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on

Properties of Dental Amalgam and Gallium Alloys
Produced by Using Spherical Alloy Powders

by

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U. S. DEPARTMENT OF COMMERCE
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Properties of Dental Amalgam and Gallium Alloys Produced by Using Spherical Alloy Powders

Abstract

Spherical alloy powders were used in an investigation of the effect of alloy particle size on the properties of dental amalgam and gallium alloys. The particles were separated into eight increments of particle diameter ranging from approximately 1 micron to 150 microns. Particle diameters had a predominant effect on the mechanical and physical properties. This effect differed in the two alloys. It was also found that the trituration time had an important effect upon the development of maximum strength in these alloys. For dental amalgam alloys the minimum trituration times required for development of maximum strength are usually less than one minute but the gallium alloys require a minimum of about three minutes in order to develop maximum strength. However, near maximum strength may be produced from alloys triturated for two minutes. After determining the correct trituration time for each particle size, specimens were prepared and tested for shear strength, compressive strength, tensile strength, setting changes and setting time as a function of particle size. The results indicated that the strength of alloys produced by using spherical alloy powders of the optimum particle size range may exceed that of the best commercial alloys.

1. INTRODUCTION

The silver-tin-mercury alloy commonly known as dental amalgam is the most extensively used restorative material for carious teeth because it has given good clinical service, is easy to prepare for insertion into the tooth, and it permits a considerable saving of the dentist's time and effort when compared with the alternative use of gold inlays or of gold foil.

It has been known for some time that the simple methods of alloy preparation used with dental amalgam may be used with other alloy combinations involving a liquid metal and a solid alloy powder which are mixed and allowed to react at mouth temperature. In recent years the use of liquid gallium has been proposed and several gallium alloys have received consideration as potentially useful materials for dental restorations [1]. The gallium alloys have been found to possess a superior wetting action, when contacting the tooth structure. Compared to dental amalgam they also exhibit high strengths at elevated temperatures, have high compressive strength, and have a small differential thermal expansion between the tooth and the alloy filling [2]. It is not yet known whether the corrosion resistance and biological tolerance of these materials will be satisfactory in the oral environment. In certain alloys an exothermic reaction occurs when extremely fine powders are mixed with liquid gallium, but recent research indicated that this reaction may be satisfactorily controlled.

The mechanical properties of dental amalgams have been known to be greatly dependent on the size and shape of the particles constituting the alloy powder [3,4,5,6]. Demaree and Taylor [7] were the first investigators to study the properties of dental amalgams made from spherical alloy powders in graded increments of particle size. Their work has shown that particle size control is a very effective means for controlling properties in dental amalgams.

In the present investigation particle size control is used to develop the optimum mechanical properties in a new and entirely different alloy system. In this system spherical powders of palladium take the place of the spherical silver-tin alloys used by Demaree et al, and they are mixed with a liquid (89% gallium + 11% tin) eutectic alloy rather than with mercury. New tests for shear strength, tensile strength, and initial setting time have been developed and applied to both the ordinary silver-tin-mercury alloys and the palladium-tin-gallium alloy.

Since the mechanical properties of these alloys are dependent upon their trituration times it is necessary to determine the optimum trituration time for each alloy and for each particle size. A comparison of the test results is then made using the trituration time which most enhances these values in each alloy system.

The new tests for shear strength, tensile strength, and initial setting time when combined with the conventional tests for compressive strength and setting change provide a comprehensive description of these amalgam-type materials. The results show that particle size control is essential for the development of the most desirable physical and mechanical properties in "amalgam-type" materials.

2. EXPERIMENTAL METHODS

2.1 Preparation of spherical powders.

The silver-tin-spherical particles described by Demaree and Taylor [7] were also used in this study. These particles were made by the Federal Mogul Division, Ann Arbor, Michigan by a patented atomization process from an experimental silver-tin-copper-zinc alloy with a composition typical of dental amalgam alloys in use.

