

THE INFLUENCE OF SOLIDIFICATION TIME
ON THE MICROSTRUCTURE AND PHYSICAL PROPERTIES
OF DENTAL GOLD CASTING ALLOY ALLOY

by

A. Bruce Ady, B.S.

A Thesis

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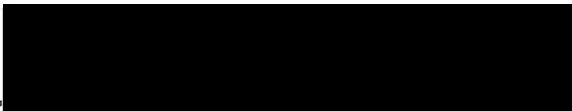
Presented to the Department of Dental Materials
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INTRODUCTION

Statement of the Problem. Dental restorative appliances fabricated from cast gold alloys are subjected to severe conditions in service. They are vulnerable to indentation by opposing teeth during mastication and they are exposed to the corrosive action of the oral fluids. In more complex applications, such as bridgework, appreciable bending forces are commonly present in normal use. A cast gold restoration must withstand service conditions to be of any clinical value. To this end, every effort should be made to create a cast structure with optimum properties. In order to define such a cast structure it is necessary to establish the properties that are desired.

In addition to aesthetics, tissue tolerance, and dimensional accuracy, the structure must be capable of withstanding permanent deformation or gross failure caused by any static or dynamic forces normally found in the oral environment. In effect, the structure must be strong and hard. Likewise, it should have a compact and uniform microstructure to minimize corrosion.

The hardness, strength and microstructure of a casting are essentially established at the moment the alloy achieves complete solidification. Further manipulations of the structure in the solid state are possible which can enhance these properties, but these manipulations are frequently impractical. It becomes necessary, therefore, to capture the best qualities possible during the solidification process.

In clinical practice, the solidification rate of a gold casting can be altered by both advertent and inadvertent changes in the casting procedure. It is conceivable that the quality of the physical properties could be significantly affected by these alterations of the solidification rate.

It was, therefore, the purpose of this investigation to (a) determine the length of time necessary to achieve complete solidification in a standard, clinical-sized casting under conditions of common practice, (b) alter the casting procedure in such a manner as to produce both faster and slower solidification times, and (c) to observe and evaluate the effect of solidification time on the microstructure and physical properties of the casting.

Approach to the Problem. The cooling and subsequent solidification of a liquid metal is influenced by the ambient temperature of the environment in which it is placed. As the liquid strives to reach thermal equilibrium with its cooler environment, it transfers heat to that environment. The rate of heat transfer is manifest in the rate of change of the metal temperature. When cooling has progressed to a temperature level coincident with the freezing point of the metal, the heat of crystallization evolved in the precipitation of solid causes a temporary arrest in the descending temperature. During the time the temperature is arrested, solid is continually precipitated until no liquid remains. At this point, heat of crystallization is no longer evolved, thereby permitting the metal temperature to resume its decline. The temperature continues to lower until a state of equilibrium is achieved (1).

The duration of the solid precipitation stage is theoretically a

key factor in the process of establishing the nature of the crystalline microstructure. If the solidification progresses rapidly, small crystals (grains) are formed. Conversely, prolonged solidification results in larger crystals (grains). An ideal cast structure consists of small, uniform, and homogenous grains which produce strength by virtue of the "slip" inhibiting nature of the grain boundaries. Cast structures containing voids and large, irregular grains would have a reduced ability to resist the deformation associated with "slip" (2). Theoretically then, gross changes in the duration of the solidification process could influence the physical properties of the cast structure. This investigation was designed to test this premise. In the null form, the basic hypothesis states that changes in the time required for solidification of a dental gold casting alloy do not significantly affect the physical properties selected for observation.

Physical tests were designed which would measure the cooling and solidification of a dental type casting under conditions of common practice. It was felt that these conditions must be met if any valid projections were to be made from the results. Thus, the temperature and time of the liquid-to-solid transitions were measured within the casting mold. The materials and methods utilized conformed to standard dental practices.

From theoretical consideration, the alloy solidification time was assumed to be a function of the temperature differential between the liquid melt and the mold. A series of pilot castings demonstrated the validity of this assumption. Specific test procedures and data are shown in Appendix A. Based on this relationship, and keeping the temperature of the liquid melt constant, the solidification time of

the cast alloy could be altered by an appropriate adjustment of the mold temperature alone.

Each test casting was then made under controlled laboratory conditions, measuring the solidification times as produced by various mold temperatures. The microstructure of each casting was observed and photographed. The castings were then subjected to hardness and bending strength tests.

REVIEW OF THE LITERATURE

The first usable casting process for dentistry was introduced in 1907 by Taggart (3). These first castings were made into a room temperature mold. For nearly three years, the new technic was the subject of great interest and study. Ward (4) observed that a larger casting remained hot longer than a smaller one. Lane (5) was concerned with dimensional accuracy and concluded that the temperature of the flask at the moment of casting was of significant importance in the control of alloy shrinkage. In 1910, Van Horn (6) advocated casting into a heated mold. Several more years of development passed, and in 1923 Shell (7) announced that the alloy temperature during casting was also of significant importance. He described what he felt to be an ideal situation:

"When the pressure is put upon the molten alloy to force it into the cavity, it should be at such a temperature that the metal adjacent to the walls of the cavity should solidify rapidly, leaving the pressure still exerted on the partially solidified mass . . . the metal should solidify immediately on striking the walls of the mold, and the pressure should be exerted until the entire mass has solidified."

In a further study, Shell (8) stated that:

". . . for a perfect casting, the metal must flow to the extreme margins before solidifying, and so it must be heated sufficiently to withstand the cooling influence of the mold

until these margins have been filled with metal. As soon as this occurs, the metal should solidify rapidly, both to form small grains and to prevent shrinkage. The temperature of the mold, then, is not of such great importance as the temperature of the gold, which should be sufficiently high to maintain the alloy in a fluid condition until it has reached the extreme margins of the casting."

Early in 1929, Wilkes (9) conducted some experiments in which he cast into a 200° F. mold with alloys ranging in temperature from 1770° F. to 2640° F. in 40° F. increments. He reported "good" castings until 2140° F. Thereafter, the castings were "too rough" and he was unable to pickle them clean.

Souder (10), in 1933, performed some tests using various methods of introducing the gold alloy into the mold, and declared no significant differences could be detected in the physical properties of the alloy cast by these methods. In this same report, he stated that "most dental castings are completed in . . . one-tenth to one-twentieth seconds."

In 1940, Myers (11) published the results of his studies with the air-pressure casting machine and remarked that "differences in the temperature of the molds between 1000° F. and 1500° F. have no effect on casting time . . . The casting time is directly proportional to the volume of the pattern chamber." Figuring casting time from the moment the air blast began, Myers recorded one-tenth second as the time for alloy to fill the mold. He reported that mold temperature, gold temperature, sprue length, and condition of the alloy (whether the gold was new or used) did not have any effect on casting time. In a sequel (12), Myers duplicated his tests using a centrifugal casting machine. He

concluded that the two methods were essentially similar in their results. The precise length of time for alloy casting and solidification was still in doubt in 1947 when Phillips (13) stated that the "gold in the mold does not solidify for seconds after air pressure has been applied."

Throughout the development of the dental casting procedure, investigators have assessed the resulting castings by observation and physical testing of assorted properties. As the assessment revealed the intricacies of metallurgy, some generalized principles were established.

In 1933, Asher (14) noted considerable variation in surface hardness of gold alloys and suggested that the variation was caused by porosity, coring and segregation, and by the formation of the intermetallic compounds Au_3Cu and $AuCu$. Wilson (15) mentioned a means of controlling porosity by providing "for additional molten metal to be drawn into the mold to furnish the areas that solidify or freeze last, thus . . . preventing voids."

Ryge and Fairhurst (16) reported the effect of temperatures on the formation of sound castings. They stated that "grain sizes were consistently larger in castings made in the electrically controlled casting unit as compared to the ordinary centrifugal casting machine . . . Cooling occurs faster in the ordinary machine . . . Mold temperatures lower than $900^{\circ} F.$ to $1250^{\circ} F.$ caused microporosity and gas inclusions. High mold temperatures caused subsurface porosity." Recently, Björn (17) used an electron probe to discover that gold alloy grains in the "as cast" condition do not have an homogenous composition. He found that the grains were heavy in copper and light in platinum at the grain boundaries.

Although the transformation experienced by the gold-copper system in the solid state theoretically does not occur during the casting process, it nonetheless is affected by time and temperature. Kurnakow (18) first published his studies on the solid state transformations in alloys of gold and copper in 1916. Many years later, in 1931, he published further information (19) concerning the formation of the intermetallic compounds Au_3Cu and $AuCu$. The formation of these compounds is accompanied by a "marked fall in volume," and can be retarded by "sufficiently rapid chilling." In the same year, Haughton and Payne (20) reported that "all alloys quenched above the transformation temperature are homogenous." This statement has been subsequently challenged by Björn, as previously mentioned. Later, in 1934, Asher (21) published a comprehensive report on the $AuCu$ transformations.

Very soon after Taggart introduced his technic, the problems of dimensional accuracy and strength became subjects for critical investigation. Price (22) determined the casting shrinkage of gold alloy on solidification to be 1.64%. In 1926, Coleman (23) reported his value for the casting shrinkage of (coin) gold alloy as 1.25%. He noted also that "the compensation of part of the total shrinkage of the solid metal may be dependent upon a difference in the rates of cooling of different parts of the casting." He further stated that (his) results " . . . indicate that porosity . . . is the combined result of a number of causes. The most prominent of these are localized shrinkage, due to different rates of cooling in different parts of the casting, and occluded gases." Coleman did not find evidence of segregation in the alloys used in his experiments.

