A STUDY OF PROPERTIES OF COHESIVE GOLD RESTORATIVE MATERIALS

by

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A Thesis

Presented to the Department of Dental Materials and the Department of Operative Dentistry and to the Graduate Education Committee of the University of Oregon Dental School in partial fulfillment of the requirements for the degree of Master of Science June, 1964

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INTRODUCTION

Among the several types of cohesive gold currently being used by the dental profession are some recently introduced products. Interest in compacted gold as a restorative material has been revived; and, presumably, advertising overtures made by the manufacturers of these new products are partially responsible for the aroused attention of the dentist.

The purpose of this investigation was to examine and compare types of cohesive gold and the methods by which they are inserted as dental restorations. The investigator believes that this study will provide information that can be used by the dentist for thoughtful selection of a material to satisfy a particular need. Also, the information may prove to be helpful to the dental instructor and to the dental student, and ultimately to the dental patient.

The research activities of some of the early dental scientists (particularly, G. V. Black and R. W. Rule) were intuitive and inspiring. Much of the work, following their experiments, suffers by comparison. Even though finer measuring tools and analytical techniques now exist, helpful, substantiated conclusions about cohesive gold are something less than abundant.

It was felt that a new approach to the problem, with particular emphasis on control of variables and statistical analysis, would provide needed information.

OBJECT OF STUDY

Five types of cohesive gold were condensed, using three condensation methods. A steel mold, described below, was devised as a simulated cavity and all samples were prepared in one mold. The prepared samples were then subjected to tests to compare hardness, strength, and density. In addition, the time required to prepare each sample was considered.

A list of the materials selected for testing appears in Table 1.

TABLE 1
MATERIALS TESTED

Gold Type	Description	Manufacturer	Date Re ceived
Mat Gold	segments of spongy crystalline gold	Williams Gold Refining Co.	9-3-63
Goldent	pellets of powdered gold wrapped in gold foil	Morgan, Hastings & Co.	1-13-64
Biofil	clumps of powdered gold	B. L. Dental Co., Inc.	9-23-63
Gold Foil	pellets of rolled foil	Morgan, Hastings & Co.	9-10-63
Combination mat+gold foil	both are described above	see above	see above

The methods used for condensing the samples were:

- 1. Hand pressure a packing technique similar to that used for amalgam restorations.
- 2. Electromallet a technique using an instrument representative

of those mechanical devices delivering a series of blows to condense the gold into the cavity.

3. Combination of these two methods — wherein the bulk of the cavity was filled by hand pressure and the electromallet was used to pack the veneer or surface layer.

Care was taken to select those materials and condensing techniques that are used clinically. It is not common practice to use haphazardly all of the condensing methods for each type of material. In other words, a material is usually inserted in the cavity by a single method. However, by employing a factorial experimental design with two variables, it was felt additional information might be forthcoming. Perhaps a condensing method, considered to be less traumatic to the oral tissues, would be applicable for any of these materials. This information could remove some of the objection to the usage of compacted gold.

Basis for Tests

The principal desirable qualities of a filling material, according to Black (1), are: 1) indestructibility in mouth fluids, 2) adaptability to the cavity walls, 3) freedom from shrinkage or expansion, 4) resistance to attrition, 5) sustaining power against forces of mastication. Color, thermal conductivity, and convenience of manipulation were considered by Black to be of secondary importance. The properties tested for this study suggest an evaluation of dental restorative materials based on Black's primary considerations.

Transverse strength. It is not clear what values of strength are necessary in a compacted gold restoration. It is believed that many of these restorations are placed in areas of the tooth that are not

subjected to direct masticatory forces. Ferrier (2) classified 12,000 foil restorations as to their location on the tooth. A large percentage of these were gingival restorations (32%), and 37% were interproximal restorations in anterior teeth. It seems reasonable to believe that "edge-strength" requirements are present even in the absence of heavy masticatory forces. This edge strength (or bending strength) might be needed at the retentive projections and the marginal angles of a restoration.

An extensive discussion of stress analysis in a dental restoration is found in a paper by Mahler and Terkla (3). The following concepts are extracted from that study:

- 1. Transverse strength is bending strength.
- 2. Bending a beam (or restoration) produces stresses of three types: compressive, tensile, and shear.
- 3. Bending strength is related to the term "edge strength" an edge subjected to bending exhibits tensile, compressive, and shear stresses.
- 4. Any discontinuity within a structure will produce a very high stress in the region of the flaw this might happen in a poorly condensed gold restoration.
- 5. Fracture in bending is propagated from regions of maximum tensile strength; and because of the relationship of tensile strength to cohesion, the bending strength in part measures cohesiveness.