The spherical palladium powder was produced from three palladium ingots having a total weight of approximately 5 pounds and a nominal purity of 99.9%. These ingots had been vacuum melted in a high frequency furnace and were slowly cooled in the alumina crucibles used to contain the melt. They were made into spherical powders by the Federal Mogul Division. A metal loss of less than 1% was incurred in the atomization. The spherical palladium powder was supplied in two fractions of plus 100 mesh and minus 100 mesh. All of this material, received in spherical form, was separated into various increments of particle diameters. The increments of larger size particles were separated by means of U. S. Standard Sieves ranging in sieve number from 50 to 325. The portion of the material that passed a number 325 sieve was separated by elutriation. The elutriation flow rates used by Demaree and Taylor [7] were increased by 25 to 30% in order to collect fractions containing particles with the same size ranges as were obtained with silver-tin alloy. These increased flow rates were necessary since palladium has a specific gravity of 12.02, whereas the silver-tin alloy has a specific gravity of approximately 8.33.

Table 1 summarizes the separation of spherical particles of both the silver-tin and of the palladium powder into the various diameter ranges. These increments of particle size are hereafter called size 1, 2, etc.. The silver-tin particles showed a peak of distribution in size 4 which constituted 26.6% by weight of the total material. The palladium particles had a peak of distribution in size 8 constituting 22.9% by weight of this material. Figure 1 contains photomicrographs of the spherical palladium powders in sizes 2, 4, 6, and a combination of size 4 (2/3 by wt) with 1-2 micron precipitated powder (1/3 by wt).

2.2 Specimen preparation.

Specimens were made from each of the various sizes separated, except palladium powder sizes 9 through 11. The equipment used for specimen preparation is shown in Figure 2. The powder and liquid were pre-weighed into gelatin capsules (A) with the exception of the liquid gallium eutectic alloy which was dispensed directly into the mixing capsule by means of a micrometer buret.* The powder-liquid ratio was kept constant for all tests. For shear and compressive strength specimens, 0.90 ± 0.03 grams of mercury was added to 0.60 grams of silver-tin alloy and similarly 0.15 cc (0.92 ± 0.05 grams) of gallium-tin alloy was added to 0.70 grams of palladium. Each of these amounts was increased by an additional one-third for the setting changes and the tensile specimens. The powder and liquid were mixed in an automatic S. S. White amalgamator #1 (B), controlled by an electronic timing device (C).** The mixing time used was determined individually for each size as that time which produced the highest compressive and shear strengths in 24 hours. This procedure will be explained in detail later in this report. After trituration the mixed mass was gently inserted into a cylindrical mold (D); a condensing rod of 0.0010 ± 0.0001 inches clearance was placed into the mold and after 1/2 minute had elapsed from the end of trituration, a load of 150 kg/cm^2 was applied to the compressive (8), shear, initial setting, and setting change specimens by the press (E). A load of 390 kg/cm^2 was applied to the tensile specimens made in mold (H). After 3 1/2 minutes the load was removed and the specimen recovered from the mold.

* Manufactured by Roger Gilmont Instrument, Inc., 1 Great Neck Road, Great Neck, New York.

** Gra Lab Universal Timer, Type 172, manufactured by Dimco-Gray Co., Dayton, Ohio

The length and the diameter of the specimen were measured with a micrometer just prior to testing. The specimens made were limited to compressive-type or slightly modified compressive-types.

When the palladium-gallium eutectic specimens were made, a micrometer buret (F) was used to dispense the 89% gallium + 11% tin eutectic alloy (Ga_E) to an accuracy of 0.01 ml. This liquid metal was dispensed directly into the Teflon capsule (G) which is a hollow tube with two male end plugs. The material, after mixing, was pushed out by means of a fitted Teflon rod.

2.3 Biological investigation.

Concurrent with this investigation of mechanical properties, a biological investigation of corrosion, tissue tolerance, and suitability of palladium-gallium-tin alloy to the oral environment was conducted at the Naval Medical Research Institute. The biological report will be presented in a separate paper when hard and soft tissue sections are prepared for interpretation.

2.4 Testing procedures for setting change.

The setting changes of 1 cm long cylindrical specimens with one cone-shaped end were measured on an interferometer in a constant temperature enclosure at 37° C. Specimens were prepared using a compressive strength specimen mold with a substituted mold rod, which had one end recessed in the shape of a cone. To arrive at the 1 cm length the amount of material was increased. Ratios of 1.20 grams of mercury to 0.80 grams of the silver-tin alloy and 0.20 cc (1.23 grams) of Ga_E to 0.93 grams of Pd were used to maintain the same powder liquid ratio. Several sizes were mixed for 20, 120, and 180 seconds. After condensation the cylinder containing the specimen was inverted upon the washer-like spacer shown in Figure 2 (D). This controlled the length of the extended cone-shaped rod and the amount of specimen that remained within the end of the mold. The spacers were removed and the rod was pushed further into the mold ejecting the specimen.

