polishing (P_1) versus

Figure 14.

TIME INTERVAL BETWEEN POLISHING & MICRO-INDENTATION HARDNESS DETERMINATION

Indenter Load in Grams	KNOOP HARDNESS MEASUREMENT		Average
	WITHIN 45 MINUTES AFTER	24 HOURS AFTER	
10	77.16	83.15	80.16
300	62.14	81.17	71.66
Average	69.65	82.16	

shortly after heating with repolishing (P_4). The administration of the polishing treatment was, of necessity, non-random. Regardless of how tenuous this argument may appear, preliminary experimentation substantiated by pertinent statistical decision made earlier in this dissertation support the view that differences as suggested from Fig. 14 cannot reasonably be attributed to the effect of immersion heating followed by standard repolishing. The reader is referred to Fig. 13 in which the micro-indentation hardness values derived from polishing procedures P_1 and P_4 are listed. The application of these polishing procedures did not result in a significant difference in hardness values.

No positive stance is taken in regard to the values presented in Fig. 14 due to the lack of precise separation of variables involved. However, it is felt that these results strongly suggest a need for further investigation into the effect of the post-polishing and/or post-heating and repolishing time on the indentation hardness determination of dental amalgam. That such a difference might exist should come as no surprise if one accepts the bound state of mercury as contributing to the overall hardness measurement of amalgam (33, 46). The possibility which is of interest here is the likelihood of such a time, surface hardness, and strength relationship operating after thermogenic polishing as is expected to follow the original setting reaction (46). This would tend to bear heavily upon the immediate effects of thermogenic polishing rather than the delayed effects observed in earlier tests.

Micro-Surface Characteristics

Thus far, deformation of the amalgam surface has been mentioned principally in relation to hardness testing. In this context the surface possesses an ability to resit deformation. This resistance is

also important to another deforming procedure, i.e., polishing, and has been shown to depend upon the relative amount of mercury and possibly its freedom in the region of deformation. Hence, it appears reasonable to look beyond the measurement and inquire into the effectiveness of this resistance to polishes used in different parts of this investigation. The following is the result of such an inquiry and describes the appearance of polished amalgams previously identified.

It may be recalled that the surface of these amalgams lacked structural detail following all polishing procedures. The presence and identity of certain of these surface structures has already been discussed as a result of observing corrosion. The questions which are of interest here are: is the obscurity of surface detail due to deformation or surface flow produced by polishing and, if so, does this flow have any bearing on the appearance of details identified by corrosion or etching? It is this prospect of surface flow which will be considered. Some affirmative evidence is available for the latter question (34).

Macroscopic Observations. During preliminary surface preparation frequent observations at 10 to 15 X magnification suggested that distortions of surface pits or voids was occurring in the direction of abrasion. Since there was no way of predicting the shape and presence of these pits upon the surface, it was difficult to rely on these changes in contour due to polishing. Because of this difficulty it was decided to employ a surface defect of known architectural properties which could be altered by dental polishing. A 100 gram Knoop indentation measuring approximately 100 x 16 x 4μ was selected for this purpose.

Microscopic Observations. Standard repolishing of previously indented regular and mercury-rich amalgam surfaces resulted in a partial

Plate 1

Fig. 15. Knoop Indentation on Regular Amalgam Surface before
Standard Polish.

Fig. 16. Knoop Indentation on Regular Amalgam Surface after
Standard Polish.

Figure 6 15

Figure 9 16

or complete filling-in of previous indentations. An example of the same intact and partially filled indentation in regular amalgam surface at 600 X magnification is shown in Figs. 15 and 16. This standard polishing procedure could be broken down into smaller stages of time. In this manner the edge of the indentation, in advance of the abrasive, was seen to move toward its opposite margin. During this movement and prior to filling, the internal detail of the indentation was observed to be free of polishing debris and sharply defined.

The appearance of these repolished indentations in regular amalgam did not seem to vary on re-examination after 24 hours. Mild etching and atmospheric corrosion revealed the presence of highly fragmented or eroded unreacted particles of alloy on the surface outlined by this repolished indentation. With the progress of the etching reaction, these alloy particles did not demonstrate a continuity with the body of sub-indentation amalgam. Etching of this area with 30% nitric acid revealed the general boundaries of the original indentation to a depth below the level of etching.

Similar observations of indentation filling were made on regular amalgam surfaces after applying polishing treatments P_2 and P_3. However, in the case of P_2, the completeness of filling was not quite as extensive as with applied abrasive. Nevertheless, the unsymmetric narrowing of the indentation was observed in the direction of prophylaxis cup rotation.

An amalgam specimen containing approximately 60% residual mercury was indented and repolished by the standard procedure. Figs. 17 and 18 demonstrate the complete filling-in of the indentation after repolishing. Fig. 19 shows the same indentation 24 hours laters. Very little

Plate 2

Fig. 17. Knoop Indentation on Mercury-Rich Amalgam Surface before
Standard Polish.

Fig. 18. Knoop Indentation on Mercury-Rich Amalgam Surface
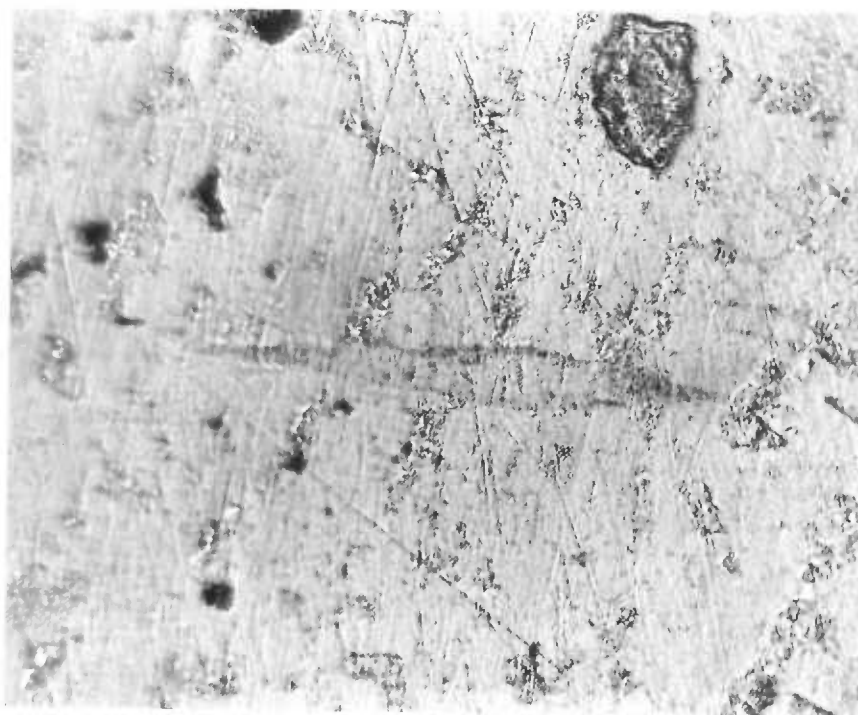Immediately after Standard Polish.