These tenets from that study (3) seem to support the contention that testing transverse strength is of value in comparing restorations.

Hardness. It is customary to consider hardness as a test parameter for materials. Convenience of measuring and a strong relationship to

other properties are reasons hardness is measured. Kehl is quoted as defining indentation hardness as a measure of the plasticity and density of metals (4). Rule (5) suggests that the hardness of a gold foil restoration is directly proportional to its tensile strength, which property in turn is related to the cohesiveness of the condensed foil. Hardness provides a rough indication of the wearing quality of a dental restoration (6). A seldom mentioned need, for hardness of restorative materials, is to provide resistance to interproximal wear. According to Black, there is a loss of 1 cm. from interproximal wear in the mesic-distal diameter of the teeth from 3rd molar to 3rd molar at age 35 (7). Stebner (8) suggests that the worn contact areas of extracted teeth show the great importance for hardness of the material used to restore contact areas.

Density. Shell (9), in a paper discussing the clinical aspects of the metallographic properties of gold, said that density is valuable in comparing internal imperfections. These flaws, as has been indicated above, could initiate fracture lines. Shell goes on to say that density determinations do not consider irregularities on the surfaces of the restoration if measured by the Archimedian principle of dividing the weight in air by the loss of weight when suspended in water. It would seem to be of value to measure density in some way that the surface imperfections are also considered. Decreased density as a result of surface irregularity would seem to imply decreased adaptation of the material to cavity wall.

Preparation time. A better insight into the problem of selecting a material for clinical use might be gained by knowing the time required to adequately complete a restoration with a given material.

METHOD OF TEST

Sample form. The samples were all prepared in a single steel mold. See Figure 1. This mold was formed by two rectangular pieces of stainless steel separated by shims so placed as to leave a space 7 mm. by 1.5 mm. by 1 mm. The steel mold was clamped on a platform. The center of the platform was allowed to move and affect strain gauges which were connected to a Sanborn recorder. The load was calibrated and condensing forces observed and recorded while preparing the samples. Figure 2 depicts a portion of the recording sheet, showing the path of the stylus as the gold was compacted. This technique was used to assist in obtaining uniformity of pressure for all samples.

Instrumentation. The condensing tools and instruments and the manipulation of these instruments were characteristic of clinical application. A Loma Linda No. 20 gold condenser was used when hand pressure was the method of condensation. This instrument is double-ended and has rectangular faces which measure 0.5 mm. by 1.2 mm. The longer dimension is curved like the runner of a rocking chair, and the instrument is used with a rocking motion on this curved face.

The Electromallet (McShirley Dental Products) was set to deliver high-frequency blows at the lowest intensity possible. The rationale here was to conform to clinical considerations. The condensing point used was the no. 6, which is supplied with the instrument. This point has a rectangular face which measures 0.5 mm. by 0.9 mm. Figure 3

depicts the instruments and the control settings of the Electromallet which were used for the preparation of the samples.

Insertion. The methods of insertion were selected because they are used with clinical acceptance. A clinically compatible force for each method was approved and this force was then estimated and standardized for each sample by observing the Sanborn recording sheet during the packing of the gold. Approximately 7 to 7.5 pounds force was used while using hand pressure, and the recording indicated approximately 4 pounds when the Electromallet was used. All the samples were prepared by a single operator.

Finishing. The excess bulk (or overpack) of each sample was reduced with a fine carborundum wheel stone. A Cratex wheel of medium grit was then used to finish the gold to the surface of the mold. All samples were treated in this manner, and the samples were considered ready for measuring.

Measuring. The samples were removed from the mold by releasing the set screws of the mold device. The dimensions were measured with a micrometer. The samples were approximately the same size, as would be expected, due to the rigid mold. Certain differences occurred because of the one unconfined surface; therefore, each sample was measured. At this time, each sample was weighed on a Mettler balance. Density determinations were computed from this data.

After measuring and weighing, the samples were placed in individual envelopes and stored at room temperature until the strength testing was performed.

Testing transverse strength. The samples were fractured on an Instron testing machine, which records the fracture load on a chart.