The fiducial interferometer reading was taken 6 minutes from the end of mixing and was considered the start of the setting change. This was 2 minutes from the end of condensing.

Readings were taken every 3 minutes for 21 minutes, then every 15 minutes for several hours, and finally, with prolonged setting changes, periodic series of 15 minute readings to establish the rate of these changes. On stabilizing, three daily readings were taken at intervals of 4 hours.

2.5 Testing procedures for compressive strength.

Twenty-four hour compressive strength tests were conducted on the amalgam specimens, made from the various particle sizes and triturated for controlled amounts of time. A capsule without a pestle was used to obtain slow mixing for better evaluation of amalgam. Each size of amalgam was mixed for 20 and 120 seconds and some selected sizes were mixed for 60 and 600 seconds. After 24 hours three to six specimens of each size mixed were loaded in compression on an Instron testing machine at a head speed of 0.01 inches per minute at a temperature of 23.0 ± 1.5° C. The breaking time of these specimens was less than 1 1/2 minutes.

Compressive strengths of Pd-Ga_E were measured after 24 hours at the same rate of loading and at the same temperature. Various particle sizes were mixed in a Teflon capsule for 20, 30, 45, 60, 90, 120, and 180 seconds.

2.6 Testing procedure for tensile strength.

Tensile strength was studied using a centrifugal apparatus, a new method introduced by W. T. Sweeney and J. Kumpula [9]. The apparatus and the rotor are shown in Figure 3 and 4. The ratio of powder to liquid was maintained but the amount of each was increased by one-third that used for the compressive and shear specimens. Ratios of 1.20 grams of mercury to 0.80 grams of the silver-tin alloy and 0.20 cc (1.23 grams) of Ga_E to 0.93 grams of Pd were used. The increase in amount of material was required by the change in the size of specimen from approximately 4 by 8 mm to approximately 2.5 by 25 mm depending on the particle size and the amount of trituration used. Both the particle size and the amount of trituration affected the external dimensions of the specimen. The tensile mold, which is shown as components (H) and assembly (I) in Figure 2, is a split mold for easy removal of the long thin cylindrical specimen. The specimen was cut to 19 mm before inserting into the horizontal slot on the top of the rotor for testing. This rotor constitutes part of an air-turbine in which the speed may be controlled by regulating the air pressure.

The resulting centrifugal force on the specimen is gradually increased until the specimen fractures. When the specimen fractures its free end moves against the end of the slot and dislodges the rotor. Light reflected from the polished half of the surface of the rotor strikes a photo cell once each rotation. The photo cell signal is transmitted to a frequency meter, for conversion into revolutions per minute. A Brown potentiometer recorder also traces out the pattern of the loading in terms of rpm vs time. More specific details are being prepared for another report [9].

2.7 Evaluation of initial setting.

In the continuous setting reaction, the initial setting may be defined as an arbitrary firmness at which point an experienced dentist would consider the mixture no longer able to be contoured or carved except by sharp or rotary instruments. The length of time needed to attain this arbitrary firmness would be the initial setting time or the end of carving time. When using commercial dental alloys containing irregularly shaped particles it is quite difficult to design a dependable laboratory test. J. Kumpula and G. C. Paffenbarger attempted to relate setting time to the length of indentations made in an elongated triangular bar formed from triturated conventional alloys without expressing mercury. Their device with modifications, as shown in Figure 5, was again used in this investigation.

In developing the procedure for measuring initial setting time, two cylindrical specimens were made simultaneously using the technique described previously for compressive specimens, except that the condensing time was 1/2 minute. One of these cylindrical specimens was placed in a V trough. A wedge shaped blade, Figure 6, was impressed upon the curved surface of the specimen by a load of 100 ± 10 grams at one minute intervals for 30 seconds duration. The other specimen was carved with a Hollenback #3 carver. When the carved specimen was considered to be no longer carvable, the simultaneous wedge indentations were noted. A few more indentations were then made and the corresponding times recorded. These horizontal markings along the curvature of the specimen were then measured using a microscope. The length of the impressions were found to be 0.675 ± 0.075 mm for all specimens when they were considered to be initially set on the basis of the carving test.