By 1932, the National Bureau of Standards had accumulated enough

information to publish a tentative specification for dental casting gold alloy (24, 25). In the succeeding years, several more significant contributions were offered. Johnson (26) refined previous tests in his evaluation of types of casting machines. His results indicated that the centrifugal and steam pressure casting machines yielded equivalent castings. Kaires and Thompson (27) studied the heat treatment effect induced by bench-cooling of castings. They used the Knoop Hardness number as criterion for evaluation. A statistical analysis of their data shows no significant differences in KHN until the casting had bench-cooled at least 15 minutes. No differences existed within the casting ring for any individual test.

The metallurgical aspects of the dental casting alloys have also been subject to scrutiny. Raper (28) experimented with compositions in 1936 and discovered that small amounts of iridium, ruthenium, osmium, or rhodium introduced into the alloy produced a fine grain structure. He rationalized the desirability of this effect by mentioning that ". . . fine grained alloys have improved mechanical properties as shown by suitable tests." A dental gold manufacturer states (29):

"It has been generally accepted that, if all other factors are equal, the alloy having the smaller grain size will have better mechanical properties. In a thorough and detailed research program, we have confirmed this, . . . In general, the elongation of the harder alloys is increased materially; the proportional limit and the ultimate tensile strengths are increased slightly. The really dramatic improvement can be seen in the uniform crystalline structure, which gives greater uniformity of properties, not only within the individual casting but also from one casting to the next."

The concept of striving for fine grains is currently popular, as evidenced in the statement by Skinner and Phillips (30) that ". . . the smaller the grain size of the metal, the better will be its physical properties." The crystallization of liquid gold was described in 1940 by Crawford (31) who said:

"It is not uncommon for dental alloys to have a melting range of 100° F. to 150° F. This means that when the molten metal is cast into the mold, it may begin to freeze at approximately 1800° F. and not be completely frozen until the temperature has dropped to 1650° F. Metals of this type usually freeze to form a typical pattern. Starting at many centers, the metal will begin to freeze at the upper end of its melting range. As the temperature continues to fall, the metal adds to the centers originally formed to produce what are called dendrites. As the temperature continues to fall, these spine-like forms continue to grow until they are arrested by collision with adjacent dendrites. After the growth of the dendrite is arrested, the remaining molten metal fills in around the originally formed dendrite."

Williams and Homerberg (32) have devoted a section of their metallurgical textbook to the phenomenon of dendritic growth. Hume-Rothery and Raynor (33) suggested that ". . . dendritic growth occurs under conditions of supercooling . . ." They continued to describe their idea of the solidification by saying that "it is almost impossible to secure continual access of liquid to all parts of a crystal during solidification, and we may therefore expect one set of voids to be produced at the grain boundaries and another set of voids on a much finer scale to be

formed in a pattern which is the inverse of the original dendrite." Like Björn, they felt that ". . . the properties of the crystal are uniform throughout . . ." and ". . . (segregation) impurities may be expected to lie partly in the grain boundaries."

Dental casting has progressed from relatively simple inlay to complicated crown-and-bridge application. In determining the type of material best suited for a restoration, certain consideration must be given to the stresses that may be applied to the restoration in service. Mahler and Terkla (34) studied the stress patterns of several types of restorations and indicated that "the dental bridge consisting of two abutments with a (soldered) pontic is another example of a beam bending . . ."

Laboratory methods have been established for testing materials subject to this type of stress pattern (35). Interpretation of experimental results in stress-strain relationships is also well established (36, 37).

The theory of thermometry has been discussed by most authors of physics texts. A description of the use of optical pyrometers and thermocouples is presented in one such text by Sears and Zemansky (38).

Several authors describe laboratory procedures for the metallographic preparation of specimens. Kehl (39) has also included the ASTM grading charts for standard grain sizes. The theory of rough and final polishing is thoroughly discussed in a publication by a manufacturer of metallographic supplies (40). Krill (41) introduced a technique of final polishing by vibration. Etching is discussed by all these authors.

METHODS AND MATERIALS

General Procedure. Three different type III gold casting alloys were cast at five mold temperature levels. Five specimens were cast at each condition as shown in the following schedule:

<u>Mold Temperature</u>	<u>Alloy A</u>	<u>Alloy B</u>	<u>Alloy C</u>
1	5 specimens	5 specimens	5 specimens
2	5 "	5 "	5 "
3	5 "	5 "	5 "
4	5 "	5 "	5 "
5	5 "	5 "	5 "

The five specimens of each alloy cast at the five mold temperatures were then observed and tested in order to determine ASTM grain size, extent of porosity, diamond pyramid hardness, proof stress, and modulus of elasticity in bending. A step-wise multiple regression analysis was used to detect the relative effect of the solidification times on these properties.

Rectangular-shaped specimen patterns were prepared using an inlay wax and vacuum-invested in an asbestos-lined steel ring. A thermocouple tip was attached to the edge of the pattern such that, upon wax elimination, it protruded into the mold cavity in which the gold was to be cast. During burnout, the mold temperature was monitored with this thermocouple. When the gold alloy was cast into the mold, its cooling and solidification was monitored by the thermocouple and

a recording system. Melt temperature of the alloy was controlled using an optical pyrometer.

Recovered castings were trimmed, mounted, and metallographically polished. Photomicrographs were taken of each specimen. The castings were then tested for hardness and bending strength.

Specimen Preparation. Test specimens were designed in the shape of a bar 2 mm. thick, 5 mm. wide, and 12 mm. long. This size closely approximates the quantity of gold employed in a large clinical casting. The shape was selected for use in determining transverse (bending) strength and hardness. Although not clinical in configuration, it does provide a dimensional relationship similar to the occlusal plane of a full crown and its attached pontic. In order to maintain control of the variable under consideration (solidification time), the test specimen size, shape, and method of preparation were kept constant within laboratory limits.

The wax patterns were made using a dental inlay wax conforming to ADA Specification No. 4 for an indirect (type II) wax. Double entry (V-type) sprues of 12 gauge wax 4 mm. long were attached to the long edges of the patterns, near the ends. A 28 gauge chromel-alumel thermocouple tip was luted to the center of the top edge of each pattern with sticky wax. This position was selected in order to retain integrity of the cast specimen and still measure the temperature changes of the alloy close to the test area. The thermocouple leads were insulated at their point of entry to the molds by a small quantity of ceramic material.

Each pattern was mounted on a rubber crucible former and enclosed in an asbestos-lined steel ring. Prior to investing, the asbestos

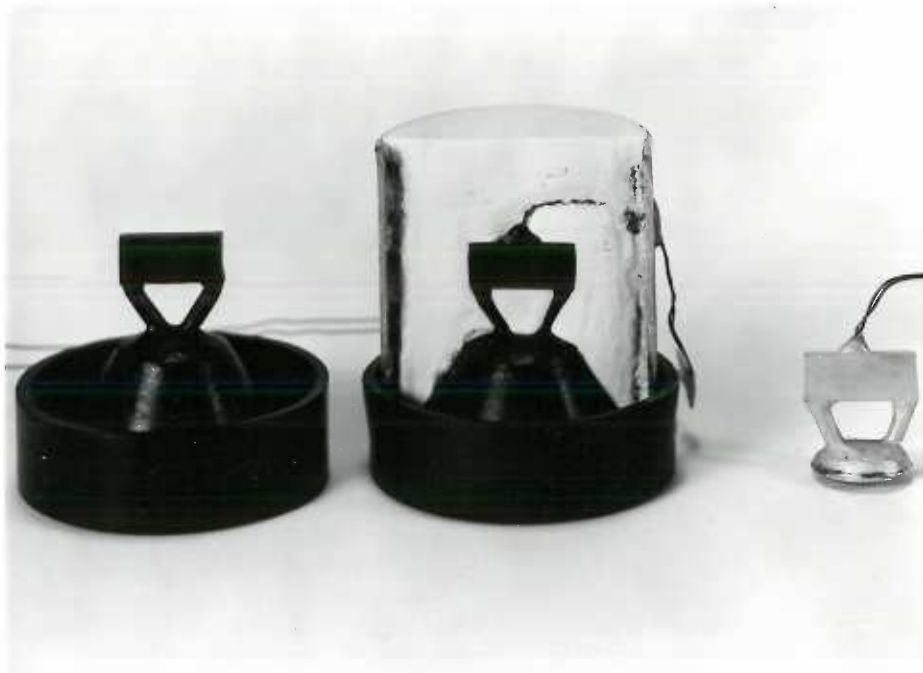


PLATE 1. Sequence in the preparation of the test specimens.

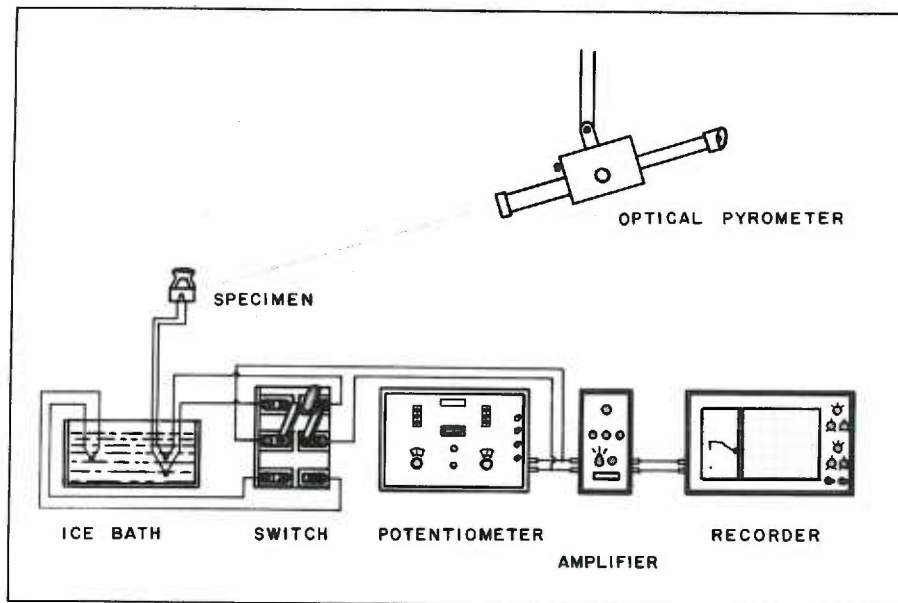


FIGURE 1. Schematic diagram of temperature recording equipment.

liner was moistened. Investing was done with a vacuum investing unit (Whip-Mix Corp.) using Cristobalite investment (Kerr Mfg. Co.) at a W/P ratio of 0.38. The molds were then permitted to set for one hour before being placed in the furnace for burnout. Plate 1 illustrates the specimen's preparation.