Figure 10 17

Figure 11 18

Figure 22|9

.Knoop indentation on mercury-rich amalgam surface 24-hours after standard polish (600 X magnification)

change is evidenced in the texture of the surface surrounding and within
the outline of the indentation after 24 hours. A slight amount of
isolated recrystallization on the surface is suggested.

Similar observations of indentation filling were made following the
application of P_2 and P_3 and the same stipulations regarding the perform-
ance of P_2 apply. Also, the same identification of filled indentation
contents, as given above, was made by etching and corrosion.

The next situation observed parallels that measured in the last
indentation test. Figs. 20 through 23 demonstrate the appearance of the
same indentation in a mercury-rich specimen over a 96-hour period follow-
ing the application of polishing procedure P_4 and indenting at 100 grams
load. The times given refer to the post-indentation interval of room
temperature storage. The general deterioration in definition of inden-
tation outline, texture, and measurable length is indicated. The
presence and growth of recrystallization of the surface and indentation
walls is evident. In the upper right-hand region of Figs. 22 and 23 is
suggested the development of visible scratches not so pronounced in
earlier viewing in Fig. 20.

Most change in appearance of surface and indentation is seen to
occur before two hours. Actually, the most striking changes were seen
within a few minutes after indentation but because of the time limita-
tions imposed by the photographic procedure, these changes are not
shown. Fig. 17 would depict rather closely the appearance of the sur-
face immediately after indentation. From Fig. 22, very little surface
change is noticeable after 24 hours.

Discussion of Observations. What has been gained from these and
other observations is the recognition of surface recrystallization with

Plate 3

Fig. 20. Knoop Indentation on Heated and Repolish, Mercury-Rich
Amalgam Surface 5 Minutes after Indentation.

Fig. 21. Knoop Indentation on Heated and Repolished, Mercury-Rich
Amalgam Surface 2 Hours after Indentation.
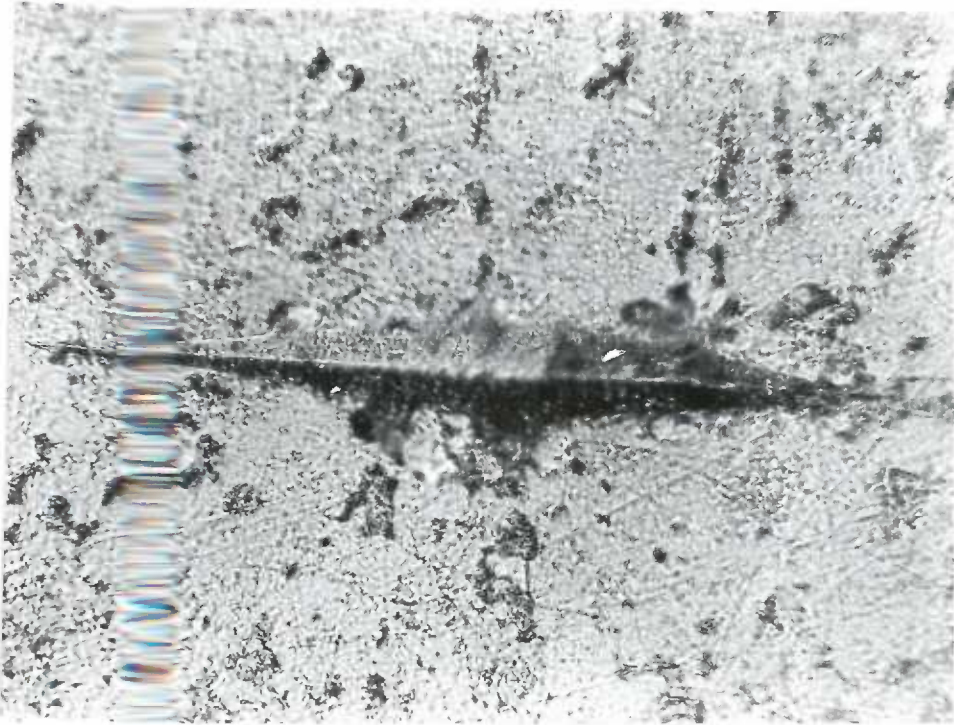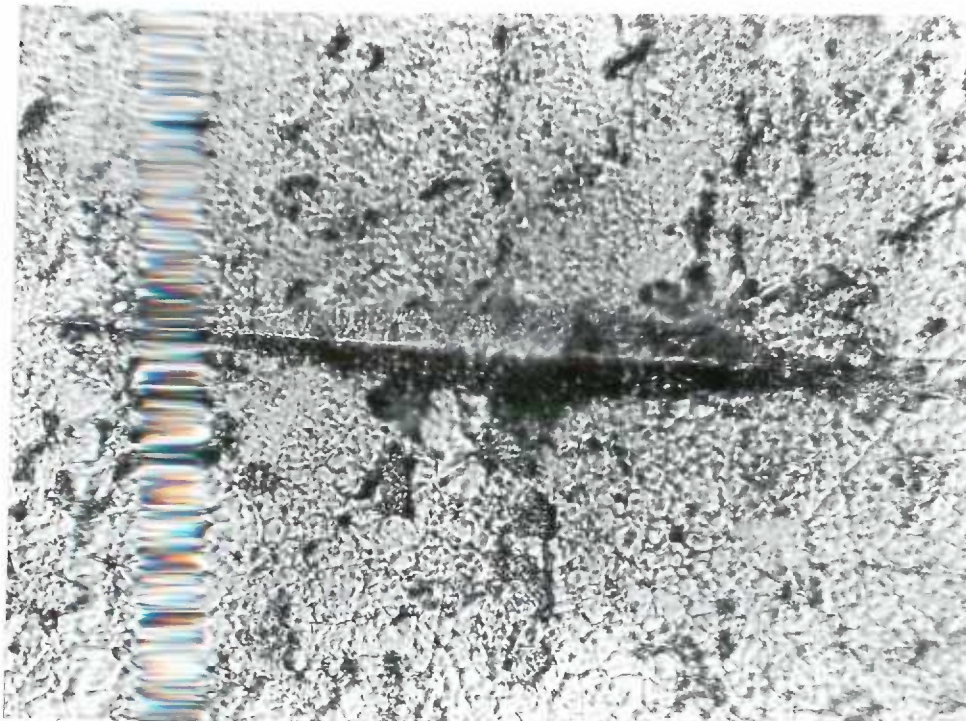
Figure 19 20

Figure 14 21

Plate 4

Fig. 22. Knoop Indentation on Heated and Repolished, Mercury-Rich Amalgam Surface 24 Hours after Indentation.