To further refine the determination of the time of initial setting, each specimen was loaded at 15 second intervals for a duration of 10 seconds. These loads were applied from one minute before to one minute after the presumed initial setting that was previously measured when correlated with carvability.

2.8 Evaluation of shear strength.

2.8.1 Specimen preparation.

Shear strength was measured by the load placed on a cylindrical specimen 4.00 ± 0.02 mm in diameter by 8 to 10 mm in length. The length depended on the particle size and the trituration time. When specimens expanded on setting, the essential diameter for testing was attained by using an undersized mold. The regular diameter was 4.00 mm and the other diameters were 3.96, 3.92, and 3.88 mm. The setting change tests gave information as to which mold would be used to produce a 4.00 ± 0.02 mm diameter at the desired time of shearing. The apparatus accomodates a diameter of 4.02 mm, but the diameter should not be smaller than 3.96 mm at the time of shearing to fit properly. Specimen bending, slippage, and the concentration of force upon small areas were thus minimized. Initially specimens were made from mixtures of 10, 15, 30, 45, 60, and 90 seconds of trituration. Twenty-four hour shear strengths were measured on these specimens to establish the mixing time to be used for further shear test specimens.

2.8.2 Description of shear apparatus.

The components of the shear apparatus are shown in Figure 7. The housing (A) has a slot in the top to guide the blade (B). The two pins (C) placed into the upper holes in the face of the housing align the blade so that the specimen (D) may be placed in the hole in the middle of the face of the housing. The specimen is centered in the blade and housing by means of the two aligning rods (E). Pins (C) are then removed. This housing is then slipped into the frame (F) and the aligning rods (E) are held firmly by the set screws (G). This assembly is placed in the testing machine as shown in Figure 8. After shearing, the specimen was removed in three pieces. A close up of this is shown in Figure 9, in which the two free ends are above and below, and the middle portion is in place. The three sections in the foreground show the relative sizes of segments.

2.8.3 Testing procedure.

The specimens sheared at the initial setting time, were loaded at a head speed of 0.05 inches per minute at a temperature of 23.0 ± 1.5° C. Others of each size were sheared at 1/2, 1, 6, and 24 hours and loaded at a rate of 500 pounds per minute in a temperature of 26.5 ± 1.5° C. The specimen broke into three pieces. This was accomplished by a broad 4 mm blade, as wide as the diameter of the specimen. The load was thus applied on two cross sectional areas for better stability of the blade and less distortion of forces on the specimen. The load was applied and considered double shear, since the load was received by two cross-sectional areas. The shear apparatus positioned in the Instron testing machine is shown in Figure 8.

2.8.4 Photomicrographic surface examination.

Surfaces of amalgam and of Pd-Ga_E specimens were observed during the hardening period by photomicrographic methods. Amalgams made from particle sizes 2, 4, 8 and 8 (2/3 by wt) mixed with 4 (1/3 by wt) were triturated for 20 seconds and 120 seconds. The mixture was spread on a glass slide using the surface of another slide as a trowel. Slides containing the mercury-rich mixture flattened on the surface were photographed at a magnification at 100 X at 5, 8, 15, 30, and 60 minutes after the end of trituration.

Similar photomicrographs were also made of the end surfaces of compressive-type specimens made from amalgam mixed as above and triturated for 20 seconds and Pd-Ga_E specimens made from particle sizes 2, 4, 6, and 4 (2/3 by wt) mixed with 1-2 micron precipitated powder (1/3 by wt) triturated for 120 seconds. Each of these mixtures was condensed as described for compressive specimens with the exception that time of condensation was 1/2 minute.

3. EXPERIMENTAL RESULTS

3.1 Packing characteristics.

Packing characteristics of the mixed mass were observed during specimen preparation. In general, measuring the length of the specimen with a micrometer indicated the packing quality,

and measuring the diameter indicated the dimensional change within 0.01 mm. The diameter of the mold fixed the starting diameter of the specimen. The end rods moved so the length of the specimen varied, depending upon the total reaction of the mixed mass up to the time that the mold rod was loaded. As the load was applied, the rod moved down on the mass in the mold with continuous advancement until coming to a sudden definite stop.

With the small particle sizes, such as 2 and 3, the mixed mass became firmer as the time of mixing was increased from 60 to 90 seconds and was stiff when mixed for 120 seconds. The resulting specimen was longer because these specimens packed with surface voids (and probably internal voids) under the constant load. Figure 10 shows four specimens made from material mixed for 20 and 120 seconds. In sizes 6, 7, and 8 as the time of mixing was increased to 120 seconds, the very soft runny mixed mass became more viscous, although the mixed mass was still soft. The resulting specimen was shorter in length.