Each invested mold was placed in the pre-heated furnace (Torit) and the thermocouple leads were drawn through an insulated port to the recorder connections. The furnace temperature was set with the furnace dial. Mold temperatures, however, were measured from the thermocouples attached to the specimens.

Casting Procedure and Temperature Measurements. Each specimen's thermocouple leads were connected to an X-Y-Time recorder (F. L. Moseley Co.) through an ice-bath reference junction and a direct current amplifier (Video Instruments Co.). The system was calibrated for each test against a portable potentiometer (Leeds & Northrup), and millivolt calibration marks for each recording were placed on the recording paper. Figure 1 illustrates the system schematically.

Mold temperature was monitored throughout the burnout procedure. Just prior to casting, the calibration was checked and the recorder activated. The recorded graphs, therefore, contained the calibration marks, the mold temperature prior to transfer, the mold temperature during transfer, and (when the liquid alloy was cast into the mold) the cooling curve for the gold alloy. Figure 2, page 23, is an actual recorded tracing of one of the test castings. Reference axes and identification remarks have been superimposed on the recording for illustrative purposes.

In order to simplify the instrumentation and still provide a realistic and acceptable casting procedure, an air-pressure casting

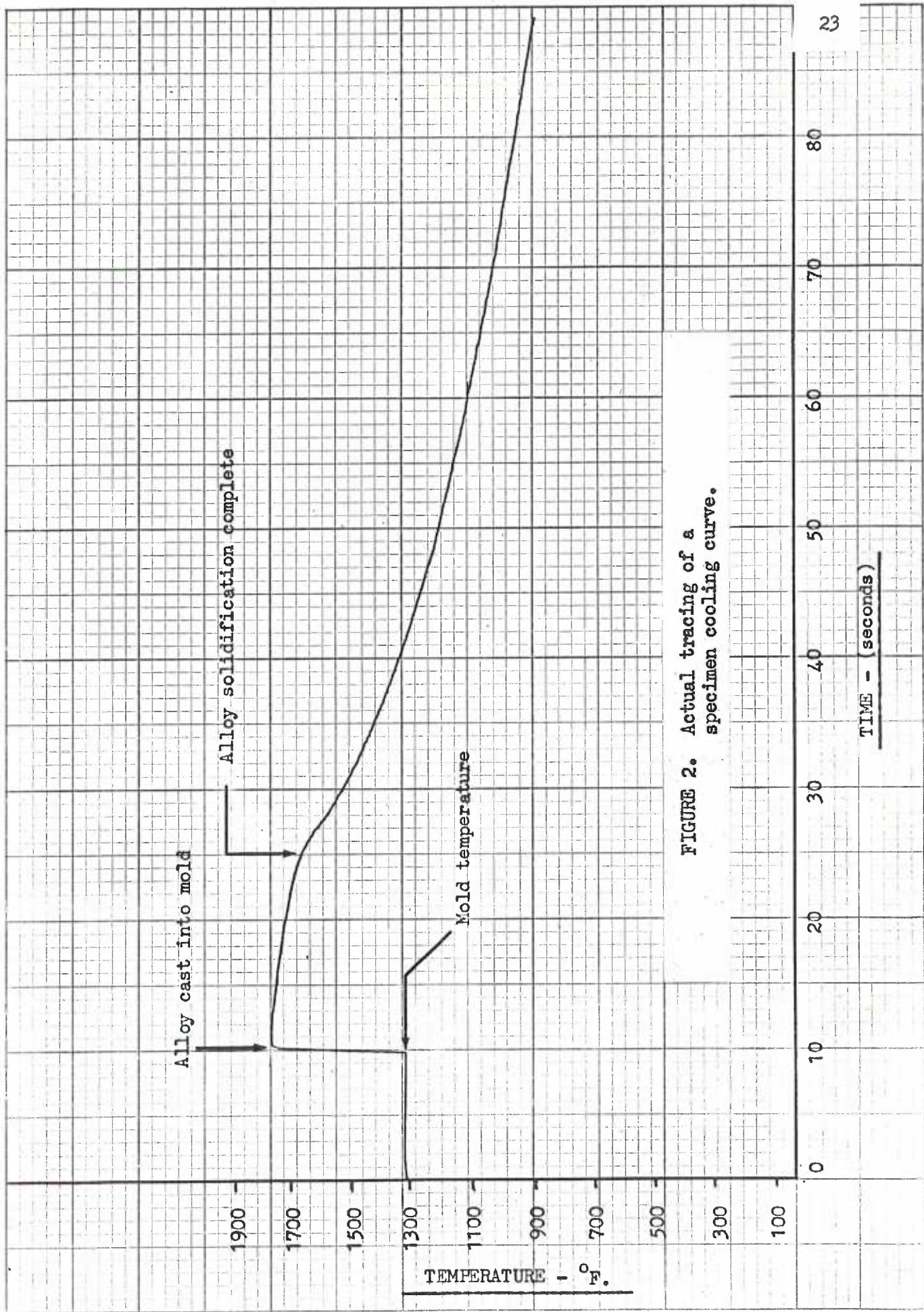


FIGURE 2. Actual tracing of a specimen cooling curve.

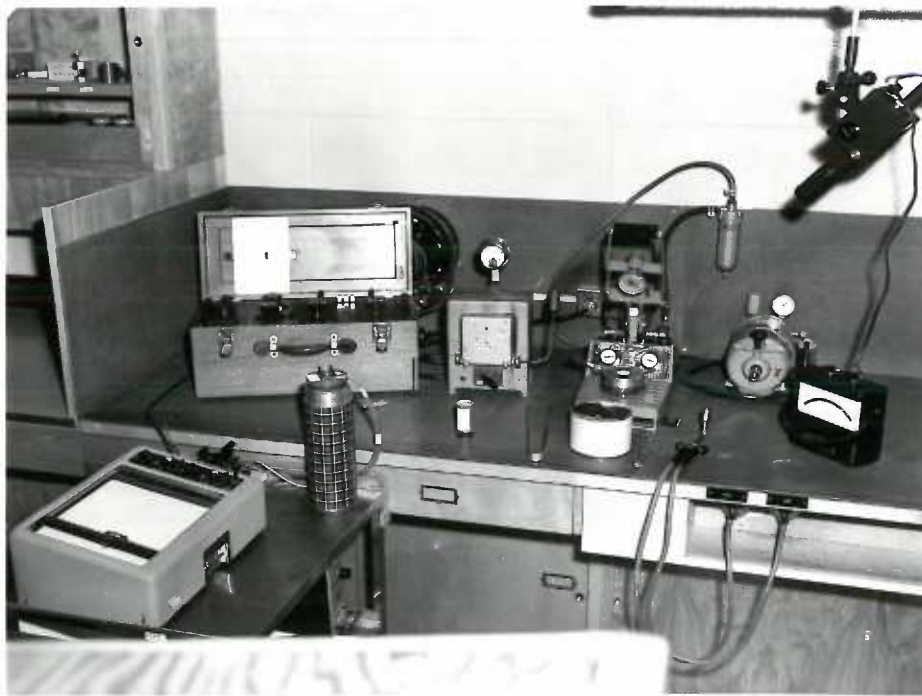


PLATE 2. Casting and temperature recording equipment.



PLATE 3. A test casting in progress.

machine (Whip-Mix Corp.) was used. Initial and final air pressures were kept constant at 7 and 25 pounds respectively. These pressures are recommended by the manufacturer as optimum for dental castings. Plate 2 shows the casting and recording equipment in panorama.

Immediately prior to the transfer of each mold, 4 dwt. of new dental gold alloy was pre-melted on a charcoal block adjacent to the casting machine. A dental carbon flux (J. M. Ney Co.) was employed to prevent oxide formation with the base metals of the alloy. A standard gas-air blowpipe provided the necessary melting flame. When the alloy had fused, the blowpipe flame was momentarily removed to allow the metal to solidify sufficiently for transfer to the mold crucible. At this time the mold was brought from the furnace and positioned in the casting machine. The barely-solid gold alloy was quickly carried to the mold crucible with quartz-tipped forceps and the blowpipe flame was re-applied. Plate 3 illustrates the casting procedure at this stage. As the alloy was being remelted, its temperature was monitored by an optical pyrometer positioned over the casting machine. When the proper melt temperature was achieved, the casting machine arm was closed and the casting completed.

Actual mold temperatures at the moment of casting were taken directly from the cooling curves and recorded to the nearest 0.1 millivolt. Solidification times were also taken from these curves. Using the nearly vertical segment of the curve (representing the sharp increase in temperature as the molten alloy struck the embedded thermocouple) as a zero point, solidification time was figured to the point where the recorded temperature drop definitely deviated from a constant rate. These time values were recorded to the nearest 0.1 second. Subsequent statistical analysis of the relationship between mold

temperature and solidification time revealed an overall correlation coefficient of 0.936. Taking into account the fact that this value included the error introduced by using three different alloys, the correlation was considered to be highly significant.