Fig. 23. Knoop Indentation on Heated and Repolished, Mercury-Rich Amalgam Surface 96 Hours after Indentation.
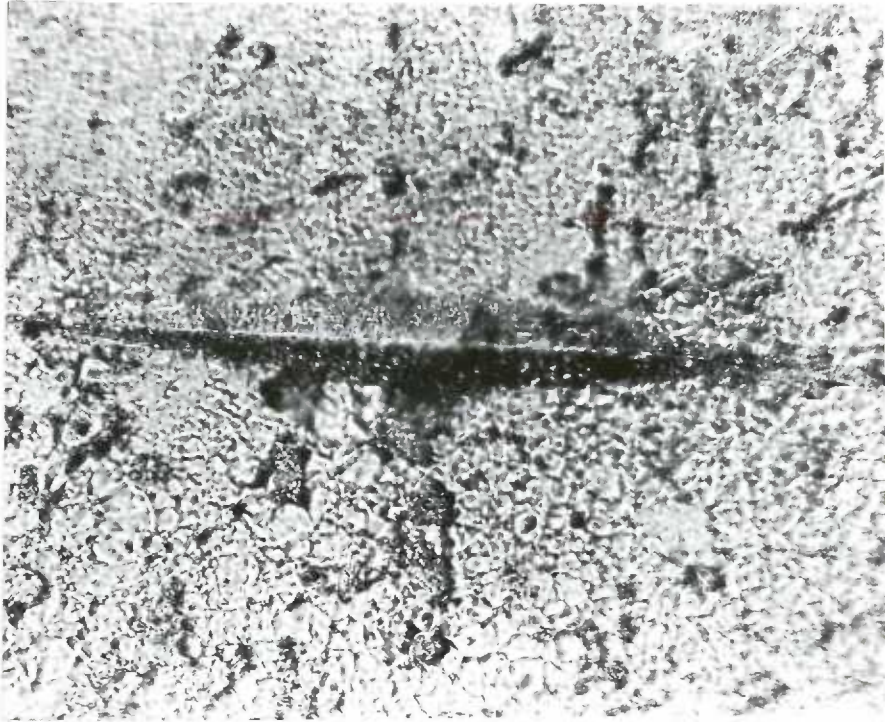
Figure 15 22

Figure 16 23
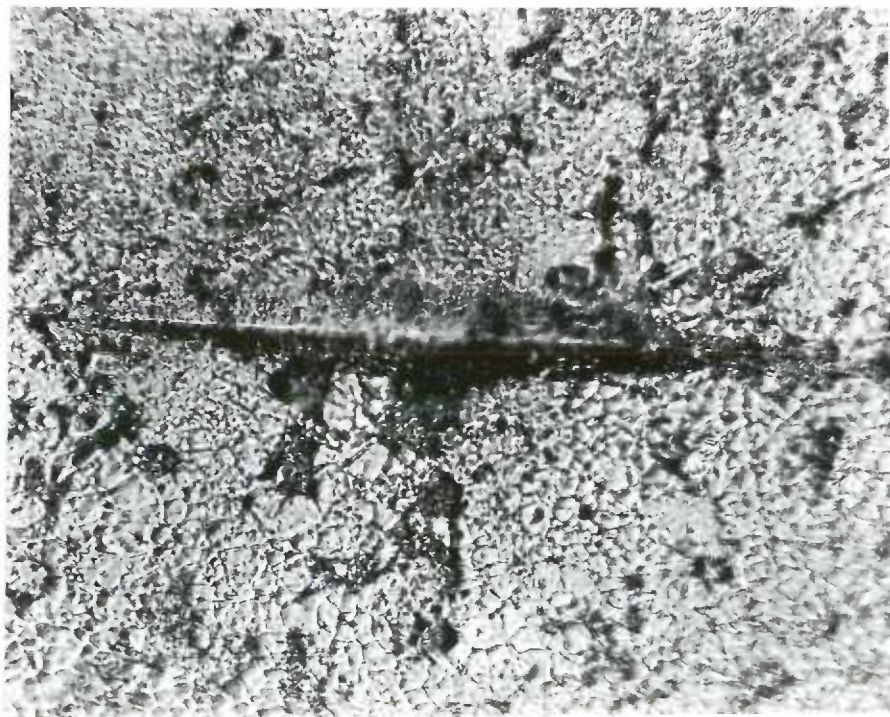
time, particularly after the application of heat to the amalgam. The time interval of observed surface change would appear to bear some relationship to the rebinding of free mercury. This interval might be expected to be influenced by the conditions of amalgam storage during observation (12, 43). The rapid change of surface texture makes the dramatic presentation of early scratch appearance and indentation outline deterioration difficult. The deterioration in indentation length appears to be one of shortening of definable terminals. Scratches which became apparent with the progress of recrystallization also tend to demonstrate the presence of flow of a freed mercury-rich phase after the procedure of heating and repolishing.

Standard polishing of regular and mercury-rich amalgam may effect some surface removal (47). Nevertheless, this procedure produces a filling-in of surface indentations with material possessing the identifying characteristics of amalgam. The most logical origin of this filling material is the amalgam surface adjoining the indentation. It may, therefore, be inferred that polishing by procedure P_1 results in a measure of surface deformation or flow of regular and mercury-rich amalgam which is not accountable exclusively to surface debris.

Abusive polishing procedure P_2 makes some contribution to surface flow but does not appear to be quite as capable as P_1 and P_3 in effecting indentation filling on regular and mercury-rich amalgam surfaces. The explanation for this behavior is not known. Perhaps this observation has something to do with the absence of abrasive in procedure P_2.

The importance of surface flow of amalgam to our understanding of clinical polishing is not clear, but there seems to be sufficient evidence to suspect its presence following the application of these

polishing procedures using the prophylaxis cup. The procedures P_1 and P_2 would seem to represent some practical extremes in the spectrum of thermogenic polishing (1). But, it is the observation of surface flow following what might be considered a very careful polishing treatment that may be of importance, since the use of this type of polishing, particularly within the confinements of the cervical and proximal regions of the tooth, may reflect the prevalence of surface flow in the clinical restoration.

In the instance above, the function of flow appears to be that of smoothing over small surface irregularities of amalgam (36). The durability and importance of this type of microscopic smoothing of amalgam, especially that approximating gingival tissues, is difficult to assess in terms of much larger surface voids observed with less desirable handling of the material (e.g., mercury-rich amalgam surfaces).

With respect to these surface voids, the expectation of their appearance (3) in inordinate numbers following the rebinding of freed mercury is not supported by observation in any portion of this experiment. An explanation of their obscurity, particularly where repolishing has following amalgam heating, may involve the presence of surface flow (36).

SUMMARY

The influence of an abusive and careful repolishing procedure upon
the surface properties of dental amalgam has been studied through the
measurement of micro-indentation hardness and transverse strength, and
from the observation of immersion corrosion. Certain statistically
significant but low order changes in test measurement have been recorded
after the application of abusive polishing treatment to the amalgam sur-
face. These changes have not been considered to reflect an important
depreciation of the clinical performance of this restorative material.
The gross observation of the overall extent of corrosion in two media has
not revealed any distinct difference between the amalgam surfaces pol-
ished by careful and abusive methods employed in this study. No defini-
tive clinical interpretation regarding the effect of abusive polishing
on corrosion has been offered.