The loading device and sample are depicted in Figure 4. The specimens

were supported by two parallel steel rods, .030 inch diameter, which were located 5 mm. apart on a platform. Load was applied through a 1/16-inch hardened steel ball at a rate of 0.05 in/min. The samples were positioned to allow the steel ball to make contact on the center of the width (1.5 mm.) dimension. Transverse strength was determined by using the following formula:

$$S_{Tran} = \frac{3 \text{ LP}}{2 \text{ bh}^2}$$

where

L = distance between support rods

P = fracture load

b = width of specimen

h = thickness of specimen

Hardness. For hardness determinations, groups of 15 samples were mounted in self-curing clear acrylic. Each group represented one replication of the 15 conditions of the experiment. The top surfaces (finished surfaces) of the samples were aligned so that all could receive equal polishing while being held in this block of acrylic. Standard metallographic techniques were used to polish the samples prior to indentation. Metallographic paper of grit 240 C was used to finish down to the surface of the gold. Metallographic papers of progressively finer grit were used to produce a smooth surface. The samples in the blocks of acrylic were guided by hand across each paper for a uniform number of strokes. After the aforementioned 240 C paper, the polishing consisted of 20 excursions on 400 A silicon carbide paper, followed by 40 strokes on 600 C grit, and finally 20 strokes on 3/0 finishing emery paper. This procedure provided a surface compatible for microhardness measuring.

Indentations were made with a Knoop indenter in a Kentron Microhardness Tester with a load of 300 grams. Ten indentations were made on each specimen and these indentations were applied over the entire length of the sample. The indentations were made approximately in the middle (in regard to width) of the samples.

Knoop hardness values were obtained by converting the lengths of the indentations after considering the load and the angle of the diamond indenter.

Experimental design. To provide for statistical analysis, the experiment was performed as a factorial study with two variables of classification. These tested variables were the materials and the condensing techniques (5 x 3). Therefore, there were fifteen situations in each replication and five replications of the experiment. In other words, each of the five materials was inserted by each of the three methods. Replication provided a sample size of five for each of the fifteen different test situations. The complete study was comprised of 75 samples.

Care was taken to avoid possible sources of error. Sample mold size was maintained essentially constant. The condensing pressure was relatively uniform for each technique. Randomization of the order of preparation was enacted. The samples were all prepared by one operator.

In addition, accepted clinical principles were observed. The heat purifying (annealing), handling, insertion, stepping, and finishing of the gold were performed as they would have been had these samples been dental restorations.

Table 2-Mean Values (±Standard Deviation) for the Tests Performed on 5 Types of Cohesive Gold.

Treatment	Transverse P.S			ardness nber		sity cc.	Prepar Tin Minu	ne
Mgt					7.7.			
Hand Condensed	22970	(8282)	52.2	(16.8)	14.33	(1.04)	18	(5)
Electromallet	24091	(2841)	61.5	(18.5)	14.66	(0.48)	24	(4)
Combined	24054	(9827)	53.3	(22.2)	14.48	(1.05)	23	(5)
Biofil								
Hand Condensed	24884	(5365)	42.5	(17.2)	15.42	(0.27)	28	(6)
Electromallet	15790	(3208)	53.9	(17.3)	15.13	(0.31)	26	(6)
Combined	17978	(2966)	53.2	(15.7)	15.13	(0.45)	22	(4)
Goldent								
Hand Condensed	23640	(6759)	55.2	(17.9)	14.40	(0.89)	21	(4)
Electromailet	22!76	(2728)	63.7	(16.5)	14.50	(0.30)	20	(3)
Combined	27070	(4657)	57.6	(16.2)	14.93	(0.66)	23	(2)
Foil								
Hand Condensed	42301	(7854)	68.5	(12.8)	15.90	(0.41)	35	(5)
Electromallet	37902	(5068)	69.7	(16.0)	15.83	(0.42)	34	(4)
Combined	38994	(6138)	68.6	(15.7)	15.83	(0.44)	35	(4)
Mat → Foil								
Hand Condensed	27971	(4431)	69.7	(15.3)	14.99	(0.48)	22	(2)
Electromallet	29397	(1435)	70.9	(12.4)	15.15	(0.17)	28	(3)
Combined	32382	(2929)	75.0	(9.6)	14.95	(0.52)	23	(속)

RESULTS AND DISCUSSION

A tabulation of the means and standard deviations of the 15 different test situations is provided in Table 2. This data includes each of the parameters considered. The data was submitted to statistical analysis for testing the hypothesis that the 15 combinations of materials and methods of insertion provided equal samples. The method used was to test the data for homogeneity of variance by using Bartlett's Test and then to proceed with analysis of variance if the variances proved to be homogeneous. Each test parameter was considered separately. The results of the Bartlett's Test indicated homogeneity of variance for each parameter tested. Tables showing the factors of analysis of variance for the data of each test are contained in the Appendix. These tables demonstrate the significance values at F.95 and the F ratio value indicating significance or non-significance for each variable.