Selection of Test Alloys and Mold Temperatures. The three dental gold alloys used in this study were selected after consideration of several factors. First, in order to maintain conditions of clinical application, type III (heat treatable) alloys were indicated. Second, these alloys should conform to ADA Specification No. 5 for alloys of this type. Third, for purposes of better experimental control, it would be desirable to have alloys with similar melting temperatures. From information supplied by the manufacturers, three such alloys were found. The melting range of one alloy (Crown Supreme, General Refineries) was relatively short, about 35° F. This short-melting-range alloy is referred to throughout the study as Alloy A. Another alloy (Firmilay, J. F. Jelenko Co.) had a melting range similar to Alloy A, but according to the manufacturer it possessed a special fine-grain quality. This fine-grained alloy has been labeled Alloy B. It was of interest to note that the manufacturer of Alloy B advertises the fine-grain quality as a boon to dental casting. According to these advertisements, regardless of treatment during casting, alloys possessing this quality will always solidify in small, uniform grains. The third alloy (Ney Oro-B, J. M. Ney Co.) had a much longer melting range, about 125° F. This alloy is referred to as Alloy C.

Based on the melting range information provided by the manufacturers of these alloys, a standard melt temperature of 1900° F. was selected. This temperature was considered to be sufficiently high to ensure complete liquefaction of the alloys and still be within the limits of

common practice. Throughout the testing, all castings were made at the moment the alloy reached 1900° F. as measured by the optical pyrometer.

The mold temperature levels selected were 700, 900, 1100, 1300, and 1500 degrees F. The 900 and 1300 degree mold temperatures are commonly employed with present-day casting technics. Temperatures below 700° F. do not provide adequate wax elimination from the mold, and temperatures above 1500° F. result in gross chemical deterioration of the mold material.

A total of 75 castings was made. Five repetitions of each of the three alloys were made at the five mold temperature levels. Each of the test castings was quenched in room temperature water three minutes after cast. The castings were then retrieved, cleaned, and pickled in the customary manner. A carborundum separating disc was used to remove the sprues and thermocouple attachments.

Microstructural Observation. The specimens from the five repetitions of each test condition were mounted as a group for metallographic preparation. This was done in the interest of economy, quality control, and ease of specimen identification. The specific metallographic procedure employed is outlined in Appendix B.

The freshly polished and etched specimen surfaces were then observed under 100X magnification and a Polaroid photograph was taken of a representative area of each specimen's microstructure. The photographs thus obtained were used in the determination of ASTM grain size. Detailed information concerning the photographic techniques and grain size determinations is outlined in Appendices C and D, respectively.

A subjective evaluation of porosity was made on the test surface

of each specimen. A score of 0, 1, 2, 3, 4 or 5 was assigned depending on the extent of porosity visually evident under 10X magnification. Casting voids can be caused by a complex variety of factors, such as localized shrinkage or gaseous inclusions. The microstructural effect of a void is essentially the same, whether the hole is neat and round or ragged and irregular. It was not felt that any more information applicable to this study could be gained from a more extensive, sophisticated analysis of this factor.

In order to minimize bias in the porosity scoring, each specimen was unidentified as it was being judged. All specimens were sorted first into three categories indicative of negligible, moderate, and severe porosity. From these three groups, the specimens were further sorted into the six categories ultimately scored. When all the specimens had been sorted in this fashion, they were identified and their scores were recorded. This entire sorting and scoring procedure was repeated on three different occasions, keeping the individual specimen's identity unknown until the sorting was completed. The scores assigned to each specimen on the three sortings were then averaged to the nearest whole number.

Hardness Tests. The metallographically prepared surfaces of each specimen were then subjected to the hardness tests. Plate 4 illustrates the equipment used. A Vicker's diamond pyramid indenter under a 10-kilogram test load was applied with a 20-second descent and a 40-second dwell time. This load was selected in order to obtain sharp, uniform indentations. Nine indentations were made on each specimen's test surface, spaced not less than 1 mm. apart and centered along the long axis. Preliminary hardness tests indicated a sampling of nine

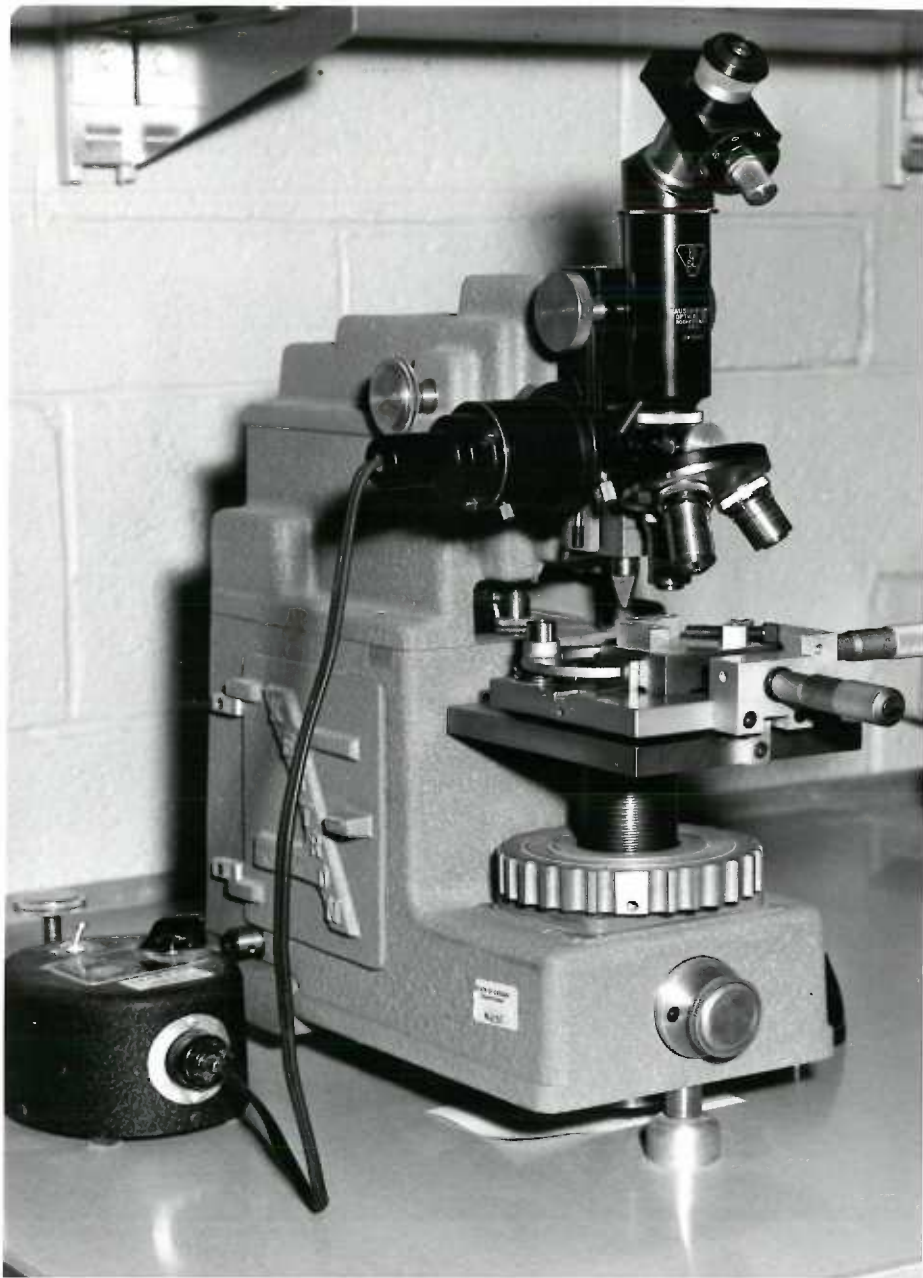


PLATE 4. Kentron hardness tester.



PLATE 6. Instron testing machine.

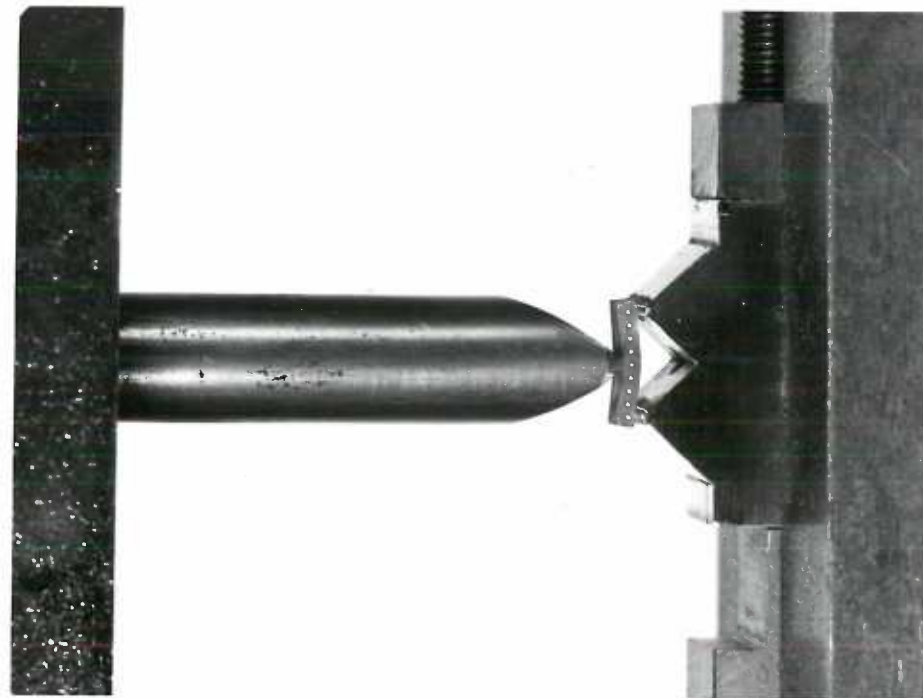


PLATE 5. Transverse test frame. Note hardness indentations on specimen edge.

indentations to be sufficient for detecting differences in the order of five hardness units at the 95% level of confidence.