A few of the background considerations of the processes of polish-
ing, heating, and corrosion of amalgam have been mentioned. The follow-
ing aspects of the phenomenon of polishing of dental amalgam have been
considered: (1) The release of bound mercury from the surface of amal-
gam have been observed to occur above 177° F.; (2) The abusive polishing
procedure used has been arranged to provide surface temperatures in
excess of that required to release bound mercury from amalgam; (3) What
has appeared as structural components of dental amalgam from early
corrosion in saline, and identified by acid etching has provided the
basis of an observable difference between careful and abusive polishing

procedures in both corrosion media; and (4) The presence of surface flow has been suggested under circumstances prevalent in the polishing procedures (P_1, P_2, and P_3) used in this study.

These observations have suggested the possibility that the complex of what has been described as an abusive polishing procedure may embrace not only the release of bound mercury, but some flow and further solution of mercury in the unreacted phase of the amalgam surface.

The residual mercury content of amalgam has been shown to have an influence upon surface hardness. The time after thermogenic polishing, or mercury release, has been implicated as a factor in the hardness of amalgam containing high mercury content. In the latter situation, there is some indication that the factor of time may have a bearing upon the immediate effects of thermogenic polishing of amalgam with this composition.

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APPENDIX I

Review of Literature

Polishing. The term polishing is here intended to describe the latter stage of smoothing and finishing of the metal with a fine abrasive grit material to yield a surface of high reflective lustre which is free of scratches and pits. The process by which such a metallic surface is obtained is not exclusively dependent upon employing the proper application or choice of abrasive material, but is, in many instances, affected by the manner in which preceding stages of abrasion and grinding are conducted (47). The distinction between the processes of abrasion and polishing is essentially a matter of the degree to which the following may be present:

(1) much finer scratches and shallower deformation in sub-surface layers,

(2) looser support or fixation of abrasive grit in polishing than in abrasion (24), and

(3) the presence, amount or absence of layers of fragmented and flowed material on the surface (47).

Two theories regarding metallographic effects of polishing metallic surfaces are currently reported (47). One theory does not recognize any difference between abrasive grinding and polishing, the latter being nothing more than the former carried beyond the microscopic limit (24). A second theory considers that no further metal is removed from the surface while polishing and that, under high local pressure of the polishing

grit and the temperature created by the polishing carrier, there occurs
a surface melting and plastic flow of the surface metal (48). Under
these conditions, the metallic surface assumes either an amorphous struc-
ture described as the "Beilby Layer" or a structure of exceedingly
minute crystallites in a highly disordered arrangement (47). Which of
these theories presents the truest picture of what is occurring at the
metal surface is not yet determined.

The distinction between these two types of resultant surface struc-
tures is of primary importance here only to the extent that it presents
the possibility of increased surface internal energy in these flowed
and deformed regions of the surface and sub-surface, respectively (48),
altered resistance to corrosion (5), and momentary frictional temperature
offered to the body of the polished object which may reach 1000° C. at
the polishing-grit-metal contact only a few angstroms in width (47).

The effect of alteration by the induction of superficial shear
deformation and plastic flow has been described as endowing the metal
surface with properties which are different from the original crystalline
character. The order of depth has been studied for various metals and
has given rise to speculation regarding the effect of abrasive surface
preparation on the anomalously high results obtained in low-load hard-
ness tests (24). In general, the depth of deformation resulting from
previous polishing or abrasion treatment of the surface is held to vary
inversely with the hardness of the material (24) or directly with the
work hardening propensities of the metal (48). The cutting rate during
mechanical polishing has been shown to vary, in a general fashion, with
the melting point of the material polished (24). These observations
would indicate a lesser degree of difficulty in obtaining a more nearly

strain-free polished surface in a metal of low fusion point and substantial hardness. Brittle materials such as bismuth have been shown to exhibit flow deformation when subjected to the local pressures of polishing (47). The surface effects of industrial polishing more closely approach those of abrasion than in metallographic polishing procedures (24). The implication which can be drawn about induced surface deformation appears to be focused not so much on the materials used but the care employed within each procedural sequence of polishing. This would certainly have some bearing upon polishing under conditions which, of necessity, cannot approach metallographic standards, viz., in the mouth.

The metallographic changes which occur at the polished amalgam surface are not well understood. Smith, et al. (34), when studying the micro-structure of amalgam, concluded that the relatively low temperature at which amalgam begins to melt (60 to 65° C.) and the high mercury content of the hardened material, had contributed to the difficulty of obtaining the micro-structure. The implication is that flow of the amalgam surface may occur during polishing (135-36). During the process of etching the polished amalgam surface with 30% HNO_3, the etching time employed suggested that the thickness of the disturbed layer of the surface was greater for higher mercury content (51%) of the amalgam (34).

Release of Bound Mercury. Several references to mercury being released from hardened amalgam are prevalent in the dental literature (5-13). Black was among the most prominent early observers of the phenomenon of amalgam heating when he noted a volumetric expansion and changes in the appearance of the hardened metal subjected to boiling water (7). Others (8) have described changes in dental amalgam in the temperature range of 65 to 100° C., depending upon compositional

factors. In particular, a "transition point" for hardened amalgam was shown to exist in the region of 70 to 80° C.. This region has been characterized by Gray (9-10) as possessing:

(1) A marked and irreversible absorption of heat at about 77° C..

(2) A sharp minimum in linear thermal expansivity near 75° C..

(3) A lessening of the above temperatures with successive heatings.

(4) A marked lowering of the compressive strength of the amalgam at approximately 72° C..

(5) An influence upon the magnitude of the transition temperature, heat absorption, and expansion in alloys containing zinc.

In addition, he observed that hardened amalgams compounded from zinc-containing alloys evidenced what appeared to be a surface mercurial exudate near the transition region. The surface crystals, felt to belong to the hexagonal or orthorhombic system, were seen to form on the heated amalgam surface at a crystallization temperature near 62° C.. The amount and size of mercury droplet formation and surface crystalline growth, respectively, was influenced by the presence of zinc and the amount of mercury incorporated in the formulation of the final hardened amalgam (10).

The direction of dimensional change of amalgam experienced on heating has been substantiated and the presence of uncombined mercury in the hardened mass cited as the causative agent (43). Mitchell, et al., have indicated from x-ray diffraction examination that mercury becomes freed from its crystalline affiliation and diffuses through hardened amalgam only when the latter is subject to heating to 65° C. or by polishing procedures producing excessive heat (12). Such uncombined mercury is seen to disappear or recombine with other phases present in amalgam when

aged at 37° C. (12). The disappearance of the original alloy particles and the loss of distinction between the reaction products in the silver-mercury and tin-mercury phases has been attributed to the heating of amalgam to 80° C. (38).

The mechanism of recombination of free mercury in hardened amalgam is presently unknown. Moreover, the major site of recombination has not been established. The observation of surface crystalline growth and change of amalgam appearance following heating leads one to suspect that some portion of this recombination runs near the surface.