Transverse Strength

The data for the strength test may be seen in bar graph form in Figure 5. The analysis of variance at the 95% significance level indicates that the materials variable provided samples of different strength values. There was insufficient evidence to indicate that the method of condensation produced differences, nor was there any interaction indicated. The effect of replications was significant but was removed from the consideration of the materials and methods. By removing the replication sum of squares from that of the error sum of squares, increased sensitivity was imparted to the analysis.

Duncan's Multiple Range Test was then employed to determine which materials were significantly different than others. The results are indicated by solid bars connecting those materials which are not significantly different at the 95% level:

	Biofil	Mat	Goldent	Mat+Foil	Foil
Means PSI	19,550	23,705	24,295	29,917	39,732

The specimens made from foil were significantly stronger. Those made from the mat+foil combination were stronger than those from Biofil but were not different than those made from mat or Goldent.

Density

Statistical analysis of the density data indicated the materials variable was significantly different at the 95% level. As with transverse strength, the available evidence did not suggest a difference caused by either condensing methods or interaction. A bar graph representation of the data is seen in Figure 6. The results of the range testing were:

	Mat	Goldent	Mat+Foil	Biofil	Foil
Mean Density	14.49	14.61	15.03	15.23	15.85

It can be seen that samples produced from foil were of higher density than those made from mat, mat+foil, or Goldent. The density of the Biofil specimens was not significantly different than that of the foil samples. This was an interesting development, in that density and

strength of cohesive gold restorations are thought to be closely related. As we have seen above, however, the Biofil samples were significantly weaker. The possibility exists that the powdery texture of Biofil precludes gross porosity and improves density, but does not supply optimal cohesion.

Hardness

The analysis of variance of the hardness test data was minutely complicated by an additional source of variation which had to be removed. This source had its origin in the multiple testing (10 indentations) of each sample. Both of the tested variables, materials and methods, were different at the 95% significance level.

Duncan's M.R. Test was applied separately to each variable, as there was not evidence that interaction was a factor. The results of the test for the means of the materials variable were:

	Biofil	Mat	Goldent	Foil	Mat+Foil
Mean KHN	49.9	55.6	58•€	69.0	71.9

The samples from foil and mat+foil had higher Knoop hardness values than samples from the other materials. It was not surprising that foil and mat+foil did not differ. In each case, the foil is the material tested because of the foil veneer of the mat+foil combination. The hardness of the Goldent samples was significantly higher than that of the Biofil samples.

The multiple range test was then used to determine which condensation method produced different hardnesses. The test showed:

	Hand Condensed	Combination	Electromallet
Mean KHN	57.6	61.5	63.9

The samples prepared when using the Electromallet alone had higher hardness values than the samples prepared by hand condensation, but were not harder than the combination technique specimens. Figure 7 demonstrates the mean values of the hardness data in a bar graph.

Time

The amount of time required to make samples from the different materials was shown to be significantly different by the analysis of variance. Methods of condensation demonstrated no differences. The time required to prepare the foil samples was significantly longer in duration than the time required to make samples of any of the other materials.

	Goldent	Mat	Mat+Foil	Biofil	Foil
Means Min.	21	22	24	25	35

A bar graph representing this data is found in Figure 8.

Significance of Results

The clinical importance of the properties considered is recognized. It has been shown that the sample values of these properties are different for different materials. This would seem to offer the clinician a degree of guidance. The clinician should be cognizant of the limitations of this guidance. For instance, this laboratory study

cannot offer information concerning physiologic considerations. The clinician must evaluate, by other means, the effect of a method of insertion and the effect of a material on the tooth and its contiguous tissue. The dentist must also decide whether the increased time and expense of a particular procedure is worthwhile.