Bending Strength Tests. At the conclusion of the hardness testing, the cast specimens were separated from their mounting medium and tested for bending strength. A special framework was employed which held the bar-shaped specimen suspended between two steel rods 3/32" in diameter and oriented in parallel 10 mm. apart. Test loads were applied to the specimens through a steel plunger with a 3/32" steel rod affixed to its end. The load rod was oriented in parallel with the support rods and positioned in the center of the specimen. The entire framework, with specimen, was situated under the crosshead of an Instron testing machine. Plates 5 and 6 illustrate this equipment. The rate of cross-head motion was established at 0.01 inch per minute. Calibrated loading force and resultant deformation (bending) of the specimen was automatically recorded on the Instron's chart recorder.

The modulus of elasticity in bending and the proof stress at 0.1% offset for each specimen were calculated from the load-deflection curves plotted on the recorder chart of the testing machine. Modulus of elasticity is defined as the ratio of stress (force per unit area) to strain (deformation per unit dimension). A material exhibiting a high modulus indicates an ability to absorb great stress with relatively little resultant deformation (42, 43, 44). For the bending test system employed in this study, the formula for calculating the modulus is:

$$E_b = \frac{wl^3}{4bh^3y}$$

Figure 3 illustrates these factors.

Proof stress is a measure of the limit of elasticity in a material,

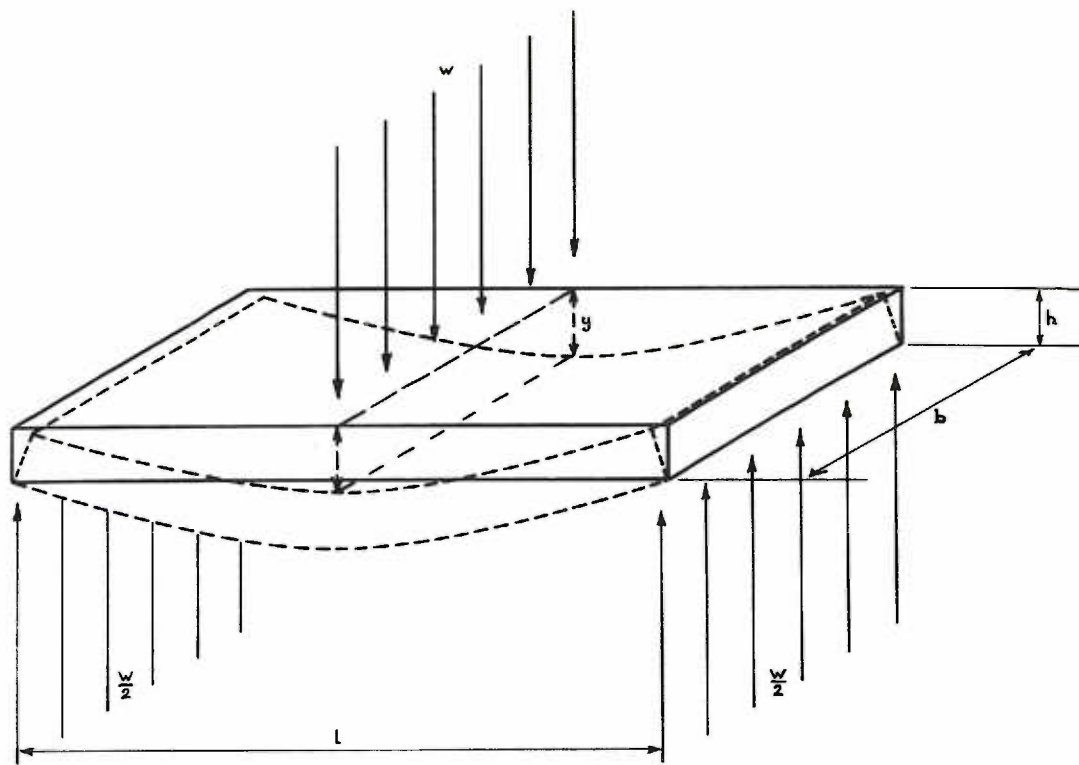


FIGURE 3. Diagram of transverse loading.

or stress beyond which permanent deformation occurs. In ductile materials such as the gold alloys, the point of deviation from linearity of the stress-strain curve is nearly indistinguishable. In cases of this kind, the preferred method is to define the limit of elasticity as the stress at a point on the stress-strain curve intersected by a line parallel to the linear portion of the curve and offset from it by an arbitrary value on the strain axis. This arbitrary value is recorded as the "offset" strain and is always included when reporting proof stress. The formula for calculating proof stress in a bending test is:

$$S = \frac{3wl}{2bh^2}$$

The preliminary bend tests yielded values for modulus in bending which were remarkably lower than theorized. These experimental moduli ranged from 2.5 to 4.5 million psi, while it was expected that the values would be in closer harmony with tensile moduli in the order of 15 million psi. The questionable factor in these determinations was the test beam deflection. The test instrumentation, as applied, utilized the amount of crosshead motion as a measure of beam deflection. In order to justify this practice, it must be assumed that the beam is deflected exactly the same amount the crosshead moves.

An aluminum alloy of known physical properties was obtained and specimens were milled to the dimensions of the gold castings. Reference marks were placed along one edge of each specimen with a Knoop diamond indenter. Each specimen was then subjected to a bend test. An initial load was placed on the specimen to "seat" the loading points. The relative positions of the reference marks were then measured with a measuring microscope. The load was increased a known amount and the

reference mark positions were again measured. The shift in reference mark positions between the initial and final loads was taken as the beam deflection and used in computing the modulus. The mean of ten samples tested in this manner was 9.13 million psi, while the rated value (tested in tension) was 10.0 million psi.

After establishing this means of determining beam deflection, the first fifteen gold test specimens, representing all fifteen test conditions, were measured using the same procedure. The measured beam deflections were plotted graphically versus the corresponding crosshead motions. A routine statistical correlation analysis was performed in order to determine the relationship. A correlation coefficient of 0.948 was obtained. This was considered sufficient correlation to justify computing a correction factor such that crosshead motion could be transformed into beam deflection without the tedious measuring of each specimen. The correction factor was computed to be $(0.486)(\text{cross-head motion, inches}) - (0.00023 \text{ inch}) = \text{beam deflection, inches}$. This factor was employed for all subsequent tests.

The data is shown in Table 1.

TABLE 1

	Mold Temperature Level - °F.	Solidification Time - Seconds	ASTM Grain Size	Vicker's Hardness	Porosity Rating	Proof Stress at 0.1% offset - PSI	Modulus of Elasticity - X10 ⁶ PSI
Alloy A --- (Narrow Melting Range)	700	5.1	4.6	126.4	1	68,900	12.35
	900	7.3	4.4	122.1	2	68,300	12.18
	1100	12.2	4.1	113.4	3	63,100	11.30
	1300	14.7	3.9	121.8	2	57,700	10.97
	1500	18.8	3.6	124.1	2	52,400	8.99
Subsample size = 5, $S_{\bar{x}}$ = ± 0.5 ± 3.1 ± ½ ± 4,063 ± 0.95							
Alloy B --- (Fine Grain Quality)	700	5.1	6.4	133.0	1	69,800	13.74
	900	7.6	6.5	132.3	0	70,200	11.90
	1100	13.0	5.7	126.0	2	62,900	12.51
	1300	15.2	5.4	124.9	2	56,100	10.27
	1500	22.7	4.1	124.3	3	49,700	8.69
Subsample size = 5, $S_{\bar{x}}$ = ± 0.5 ± 2.1 ± ½ ± 4,506 ± 1.25							
Alloy C --- (Wide Melting Range)	700	5.8	5.3	126.0	0	63,600	10.07
	900	8.2	5.5	125.8	1	68,000	11.23
	1100	13.6	6.1	115.2	2	63,800	11.21
	1300	13.7	6.1	109.2	3	59,600	11.26
	1500	19.2	5.6	113.7	3	57,700	11.54
Subsample size = 5, $S_{\bar{x}}$ = ± 0.3 ± 3.7 ± ½ ± 2,523 ± 0.91							

NOTE: Large grain size number indicates fine microstructure; large porosity rating number indicates greater porosity.