Corrosion. The destruction of metal or alloy, as manifested by contour change, weight gain or loss, film or scale formation, loss of surface reflectivity, production of pit, fissure, or crevice attack, etc., has been described as corrosion (37-p. 1-731). This process is believed to be brought about by a chemical change, or physical dissolution (37-p. 2). Experimental evidence points to the conclusion that an electrochemical process of corrosion of most metals is involved under conditions of exposure to an aqueous environment (39). The inertness of metals in the body has been attributed to their (metals) electrochemical stability. Also, it has been shown that in vitro electrochemical measurement of metals in physiologic saline provides a usable criterion of predictable tissue reaction (49). The oral anatomy, biochemistry, and physiology place certain requisites upon the fluid, gaseous, and solid environment which must be shared by the dental restoration. These circumstances suggest that corrosion deterioration of this restoration would be of a type of partial or full immersion in an electrolyte bath, as described by Evans (37-p. 87). The point above is offered to consider the term "tarnish" which has been described as essentially a dry

corrosion process and which, in general, has been felt to occur below some critical value of relative humidity, usually 70% (37-p. 483).

The restoration, once placed in the tooth, must perform its useful function in an environment highly conducive to corrosion attack. The smooth polished restoration is felt to be rendered less prone to corrosion, and to the accumulation of debris about it which might further this process (4-p. 341). Aside from the obvious detracting esthetic results of corrosion, the extension of this process may ultimately lead to changes in the physical properties of the restoration occasioned by its weakening and failure (4-p. 341, 22).

The exact mechanism and the predominant controlling factors in the process of corrosion of dental amalgam are not known through reports available to this author. Moreover, no test has been advanced which affords a valid basis of predicting the rate and distribution of corrosion of this material in the environment of the oral cavity (23). The general phenomenon of corrosion attack is said to depend upon numerous factors which, in the main, seldom act independently of one another. Instead, the dependency of action is based upon a complex interaction of factors (50). From this consideration and for the reason that such a complex interaction is not known for oral conditions, a lengthy discussion of the many factors known to affect the general process of corrosion, with pauses to consider the possibility of their oral analogy, will not be attempted.

What is known or suspected regarding this phenomenon in dental amalgam will follow. Dental amalgam has been observed to suffer corrosion attack in the mouth (4-p. 345-8, 40). Electric currents have been measured in the mouth containing amalgam restorations (51) and in

in vitro electrolyte solutions containing amalgam–amalgam and amalgam–gold couples (22, 52). Polished amalgam restorations do not corrode as rapidly as unpolished ones in the mouth (4-p. 345-8). Presumably soluble or mobile products of oral corrosion containing traces of tin, copper, silver, and zinc have been identified along dentinal walls of amalgam restorations (22, 53). The major product of in vitro corrosion in salt solution has been identified as tin (22). The products of oral tarnish are felt to include either Ag_2S or an $Ag(Hg)S_x$ complex (21). The major products of in vitro tarnish in Na_2S solution have been identified as mercuric sulfide along with lesser amounts of silver and tin compounds (21). Solutions of NaCl, Na_2S, H_2O_2, and synthetic saliva have been credited with providing a suitable environment for corrosion to occur in vitro within a reasonable period of time (21). Solutions of acid and base and anodic and cathodic polarity (22) of the amalgam specimen have been considered to influence in vitro corrosion (54).

APPENDIX II

Justification for Choice of Tests
and Polishing Procedures

Since the effects of polishing are most likely to be found near the surface, it seems reasonable to employ those tests which either directly or indirectly assess the integrity of this region. The tests chosen for this purpose are Knoop micro-indentation hardness, transverse strength, and the observation of corrosion in electrolyte solutions.

The clinical significance of these tests exists in their ability to serve as predictors of the structural integrity of a functioning restoration in the oral environment. Although the valid predictability of these tests is yet to be established, there is some evidence to suggest their usefulness in this regard.

What has been observed as departures from successful clinical performance of amalgam restorations is surface roughening, marginal deterioration, corrosion, and gross fracture (40). In a clinical study (40), the compressive strength of amalgam has been cited as a possible index for evaluating marginal deterioration and corrosion in amalgam restorative failures. In this same study, excess residual mercury content of amalgam restorations has been associated with surface and marginal deterioration. Indentation hardness and mercury content have been shown to be related to the compressive strength of amalgam (46). Transverse strength has been reported to bear a relationship to residual mercury content of amalgam similar to that for compressive and tensile

strength (25). On the strength of these observations, it is felt that the import of alterations of these tested properties of the amalgam surface may have clinical implication.

Knoop Hardness Test. The surface hardness of a material, like dental amalgam, is a difficult property to define. It is thought that, in general, the values of hardness derived from restorative materials are indicative of a quality such as resistance to wear (14-15), strength characteristics (14, 16), or ability to be adapted or deformed (14, 16-17), rather than the unique or exclusive property of hardness. However, it is generally held that the surface hardness of a material can be measured in terms of its resistance to permanent indentation and deformation. It is this resistance which is perhaps the most commonly held concept of hard and soft substances (14).

Qualities such as resistance to wear and deformation and possession of the general characteristic of strength are seemingly desirable attributes of a clinical restoration. Deterioration of the quality of occlusal wear is cited as contributing to the failure of amalgam restorations under conditions of strict clinical examination (40). The possibility of indentation hardness having a bearing on the clinical performance of amalgam restorations has been noted (17-18). Thus, it is reasonable to assume that a decrease in hardness after polishing may indicate a reduction in the clinical performance of a restoration similarly polished.

This test subjects the material examined to a complex system of stresses in the process of surface penetration. The index of hardness (Knoop hardness number) represents the ratio of the load required to obtain the indentation of the surface divided by the unrecovered projected

area of indentation. By the use of light loads the depth of indentation can be limited to reflect the resistance to indentation of a narrow region of the surface boundary. The test is employed here to ascertain the effect of thermogenic frictional polishing on the property of resistance to indentation of amalgam.

Transverse or Bending Strength Test. The property of transverse strength is essentially the ability of a material, freely supported at each end, to resist rupture due to a centrally applied static load (4-p. 23). The test subjects the material to a combination of shear, tensile, and compressive stresses which may be likened to that believed to cause fracture of occlusal amalgam margins (20) or under occlusal flexure of the restoration due to the forces of mastication and dentinal deformation (55). Amalgam behaves as a brittle material and as such is particularly susceptible to rupture when subjected to tensile stress (19). It is felt that any change of the amalgam surface after polishing which would initiate an early fracture under tension may be detected by this test.

The test of transverse strength has been held to provide a better measure of amalgam failure than is provided by the compressive strength test (20). On the basis of these assertions, any major change in this property may be looked upon as suggestive of adverse amalgam performance in the restoration.

The test is employed here to determine if thermogenic polishing of the amalgam surface results in a change in the ability of the material to resist transverse loading.

Observation of Corrosion. The ability of solutions of NaCl and Na_2S, of comparable molar strength, to produce surface changes in dental

amalgam (i.e., eppearance, contour, light reflection) represents no immediate or implied parallel with the expected reactions of this material in the oral environment. The basis of our present inability to synthesize a useful oral analogue is predicated upon the variety of changes inherent in the environment itself. Some of the factors which are known to affect the rate and distribution of corrosion attack, in general, and are likewise known to exhibit a characteristic state of flux within the oral environment are: (1) pH, (2) crystalloid and colloid species, concentration, and associated charge (56-57), (3) oxygen gradients due to physical solution and convection (58), and (4) temperature in the fluid and vapor environment surrounding the restoration (50, 59-60). The amalgam restoration, existing under the above conditions, may contribute other factors which are considered to influence the rate and extent of corrosion. Some of these are: (1) inhomogeneity of structure within the surface, within the body, and between surface and body (36, 61), (2) localized residual and induced stress, (3) impurities within the crystalline structure, especially when concentrated at grain boundaries, (4) surface finish and the presence of surface pits, fissures, and other deformations, and (5) chemical nature of the material (4-p. 345-8, 62).