Characteristically, many of these clinical evaluations are quite subjective. There is, for example, a comment made by dentists using Goldent that it "finishes down" better than gold foil. This would imply a better marginal adaptation because of superior ductility. It is believed that severely work-hardened materials (such as gold foil) gain surface hardness but lose ductility. Skinner (10), in discussing gold alloys, suggests that this loss of ductility is also related to a loss of resistance to abrasion. Reasoning might suggest that, depending upon the particular need, the higher hardness of a surface might be detrimental to the desired result. It is speculated that the "finishing" superiority of Goldent is related to its demonstrated lessened hardness as compared to gold foil.

The materials and the mean values of the samples for all of the tests are listed in Table 3. Those values which were not significantly different are indicated by horizontal lines from each vertical bar.

TABLE 3

COMPARISON OF MEAN VALUES

Material	Strength	Density	Hardness	Time
Foil Mat+Foil Goldent Mat Biofil	39,700 29,900 24,300 23,700 19,600	15.9 15.0 14.6 14.5 15.2	⁶⁹] ⁵⁹] ₅₆]	35 24 21- 22- 25

The use of gold foil offers superior values for the properties tested but requires considerably more time than the other materials. At least two of the materials, foil and mat+foil, are known to be clinically acceptable. The proximity of the values for Goldent and mat to those of mat+foil would appear to make them logical candidates for clinical evaluation.

No evidence is offered, but the use of Goldent and mat gold seems to offer the advantage of improved ductility. Certainly, a need exists for a test to evaluate the subjective feeling that Goldent and mat were easier to use. There is importance to the observation that a particular cohesive gold material is easier to "start" and "finish down" than others. Clinically, those two procedures are crucial to the resolution of the tooth and to the success of the restoration.

In certain instances the mat+foil combination would be advantageous. Specimens prepared this way offered the same hardness as the foil samples, plus the ease and time economy of using mat for the bulk of the sample.

The condensing techniques did not demonstrate great differences of results. Only in considering hardness was there a difference — none was demonstrated in the strength and density and time tests. The clinical implication here is that many dentists consider the malleting of cohesive gold a necessary but unkind influence on the tooth and its integument. The results of this experiment would seem to negate the "necessary" aspect of the procedure. Even with the hardness test, the use of the Electromallet alone offered no advantage over the technique of filling the bulk of the cavity by hand force and then malleting the veneer portion.

The use of each of the materials tested, with the likely exception of Biofil, offers certain clinical advantages. It does not seem to be necessary to use malleting forces entirely when preparing cohesive gold restorations.

SUMMARY AND CONCLUSIONS

Samples of several types of cohesive gold prepared by different condensation methods were compared by testing their transverse strength, density, and surface hardness. The time required for sample preparation was also considered. The samples were prepared in a steel mold using uniform forces and randomization of order. The tests were performed by using accepted laboratory instruments and techniques.

The results were examined for homogeneity of variance and the analysis of variance procedure was used. Where significant differences, at the 95% significance level, were indicated, Duncan's Multiple Range Test was used to determine rank.

Under the conditions used for this experiment the following conclusions can be made:

- 1. The methods of condensation (hand pressure, Electromallet malleting, and a combination of the two) produced samples which were not different in transverse strength and density.
- 2. The samples produced with the Electromallet were slightly harder than those prepared with the hand force technique, but were not harder than the samples made by the combination hand force-Electromallet method.
- 3. The use of gold foil resulted in specimens which were stronger than the samples of Biofil, mat gold, Goldent, and mat+foil combination.
- 4. The density of the foil specimens was superior to the samples

made from mat, Goldent, and the mat+foil combination.

- 5. Samples of foil and mat+foil had higher Knoop hardness values than those prepared from Biofil, mat, and Goldent.
- 6. More time was required to prepare samples from foil than from any of the other materials.

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Figure 1. Split Sample Mold and Strain Gauge Loading Platform

Figure 2. Portion of Recording Sheet from Sanborn Recorder



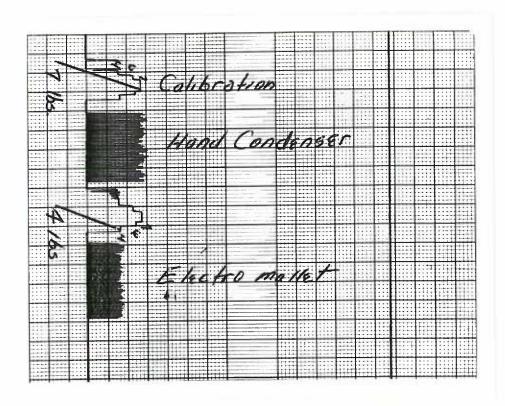


Figure 3. Instruments Used for Condensing the Gold

Figure 4. Load, Sample, and Platform for Transverse Strength Test





Fig. 3 - Effect of the Condensation Method on the Transverse Strength of Several Types of Cohesive Gold. Mean Values P.S.I.