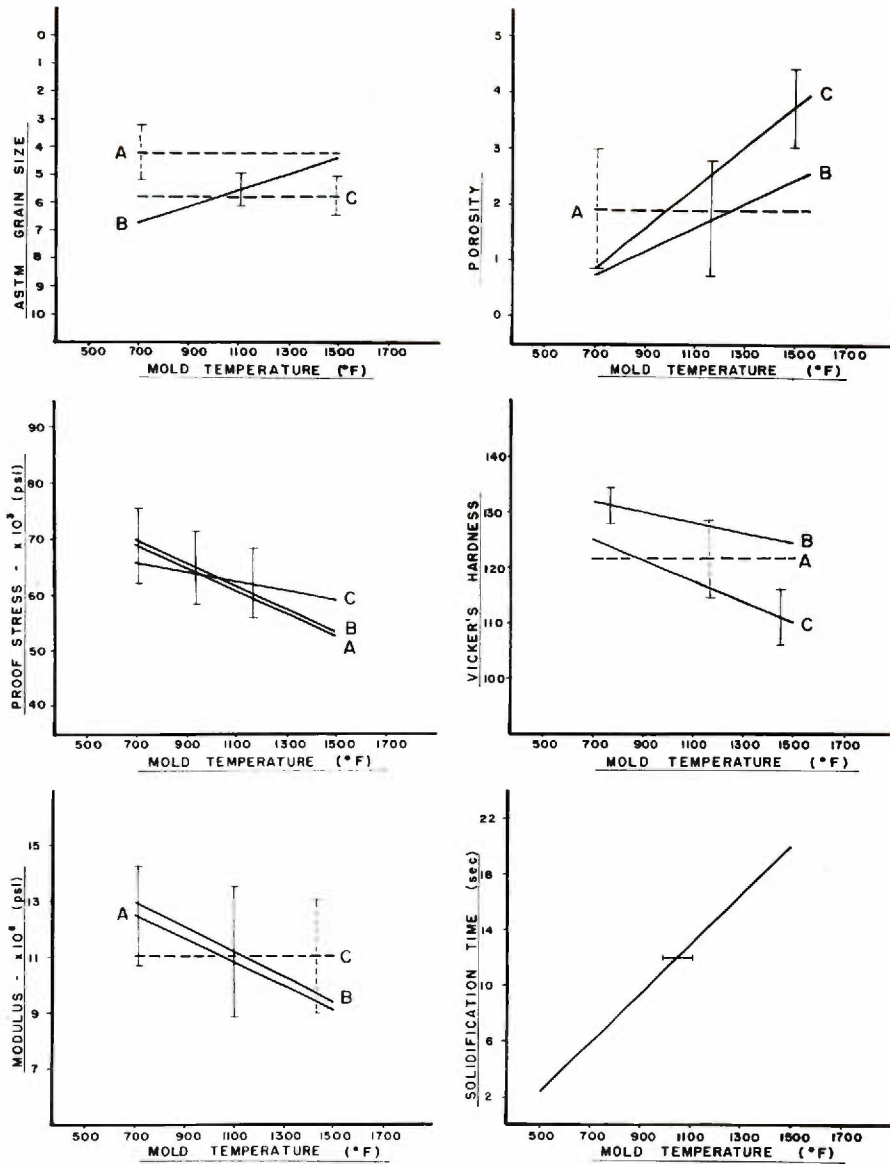


FIGURE 4. Regression lines — Solidification time vs microstructure and physical properties.

RESULTS AND DISCUSSION

General. The regression lines computed from the data are illustrated in Figure 4. Variables with statistically significant correlations are shown as solid lines, while variables without correlations are shown as broken lines. Each regression line's standard error is represented by the superimposed vertical lines. These standard errors have been corrected for the variability imposed by the changes in solidification time.

Grain Size Measurements. The measured ASTM grain sizes of the 25 specimens cast with each alloy were plotted graphically versus their corresponding solidification times. A statistical test for independence of the variables was performed on each alloy. Only one of the alloys, fine-grained Alloy B, showed a significant correlation. Plate 7 shows representative photomicrographs of the specimens' microstructures. The regression line for this alloy was computed and drawn on the graph. As mold temperature and solidification time were shown to be highly correlated, the graph axis was labeled "mold temperature" for illustrative purposes. Using this regression line as a basis for comparison, the grain size of Alloy B varied from an average of 6.6 (48.5 grains per square inch at 100X) at the 700° F. mold temperature level to 4.3 (9.9 grains per square inch at 100X) at the 1500° F. mold temperature level. This is a difference of over ten times in grains per unit volume. The reason that the fine-grained alloy alone demonstrated a

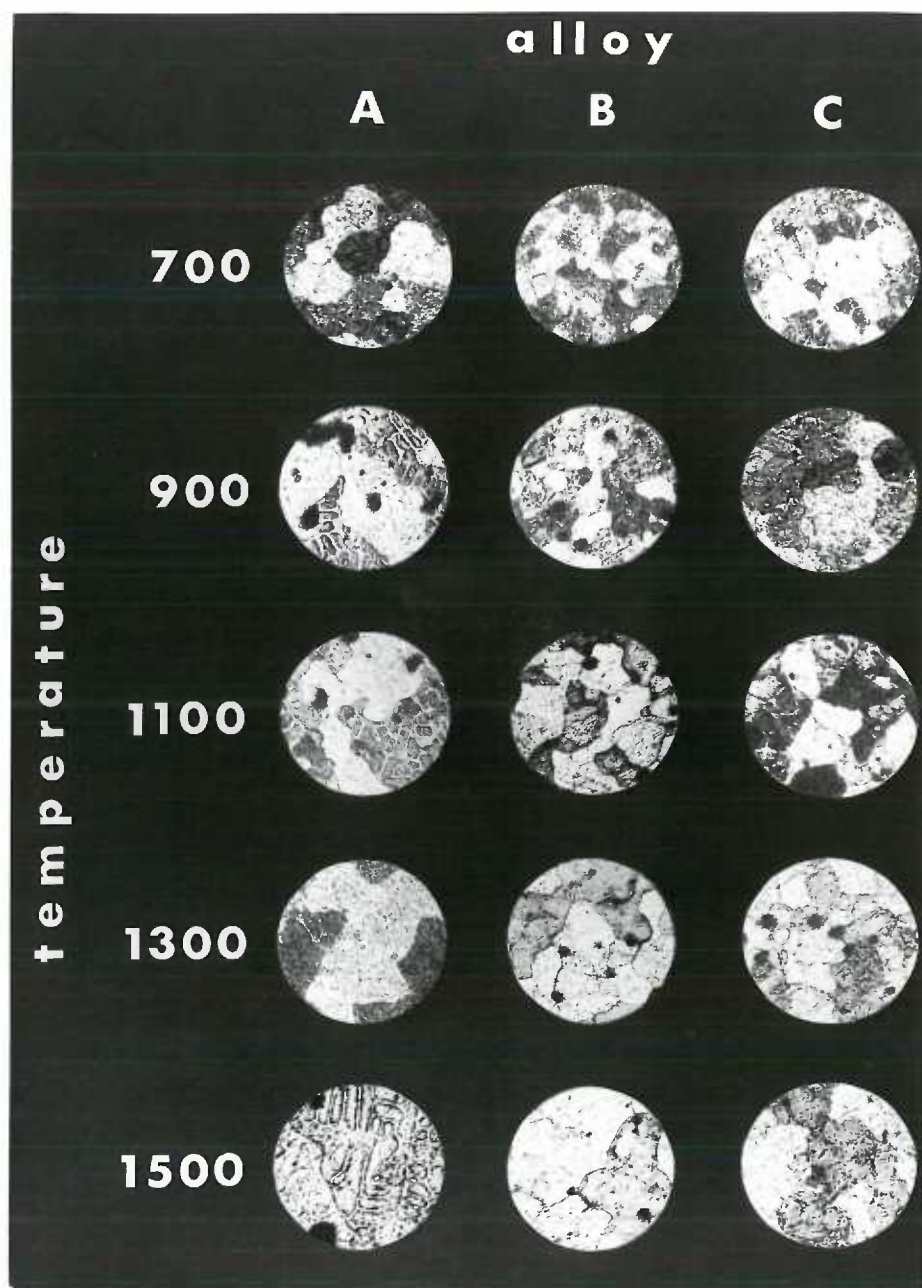


PLATE 7. Photomicrographs at 100 X magnification of the three gold alloys at the five mold temperature levels, illustrating typical microstructures.

significant change in grain size with changes in solidification time was not known.

Porosity Ratings. The assigned porosity ratings for each alloy were plotted graphically versus solidification time and statistically tested for independence. Two of the alloys, B and C, showed significant correlations. Their regression lines were computed and drawn. The results indicate, particularly with the wide-melting-range alloy, C, an increased tendency to exhibit porosity with increased mold temperature.

Hardness Measurements. For the results of the hardness tests, graphic representation and statistical tests of independence showed significant correlations with solidification time for Alloys B and C. The narrow-melting-range alloy, A, again appeared insensitive to changes in mold temperature and solidification time.

Proof Stress and Modulus of Elasticity Measurements. All three alloys demonstrated significant correlations between proof stress and solidification time. Within the mold temperature limits of this investigation, the proof stresses of the alloys changed with solidification time by as much as 16,000 psi, nearly four times the standard deviation of the samples. Between the routinely applied burnout temperatures of 900° F. and 1300° F. this reduction in proof stress amounts to slightly over 12%.

Significant correlations between modulus of elasticity and solidification time were obtained with Alloys A and B. Of probably greater importance, however, are the higher correlations between modulus and proof stress for the same two alloys. Modulus of elasticity is an intrinsic property of a material and theoretically should not be