The test of immersion is intended to allow the observation of the in vitro effect of thermogenic polishing upon the amalgam surface, under conditions reported to produce corrosive attack (21).

Polishing Procedures Employed. The polishing procedures to be applied to amalgam in this investigation consist of a standard polish, an abusive polish, and an abusive polish followed by a standard repolish.

The clinical parallel of these polishing procedures may be

considered as exaggerations of common practice. What constitutes common clinical polishing practice is not readily definable, nor is it the intent of this study to explore the nuances of this topic. Instead, it is proposed to extract from this spectrum of clinical activity the methods and materials of a not uncommon polishing procedure capable of being performed in the mouth.

The standard polishing and repolishing treatments are felt to be representative of a class of careful polishing procedures which may be employed in the mouth. The factors of application of load, rotary speed and duration are to be restricted to minimal values. The rotary speed of abrasive application is believed to simulate the lowest consistent speed equal to, or less than, that which may be obtained from many presently used dental units. The re-employement of this careful polishing procedure after one of thermogenic abuse represents what is believed to be one practical and readily applied method of attempting to negate the effects on the amalgam surface of previous frictional polishing.

The practice of applying a dry abrasive powder, i.e., amalgloss, with a prophylaxis cup and allowing the abrasive to be depleted from the area of polish has been examined (1). The powder is seen to rapidly leave the immediate area of the abrasive carrier (viz., prophylaxis cup) and, unless replenished, the carrier exerts an increasing amount of frictional activity upon the surface of the restorative material. Similar conditions are seen to occur when the initially wet abrasive slurry is allowed to dry, presumably by the absorption of energy, and then becomes dissipated through further polishing. It is the protracted application of the prophylaxis cup after the loss of the abrasive which is felt to represent an abusive thermal polishing practice.

The specifications descriptive to this procedure appear to represent one of many possible combinations of factors which was chosen because of either the resemblance to conditions of preliminary observation, the convenience of altering standard conditions, or as a result of an approach to standardization.

APPENDIX III

Preliminary Observations on the Temperatures
of Polishing and of Mercury Release

When considering the objectives of this investigation, it was believed important to examine some of the factors involved in the association between amalgam surface temperature changes and the visible effects issuing from such heating of a current dental amalgam. The questions asked were:

(1) At what surface temperatures do currently available amalgams release mercury?

(2) Under what conditions of polishing, having clinical analogy, can the above temperatures of the surface be reached and controlled?

(3) By what visible signs can the results of the above manner of polishing be detected?

A DC voltage stabilized brass thermode was constructed having a 1 mm.^{2} heating surface. The exposed metal portion of the thermode, other than the surface contacting the restorative material, was covered with asbestos ribbon to minimize heat loss. This heating surface was determined to be linear in temperature rise at $2.41 \neq .05^{\circ}$ F./second over a 90-second heating period, as monitored by thermocouple.

Polished class V amalgam restorations were placed in ivorine teeth. A $\frac{1}{2}$ round bur hole was placed through the lingual of each tooth and very nearly through the body of the amalgam restoration. The remaining amalgam thickness was measured to be from 0.05 to 0.08 mm.. A chromel-alumel

HT micro-miniature[1] insulated thermocouple was placed to the full depth of the bur hole and fixed to maintain its relationship to the restoration. The thermode was placed against the restoration surface in such a manner as to have one corner of the thermode directly over the thermocouple tip (T in Fig. 24).

Small crystals of tempilaq°, (a temperature-indicating paint possessing an accuracy to $\nleq$ 1% of rated value and a response time in the order of micro-seconds), were placed close to the thermode and thermocouple tip. The temperature values of these crystals were 138, 150, 163, 175 and 200° F.. The placement of thermode and tempilaq° crystals was accomplished under 25 X magnification and was arranged as shown in Fig. 24. The purpose of this arrangement was to provide as equidistant a conduction pathway, between thermode and crystals, as was possible by manual placement. The lower-melting crystals rated at 138 and 150° F. were placed at A, B, and C as shown in Fig. 24. The tempilaq° crystals were then considered to melt when the metal surface temperature in the immediate area of the crystal reached the rated value of this temperature indicator.

Using the above thermocouple and a Leeds-Northrup Speedomax[2] potentiometer to detect and record the temperature of the metal at a distance of 0.05 to 0.08 mm. beneath the surface, the events of surface crystal melting, subsurface temperature rise above room temperature, and the release of surface mercury of the heated metal could be coordinated. The potentiometer calibration and reference function temperature

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[1] High Temperature Instruments Corporation, Philadelphia, Pennsylvania.

[2] Leeds-Northrup Company, Philadelphia, Pennsylvania.

Figure 24.

ARRANGEMENT OF THERMOCOUPLE,
TEMPILAC° CRYSTALS & HEATING SURFACE


A
B° T°
C °
HEATER 1 mm²
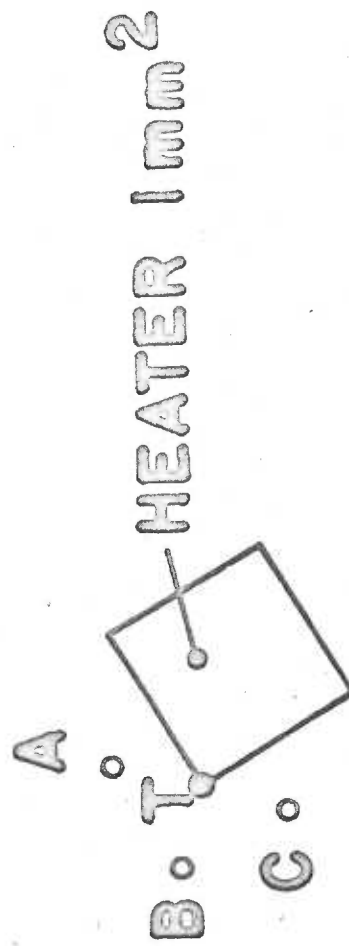
stabilization employed for these measurements has been described elsewhere (1).

A series of seven synchronous observations were obtained to provide an estimate of the surface temperature required to effect the release of mercury from hardened polished amalgam. The temperature at which mercury was observed to issue from the surface was $177 \neq 2^{\circ}$ F., as revealed by thermocouple measurement over an average heating period of $58 \neq 2$ seconds. This temperature value represents the temperature increase of metal subsurface plus that of room temperature reference. The difference between this value and the temperature standard provided by the crystals was of the order of 2 to 4° F. increase. This latter disparity, within the temperature range of crystals used, diminished at 175° F., and was felt to represent, in general, the effect of slight differences in conduction pathways and/or time delays in synchronization between the visual appreciation and manual response in indicating the presence of a mercury droplet on the metal surface. In view of this time lag and the surface heat distribution, the employment of the 175° F. crystals to monitor surface temperatures associated with mercury release appeared to be justified for periods of greater caloric uptake.