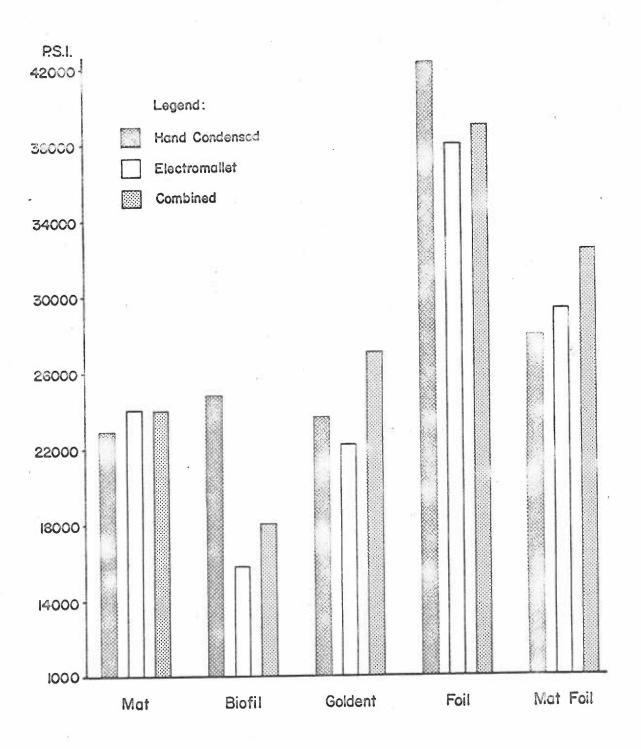


Fig.6 - Effect of the Condensation Method on the Density of Several Types of Cohesive Gold. Mean Values in grams/cc.

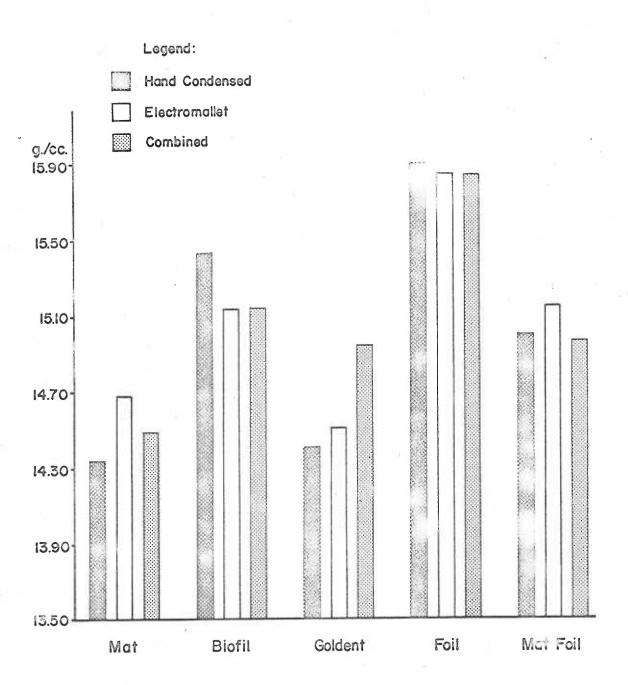


Fig. 7 - Effect of the Condensation Method on the Hardness of Several Types of Cohesive Gold. Mean Values Knoop Hardness Number (300g. Load).