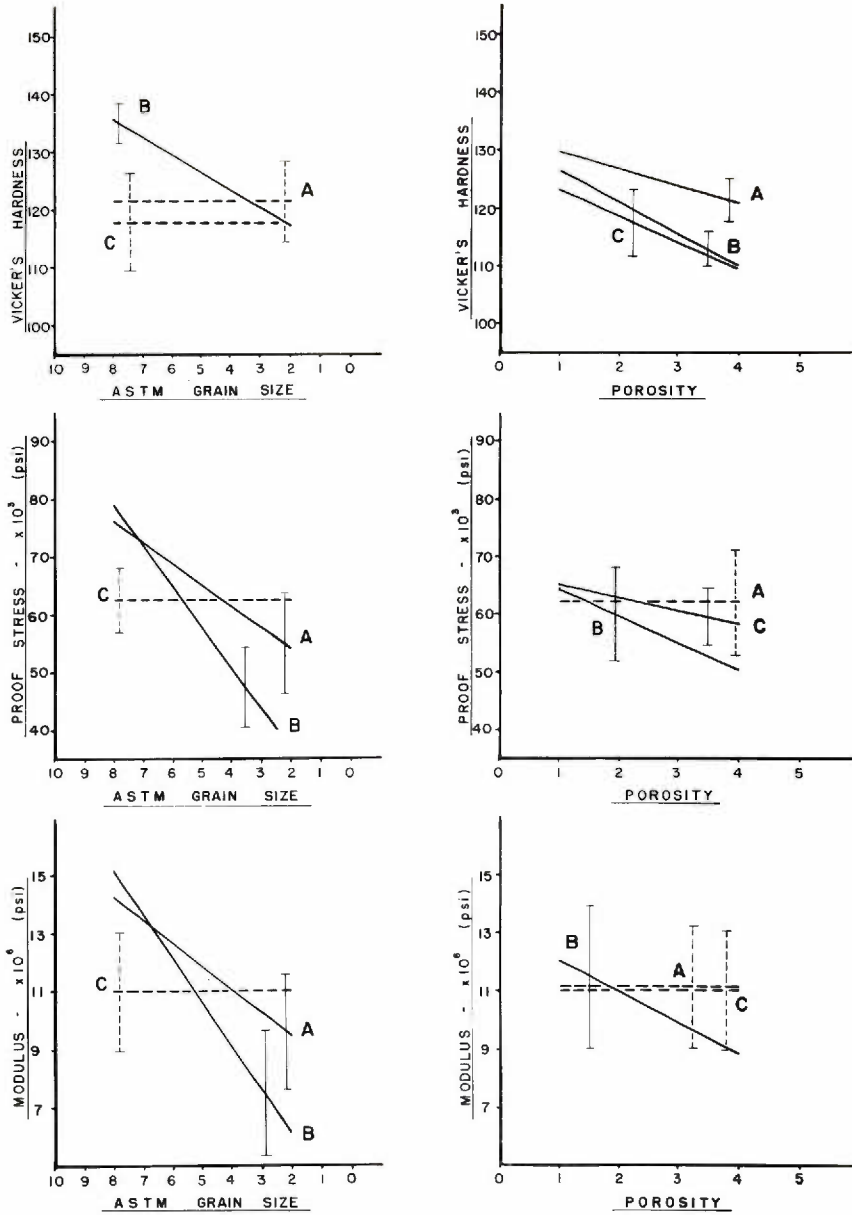


FIGURE 5. Regression lines — Grain size and porosity vs physical properties.

affected by conditions which alter other properties.

Other Observations. In order to demonstrate more clearly the a priori cause and effect relationships, regression analyses of the strength properties (hardness, proof stress, and modulus) and microstructure properties (grain size and porosity) were performed. The regression lines from these correlations are shown in Figure 5. By statistically isolating grain size as an independent variable, it is seen that the strength properties of the wide-melting-range alloy, C, are not affected whatsoever by changes in grain size. By isolating porosity as the independent variable, it is clear that porosity contributes to the decreased hardnesses and bending strengths of the alloys as much as does grain size. With still other factors to be considered, it would then seem presumptuous to state that grain size alone determines the strength. To the contrary, within the limits of this investigation, grain size has been shown to influence the strength properties little more than porosity. The traditional requirement of fine grain size in castings may not be of the singular importance heretofore believed. In addition, the special fine-grain quality imparted to currently popular alloys by their manufacturers may even cause greater variability in the strength properties, as evidenced by the higher standard deviations of the proof stress and modulus in fine-grained Alloy B. This quality appears to improve only the microstructural aesthetics of these alloys. It would be of interest to test Alloy B sans the fine-grain quality.

Discussion of Results. In general, higher mold temperatures slow the solidification process. The longer it takes for the alloy to achieve solidification, the greater is the tendency to develop larger grains and

more extensive porosity. These combined factors, in addition to the unexplored effects of different compositions, cause a decrease in hardness and a decrease in proof stress. Modulus of elasticity, while affected, appears to follow the pattern set by the proof stress and cannot be logically correlated to any other factor.

The clinical significance of these property changes can only be speculated. It is possible that these lowered strengths may still be well within the limits of acceptability. However, an effort on the part of the operator to strive for the best possible results is indicated. It should be emphasized that the duration of the solidification process can be an appreciable length of time. It has been demonstrated that dental gold castings may not achieve complete solidification for upwards of 15 to 20 seconds after cast. This fact indicates a necessity for maintaining pressure on the alloy at least this length of time in order to adequately compensate for solidification shrinkage. It would not be unreasonable to surmise that larger castings would take even longer to achieve complete solidification. The practice of "hurrying along" the recovery of a casting by releasing pressure within a few moments after cast is definitely contraindicated, based on these results.

SUMMARY AND CONCLUSIONS

Summary. The solidification and cooling curves of three inlay gold casting alloys were measured from clinical-sized castings made in an investment mold at various mold temperatures. The sample castings were metallographically polished and observed under 100X magnification in order to determine the grain size of the microstructure. An evaluation was made of the extent of the porosity evident in each casting. Vicker's hardness tests and transverse (bending) strength tests were performed on each sample. The data was analyzed using a multiple regression model. Statistically significant correlations were found between the times of solidification and the strengths of these alloys. In general, as the solidification process was prolonged, the grain size and extent of porosity were increased. Likewise, the hardness, bending strength, and modulus were decreased.

General Conclusions. Based on the results of this investigation it can be concluded that dental gold alloys cast at higher mold temperatures (as opposed to lower mold temperatures) in general:

- A. Exhibit a large grain structure and marked porosity which, in addition to other factors, causes a significant decrease in hardness and bending strength.
- B. Vary in strength properties from one casting to the next. This variability appears to be greater in alloys possessing a special fine-grain quality.

Other Observations. Dental castings may require as much as 20 seconds to completely solidify in the mold, depending on the mold temperature and the size of the casting. Also, type III casting gold alloys tested for bending strength have a modulus of elasticity in the order of 11 to 12 million psi, and a proof stress in the order of 55,000 to 60,000 psi. Vicker's hardness of type III casting gold is between 120 to 130 hardness units.

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APPENDIX ARelationship Between Mold Temperature and Cooling Time

The selection of mold temperature as a means of regulating the alloy solidification time was confirmed in a series of preliminary tests. Twenty castings were made with an available Type C gold alloy (Crown Supreme, General Refineries). Mold temperatures were varied from 500° F. to 1500° F. Instrumentation and procedures for these test castings were standardized and subsequently employed in the actual experiments.

Although the furnace used for heating the molds was pre-set for a specific temperature level, it was found that the actual mold temperature as measured by the thermocouple was frequently different from the temperature indicated by the furnace dial. It was felt that the actual mold temperature was of primary importance; therefore, the thermocouple values were used exclusively for establishing mold temperatures throughout this study.

A routine statistical correlation was performed between the thermocouple-measured mold temperatures and the corresponding gold alloy solidification times. The data appears as Table 2 with this appendix. It was found that the coefficient of correlation for these variables was 0.986. This value was considered to indicate a degree of correlation sufficiently high to accept mold temperature as a means of varying solidification rate for this study.

TABLE 2

(Actual mold temperature - ° F.) (Solidification time - seconds)

<u>X</u>	<u>Y</u>
471	2.7
519	2.5
528	3.0
632	4.8
658	4.0
658	5.8
837	7.3
854	8.0
863	7.6
863	9.1
1002	11.0
1032	10.0
1032	13.0
1201	15.7
1244	16.3
1256	16.4
1377	15.5
1390	17.0
1390	18.0
1468	20.5

$$N = 20$$

$$\Sigma X = 19275$$

$$\Sigma Y = 208.2$$

$$\Sigma X^2 = 20571639$$

$$\Sigma Y^2 = 2797.72$$

$$\Sigma XY = 235566.2$$

$$\bar{X} = 963.8$$

$$\bar{Y} = 10.4$$

$$S_X = 324.1$$

$$S_Y = 5.75$$

$$b = \frac{\Sigma XY - \frac{\Sigma X \Sigma Y}{N}}{\Sigma X^2 - \frac{(\Sigma X)^2}{N}} = 0.0175$$

$$r = b \frac{S_X}{S_Y} = 0.986$$

APPENDIX BMetallographic Preparation of Specimens

In order to prepare the surface of each specimen casting for microscopic examination, it was necessary to devise an adequate and standardized procedure. A basic requisite for surface preparation is that the procedure employed must remove all "disturbed" metal and expose a fresh "undisturbed" plane through the true microstructure of the metal. That is, sufficient surface metal must be ground off to ensure that irregularities, strained areas, and artifacts are removed.

Although many principles exist in the metallographic preparation of specimens, the specific procedures are largely empirical and depend on the judgment of the operator. For this study, reference was made to several texts and technical bulletins for guidance during the preliminary testing.

Preparatory to surface finishing, the cast specimens were mounted in Bakelite. The five specimens from each condition of test were set on edge in the mounting mold and raw Bakelite powder was poured around them. A plunger was positioned into the mold and 3500 pounds of pressure was applied with a hydraulic press. Simultaneously, heat was applied to the mold by a surrounding element. After the Bakelite had undergone its polymerization reaction, the mounted specimens were ejected from the mold for finishing.