Fig. 25 illustrates a composite of the linear heating curves obtained and demonstrates a remission of temperature rise near the point of mercury's entrance into the picture. Such an observation would be consistent with the manifestation of energy absorption during the release of mercury as described by Gray (9). This dip was present to varying degrees in all recordings and on occasion was seen to occur coincidentally with a mercury droplet rising from the amalgam surface while lifting a tempilaq$^{\circ}$ crystal in the act of melting.

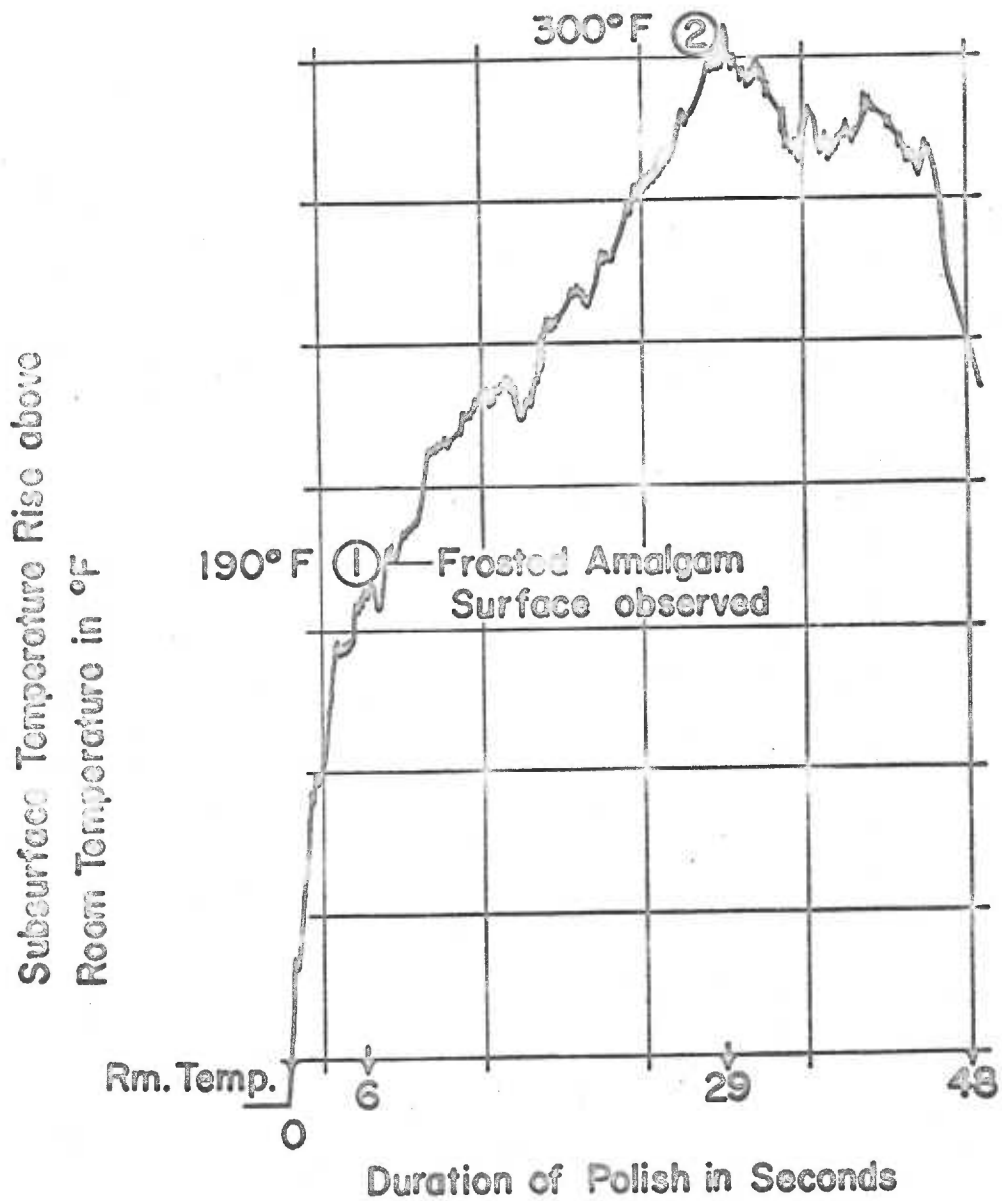LINEAR HEATING OF CL-V AMALGAM SURFACE

Figure 25.
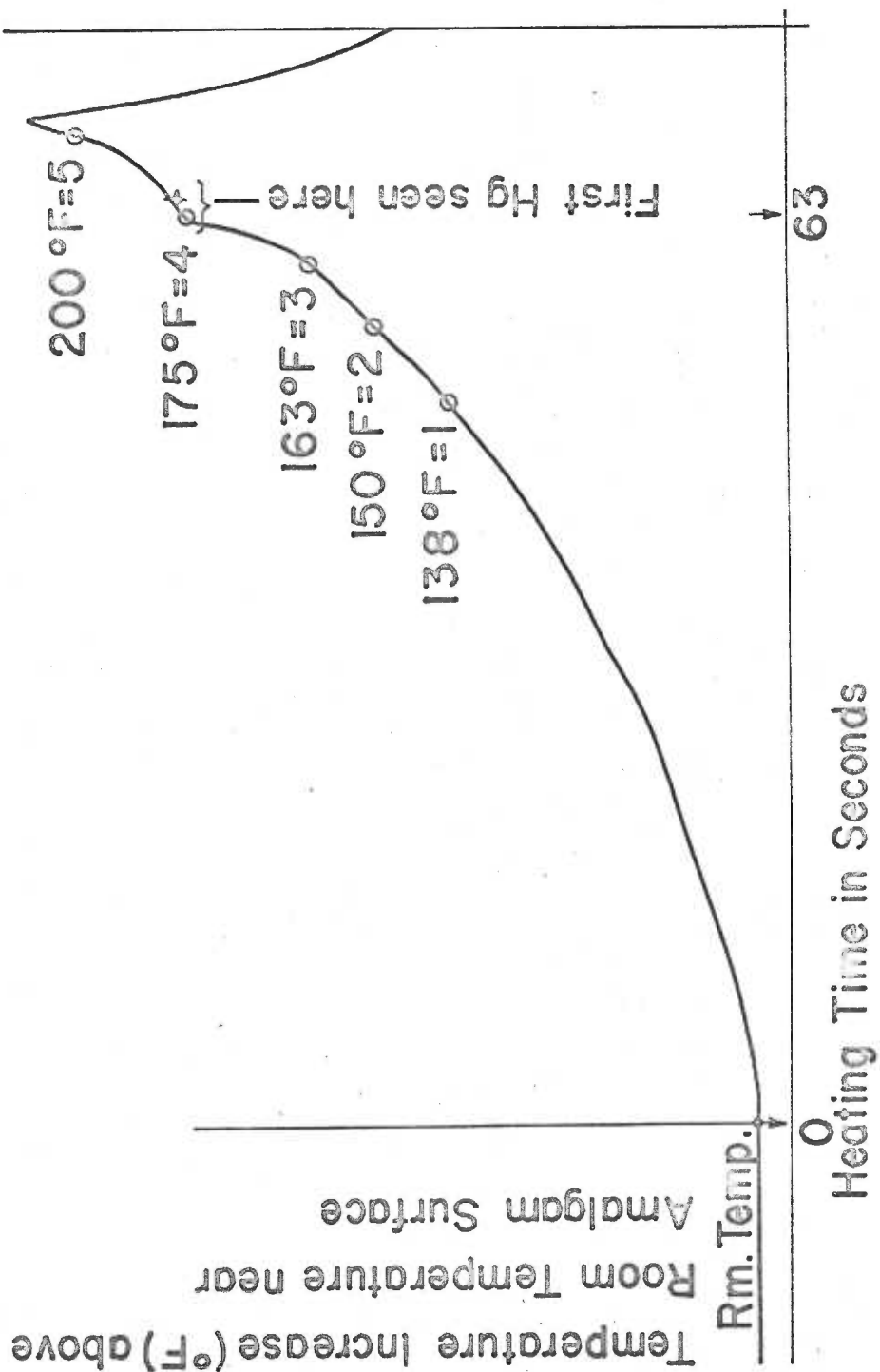
On cooling, the entire surface of the amalgam assumed a dulled appearance, different in lustre from the original.