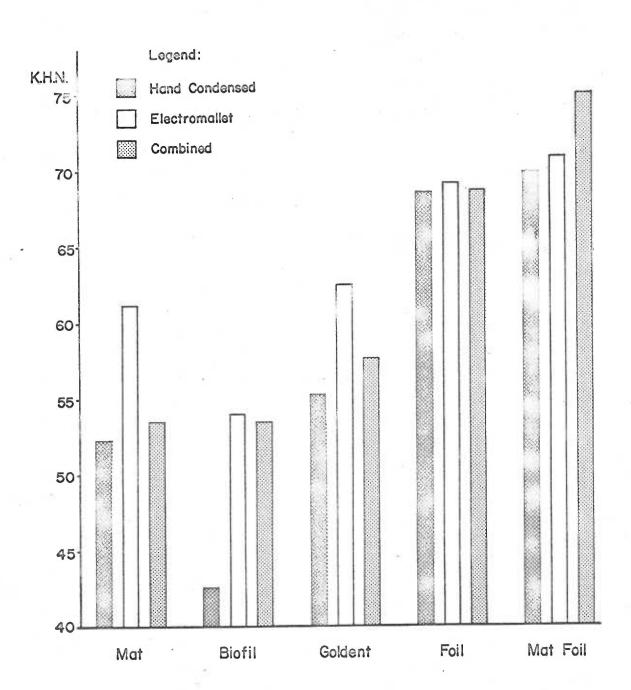
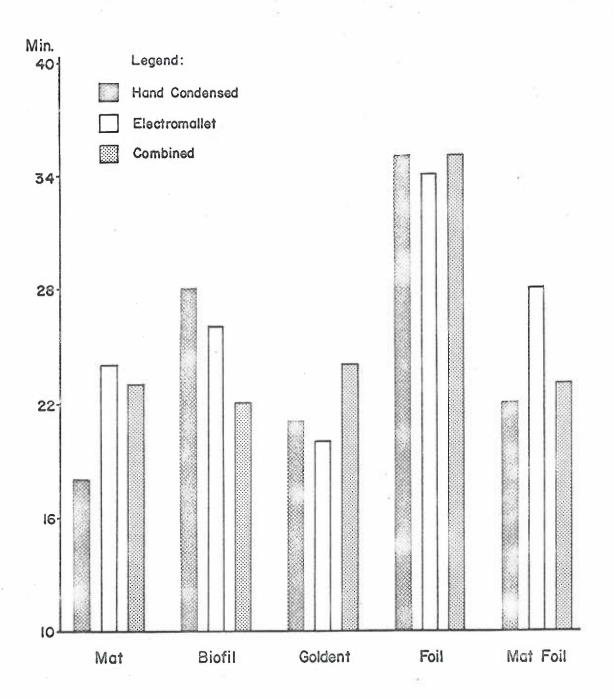


Fig. 8 - Time in Minutes Required to Prepare Cohesive Gold Specimens. Means of 5 Samples.



APPENDIX

ANALYSIS OF VARIANCE — TRANSVERSE STRENGTH

Source of variation	Degrees of freedom	Sum of squares	Mean squares	F ratio
Materials	4	3,649,788,9	66 912,447,241	*36.83
Condensing method	2	93,104,6	97 46,552,348	1.88
Interaction (MxC)	8	302,512,3	32 37,814,042	1.53
Replications	4	417,498,4	17 104,374,604	*4.21
Error	56	1,387,416,4	23 24,775,293	
Total	74	4,045,405,9	95	
	Significance	F.	95(4,56) = 2.56 95(2,56) = 3.16 95(8,56) = 2.12	

ANALYSIS OF VARIANCE -- DENSITY

Source of variation	d.f.	S.S.	M.S.	F ratio
Materials	4	17.65992	4.41248	*16.97
Cond. methods	2	.04517	.02259	.087
Interaction	8	1.41305	.17663	.68
Replications	4	5.94849	1.48712	*5.72
Error	56	14.64023	.26143	×

Significance values are the same as above

ANALYSIS OF VARIANCE -- HARDNESS

Source of variation	d.f.	S.S.	M.S.	F ratio
Materials	4	50,801.04	12,700.26	*15.84
Cond. methods	2	5,077.45	2,538.73	*3.167
MxC	8	4,287.75	535.97	.67
Sampling	675	143,975.81	213.30	.27
Replications	4	5,271.23	1,317.81	1.64
Error	56	44,884.00	801.50	
	Significance Values:		F.95(4,56) = 2.56 F.95(2,56) = 3.16 F.95(8,56) = 2.12	

ANALYSIS OF VARIANCE - TIME

Source of variation	d.f.	S.S.	M.S.	F ratio
Materials	4	1,772.45	443.11	*25.57
Cond. method	2	39.23	19.61	1.13
Interaction	8	234.51	29.31	1.69
Replications	4	7.25	1.81	.10
Error	56	970.35	17.33	