The procedure ultimately established for this study is outlined

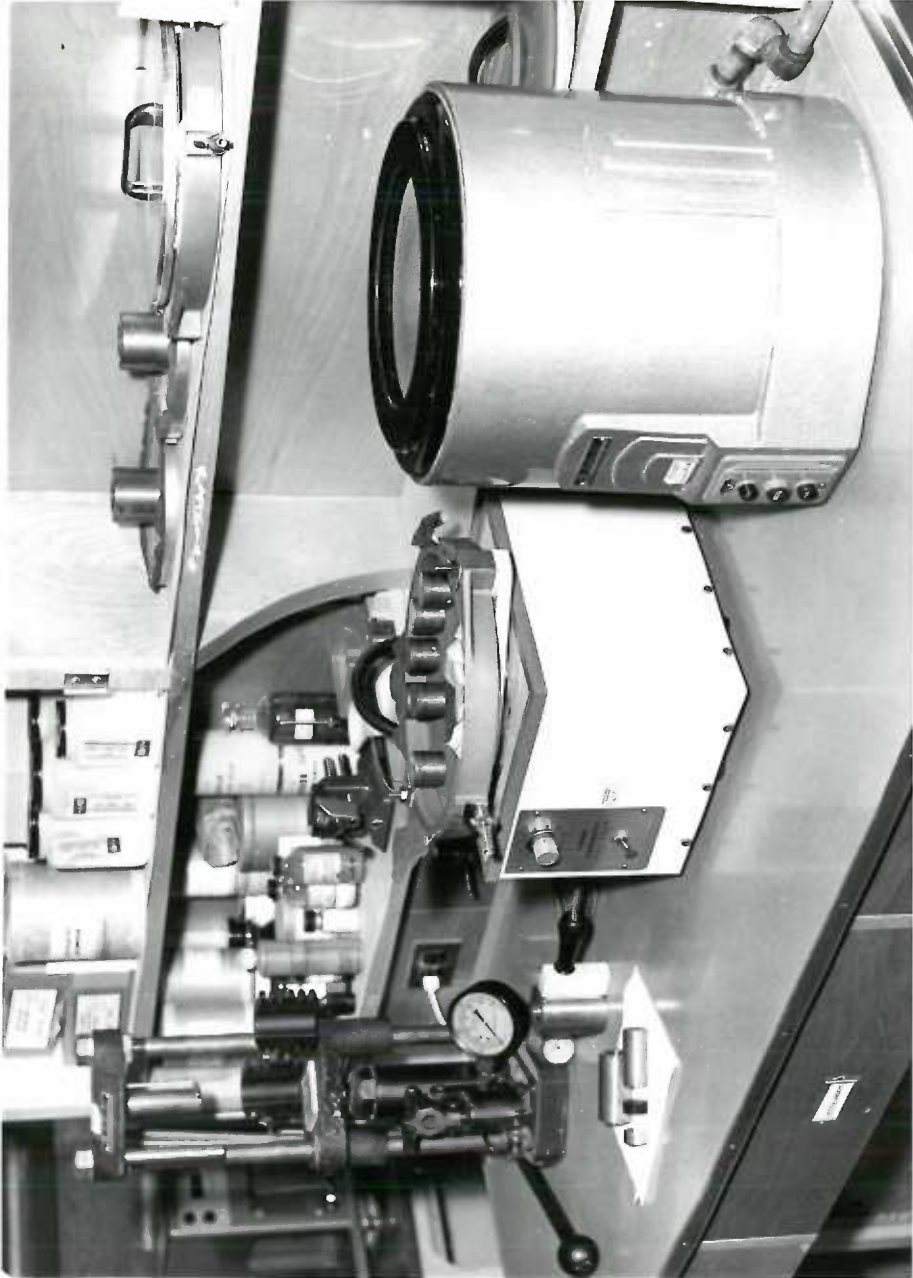


PLATE 8. Metallographic polishing equipment.

in Table 3. Briefly, the experimental edge of each cast specimen was drawn across a sheet of emery paper under moderate pressure, keeping the direction of motion constant. When the scratches left by the first emery appeared uniform in direction and size, the next finer grade of emery was used. The specimens were then drawn across this emery paper at right angles to the first paper's scratches. By changing the direction of grinding between each grade of abrasive, it was relatively easy to see when the previous paper's scratches had been removed.

Rough polishing was accomplished with a felt wheel and alumina. Because gold alloys easily "smear" during directional polishing and create an optical phenomenon called "comet-tails," fine polishing was done in a vibratory polishing bowl. The vibratory polisher is essentially a shallow bowl that is mechanically vibrated vertically and horizontally at the same time. The result of these motions is a pulsing which causes a weighted specimen to rotate on its own vertical axis while moving around the bowl in the opposite direction. Thus, a specimen placed in a slurry of the appropriate polishing abrasive moves relative to the abrasive in a constantly changing manner, eliminating the directional effect. Plate 8 shows the equipment used.

A ten-percent solution of potassium cyanide and ammonium persulfate was used as a preliminary etchant (45). The persulfate is selective in its attack on the metal surface, removing all random unit cells both at the grain boundaries and between dendritic branches. Because of this, it was virtually impossible to distinguish precisely where the grain boundaries lay. A gross etch, aqua regia, was employed as a final etchant. This reagent attacks the entire structure and clearly outlines the microstructure (46).

TABLE 3

Metallographic Sequence

<u>Abrasive Step</u>		<u>Abrasive & Media</u>		<u>Abrasive Particle Size</u>
1. Medium grind	--	240 grit paper	--	45 μ
2. Fine grind	--	400 grit paper	--	25 μ
3. Fine grind	--	4/0 paper	--	15 μ
4. Rough polish	--	wheel, levigated alumina	--	<15 μ
5. Rough polish	--	wheel, Al ₂ O ₃	--	5 μ
6. Fine polish	--	bowl, micropolish	--	1 μ
7. Etch	--	60 sec - persulfate	--	
8. Fine polish	--	bowl, micropolish	--	1 μ
9. Etch	--	60 sec - persulfate	--	
10. Fine polish	--	bowl, micropolish	--	1 μ
11. Final polish	--	bowl, gamma Al ₂ O ₃	--	.05 μ
12. Final etch	--	120 sec - aqua regia	--	



PLATE 9. Leitz microscope and camera.

APPENDIX CPhotomicrographic Data

The photographs of specimen microstructure for this study were obtained with a Leitz camera and microscope. A magnification factor of 100X was established by using an 8X microscope objective, a 6X photo eyepiece, and extending the camera bellows to 432 millimeters. The magnification was calibrated by observing a cathetometer calibration slide marked in 0.01 millimeter increments through the ground-glass viewer of the camera and adjusting the bellows extension until the image corresponded to 100X as measured with a precision scale. Several photographs were then taken and measured carefully to check this setting.

Type 52 Polaroid 4 x 5 cut film was used for the photographs. Exposure times varied with the specimens, but averaged about one second. Each photograph was fixed with the agent supplied with the film.

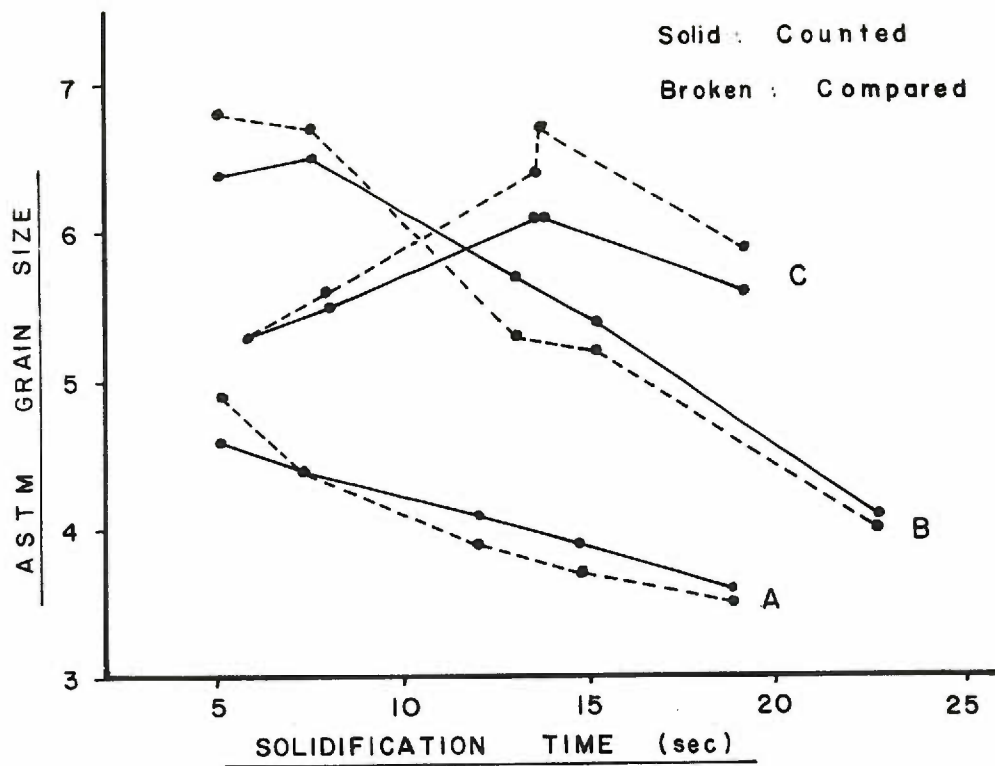


FIGURE 6. Mean values — Grain sizes, counted and compared.

APPENDIX D

ASTM Grain Size Determination

The American Society for Testing Materials has accepted an exponential function of grain density as an index or grain size standard. By definition, a microstructure is observed at 100X magnification. The number of grains per square inch at this magnification are transformed into an index number by the formula X grains per square inch = 2^{n-1} , where "n" is the ASTM grain size number. By convention, the ASTM grain size is rounded off to the nearest one-half. Thus, grain sizes are expressed as 0, $\frac{1}{2}$, 1, $1\frac{1}{2}$, 2, etc.

In this study, grain size was determined by counting the grains and partial grains enclosed in a one-inch square superimposed and randomly oriented over the photograph (taken at 100X magnification). The number of grains counted was then converted to an ASTM number and rounded to the nearest one-half.

Later in the study, the coded photographs were randomly compared to ASTM standard grids, a practice more widely accepted but less precise than counting. Each photograph, unidentified to minimize bias, was assigned an ASTM number by the comparison method. The results of the ASTM grain size assignments by counting and matching were then compared to check the validity of the systems. The data is shown graphically in Figure 6. Each point represents the average of five specimens. As can be seen, the results are similar and demonstrate the accuracy of the standard grid matching method.