Additional class V amalgams were treated in a like manner regarding thermocouple placement and subject to frictional polishing activity of a prophylaxis cup. Fig. 26 demonstrates the increase in temperature at 0.10 mm. beneath the amalgam surface when the prophylaxis cup was applied to the metal surface, over a 20 to 30 second period, at an application load of 8 to 16 ounces, and at about 30,000 r.p.m.. The resultant amalgam surface, on cooling, presented a dull, frosty, pebbled appearance unlike the original.

A series of 10 recordings at six to eight ounce load application of the prophylaxis cup against the buccolingual profile specimen shown in Fig. 5 revealed that, at a loaded rotary speed of 10,500 $\neq$ 100 r.p.m., a polishing period of 13.3 $\neq$ 1.3 seconds was required for the amalgam surface to uniformly exceed 175° F.. In this case, the temperature excess depended upon the time lag in observation of crystal melting and the thermal distribution about the peripherally located crystal. Both conditions would provide sufficient insurance that the temperature of mercury release was being attained.

The degree of uniformity of heating was determined by the melting of four symmetrically located tempilaq° crystals at the edge of the specimen. Rotary cycling of the prophylaxis cup at near normal attitude to the specimen produced nearly simultaneous melting of the crystals in the time interval considered here. From this observation, and due to the difficulty of observing four events in near succession, it was felt that two crystals monitoring normal conduction paths might serve equally well.

Figure 26.

300°F ②

Subsurface Temperature Rise above
Room Temperature in °F

190°F ① — Frosted Amalgam
Surface observed

Rm. Temp.

0   6   29   48

Duration of Polish in Seconds

TEMPERATURE INCREASE NEAR SURFACE
OF POLISHED CL-V RESTORATION

Again, on cooling, the surface of this amalgam specimens presented a uniformly frosty surface.

Conclusions based upon these preliminary observations of the conditions for the release of mercury are:

(1) that release occurs at a surface temperature of amalgam near 177° F. (80.5° C.).

(2) that polishing in a thermally abusive manner, capable of being performed in the mouth, can result in temperature increases at the amalgam surface in excess of that attendant mercury release from set amalgam.

(3) that the application of this polish using just the prophylaxis cup can be controlled, for specified conditions of load, time interval and speed of polishing, to effect mercury release from the amalgam surface.

(4) that the amalgam surface, having been heated or polished in a manner to release of mercury and then allowed to cool, evidenced a frosty appearance different from the original.

APPENDIX IV

Tables of Analysis of Variance

The tables of components of the test of analysis of variance compiled from the previous test data are listed on the following pages.

ANALYSIS OF VARIANCE OF
MICRO-INDENTATION HARDNESS

Table I.

SOURCE OF VARIATION	DEGREES OF FREEDOM	SUMS OF SQUARES	MEAN SQUARES	F	λ_1/λ_2	$F_{.05}$
Among Polishing Procedures (P_1, P_2 & R_3)	2	6,720.14	3,360.07	6.463	$2/205$	2.995
P_1 versus P_2	1	2,082.25	2,082.25	4.006	$1/205$	3.842
P_2 versus P_3	1	1,308.74	1,308.74	2.518	—	—
Among Indenter Loads	4	3,804.43	951.11	1.829	$4/205$	2.372
Polish × Load Interaction	8	6,408.15	801.02	1.540	$9/205$	1.938
ERROR	285	148,146.91	519.81			
Sampling	1200	676,931.40	564.11			
Total	1499	842,011.05	561.71			

Table 2.

ANALYSIS OF VARIANCE OF TRANSVERSE STRENGTH

SOURCE OF VARIATION	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARES	F	λ_1/λ_2	$F_{.05}$
Among Polishing Procedures (P_1,P_2&P_3)	13,218,272	2	6,609,613	5.3498	2/30	3.15
P_1 versus P_2	8,372,322	1	8,372,322	6.7766	1/60	4.00
P_2 versus P_3	211,296	1	211,296	0.1710	—	—
Daily Output	5,760,655	2	2,880,327	2.3313	2/58	3.15
ERROR	71,644,381	58	1,235,479			
Total	100,623,263	62				

ANALYSIS OF VARIANCE OF MICRO-INDENTATION HARDNESS

Table 3.

SOURCE OF VARIATION	SUMS OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARES	F	λ_1/λ_2	$F_{.05}$
Among Specimen	214,463.720	11	19,496.702	11.746	11/8	3.3156
Among Hg/Alloy Ratios	197,998.151	1	197,998.151	119.291	1/8	5.3177
Among Polishes (P_1 & P_4)	3,165.582	1	3,165.582	1.907	—	—
Hg/Alloy Ratio × Polishing	21.669	1	21.669	0.0130	—	—
ERROR (I)	13,278.318	8	1,659.790			
Among Indenter Areas	31.793	2	15.897	0.069	2/18	3.5546
Area × Hg/Alloy Ratios	175.957	2	87.978	0.3835	—	—
Area × Polishing	63.723	2	31.862	0.1389	—	—
ERROR (II)	4,128.723	18	229.369			
Among Indenter Loads	1,556.776	1	1,556.776	0.8989	1/25	4.2417
Load × Hg/Alloy Ratio	13,943.907	1	13,943.907	8.0513	—	—
Load × Polishing	3,606.359	1	3,606.359	2.0823	—	—
Load × Area	122.972	2	61.486	0.0355	2/25	3.3852
ERROR (III)	43,297.184	25	1731.887			
Sampling	1,446,756.675	1,380	1,048.37.			
Total	1,732,548.219	1,439	1,203.5.			