The surface texture was modified mainly by particle size, but also by trituration as can be seen in Figure 10. The size 2 specimen after having been triturated 20 seconds was smooth on the surface, while the specimen triturated 120 seconds showed surface voids. The size 8 specimen of the 20 seconds of mixing was pebbly and that of the 120 seconds was smoother.

3.2 Results of setting change tests.

Setting change was studied at different mixing times. Table 2 shows the values for the mixing times of 20 and 180 seconds for three sizes of amalgam and three sizes of Pd-Ga_E. The amalgam made from size 3 contracted more at 180 seconds of mixing than at 20 seconds of mixing. Where there was expansion of the specimen, as in size 6, there was greater expansion in 180 seconds of mixing.

A drastic reduction of expansion, approximately 50%, was noted in the Pd-Ga_E setting change when the mixing time was increased from 20 to 180 seconds. There was a mechanical heating effect of the automatic amalgamator, especially after 60 seconds. All Pd-Ga_E specimens had positive setting changes as shown in Figure 11. Pd-Ga_E spherical powders of size 4 with 1-2 micron precipitated powder had a setting change of plus 104 microns and had the best mechanical properties when

the mixing time was 120 seconds. When mixed for 180 seconds the setting expansion was 87 microns and there was a decrease in mechanical properties.

3.3 Results of initial setting time tests.

The initial setting time of amalgam with respect to particle size is shown in the upper portion of Figure 12. The setting time varied from 2 minutes to 28 minutes and 30 seconds. From size 4 to 8 the time of setting increased linearly with the increase of particle size.

3.4 Results of shear strength tests.

The shear strength measured at the initial setting time for the particle size is shown in the lower part of Figure 12. This is a straight line at 1,800 ± 100 psi except for size 1 which is 2,300 psi. The shear strength at initial setting time was 10% of the 24 hour strength of two commercial control alloys.

The shear strength of amalgam was not extremely sensitive to the mixing time variation over the range used in this investigation as shown in Figure 13. Since good shear strength was produced by mixing from 30 to 60 seconds, the amalgam was mixed for 60 seconds for each particle size and was sheared at 1/2, 1, 6, and 24 hours. The results are shown in Figure 14.

Amalgam, during the first 1/2 hour, gained from 22 to 49% of its 24 hour strength. In one hour the amalgam specimens had gained 48 to 63% of their 24 hour strength. Size 4 was 20% higher in strength than the average of the two controls.

Figure 15 shows the results of Pd-Ga_E shear strength, after 24 hours, for the various sizes. The highest value for size 4 of Pd-Ga_E was 20% less than that of the highest value of amalgam, which was also size 4. However, the Pd-Ga_E mixture of size 4 with 1-2 micron powder was 21,300 psi as compared to the 21,600 psi of the size 4 amalgam.

3.5 Results of compressive strength tests.

Figure 16 shows the effects of mixing time upon the 24 hour compressive strength of dental amalgam. There was an increase in compressive strength in sizes 5 through 8 as the mixing was increased to 120 seconds. For example, size 8 had

a compressive strength of 6,700 psi for 20 seconds of mixing and 9,300 psi for 120 seconds of mixing. However, when size 8 was mixed for 600 seconds, compressive strength was lowered to 5,000 psi. Size 4 had 44,400 psi for 20 seconds of trituration, 46,200 psi for 60 seconds of mixing, and 44,800 psi for 120 seconds of mixing. Sizes 2 and 3 showed a decrease in strength with increasing the mixing time.

Pd-Ga_E specimens were much more sensitive to mixing time than amalgam specimens when they were tested in 24 hour compressive strength, as is shown in Figure 17. The highest compressive strength for the Pd-Ga_E alloys was not reached until 120 seconds of mixing. There was a continuous gain in strength in all sizes with the increase in mixing time to 120 seconds. A slight gain in strength was produced by further increasing the mixing time. For near miximum compressive strength, Pd-Ga_E required at least twice the mixing time of amalgam. The mixed mass was not completely triturated just because it was wet with the liquid metal.

3.6 Results of tensile strength.

The results for the tensile strength of amalgam after 24 hours are shown in Figure 18. There is a sharp peak in tensile strength at 7,700 psi for size 4. This strength is 24% higher than the average of the two commercial alloys used for comparison. The Pd-Ga_E made from the powders of size 4 mixed with 1-2 micron precipitated powder had a tensile strength of 4,300 psi.

3.7 Results of photomicrographic study.

Photomicrographic examination of the surfaces of "smeared" mixes and the surfaces of condensed specimens of both the amalgam and palladium alloys showed that the spherical particles, at least in the large sizes, retained their original shape during mixing and condensation as seen in Figures 19 and 20. No gross change in the diameters of the particles was observed.

A change from a surface appearance of spherical particles in a liquid medium to one of a solid material was observed, Figures 20 and 21, and in general the rate of this change could be correlated with the rate of hardening as observed in the change of other properties.

In some instances development of elongated structures which often appeared to join one particle to another were observed, Figure 22.

The photomicrographic study was not pursued at length since first, the rough surfaces were not suitable for careful examination and second, there was no assurance that the effects observed on the surfaces were representative of the internal structure of the materials.

4. DISCUSSION

4.1 Physical properties.

Throughout this investigation two factors, particle size and mixing time, were found to have major effects on the physical properties of both silver-tin amalgams and gallium (eutectic)-palladium alloys. The effects of mixing time were less noticeable with the amalgams than with gallium alloys. The major advantage in the use of spherical powders was the simple means they provided to control particle size and shape and surface area.

All properties measured were greatly dependent upon particle size. This dependence was most noticeable in the time required to complete the "hardening" reaction between the solid and liquid phases. The increased surface area associated with the smaller particles results in a more rapid reaction. This is reflected in shorter initial setting times, a reduction in the time required for completion of the dimensional changes, and a more rapid attainment of maximum strength.

In the silver-tin amalgams the most satisfactory mechanical properties were obtained when using spherical powders in size ranges 4 or 5 (15 to 50 microns). In the gallium (eutectic)-palladium alloys both shear strength and tensile strength reached a maximum at this same particle size but the compressive strength continued to increase with decreasing particle size down to the smallest size studied. The most satisfactory mechanical properties were obtained for the gallium alloys when a mixture of particle sizes 4 and 1 was used. In such a mixture the smaller particles probably serve first to increase the surface area and secondly to fill the spaces between the larger particles.

The development of maximum strength in these alloys depends on using a sufficient time for mixing the powdered and liquid metals. The mixing time required for the gallium-palladium alloys is about two minutes longer than the time required for mixing ordinary dental amalgam.

The properties of silver-tin amalgams and gallium (eutectic)-palladium alloys, both prepared using spherical powders, are compared with each other and with the average of two widely-used commercial amalgam alloys in Table 3. The compressive strength and shear strength of amalgam specimens prepared from spherical alloy powders equals or exceeds the corresponding values for the commercial alloys. The "spherical" amalgams possess a considerably higher tensile strength than the commercial amalgams when the optimum size spherical powders are used. The compressive strength of the gallium (eutectic)-palladium alloys (using a mixture of two particle sizes) is much higher than the compressive strength of either "spherical" amalgams or commercial amalgams. The tensile strength of the amalgam alloys, however, is significantly higher than the tensile strength of the gallium alloys. Their shear strengths are approximately equal.

The dimensional change for the gallium alloys during "setting" is quite high (approximately 100 μ/cm) and a separate investigation would be required to determine whether such large changes produce undesirable clinical effects. If so, the dimensional change might be reduced by suitable alloy additions.

The measurements of "setting-time" for amalgams prepared from spherical alloy powders produced a remarkably consistent correlation with corresponding "shear strength" values. The procedure used (measurement of the length of indentations produced by a blade forced against the cylindrical surface of a specimen) should be further investigated to see whether it is suitable for use with commercial alloys where a wide range of particle sizes and shapes are encountered. It would be desirable also to compare the results of such tests with actual observations of "carvability" involving a number of different operators.

4.2 Clinical characteristics.

No attempt was made to evaluate the clinical characteristics of these alloys except for the biological tests on the gallium-tin-palladium alloys which will be reported in

another paper. The biological tests did provide some information on the toxicity, corrosion resistance, and general adaptability of the gallium-tin-palladium alloys. In general the results indicated a very low toxicity and good adaptibility of these alloys as filling materials. The corrosion resistance, however, was generally unsatisfactory and further research is necessary in order to improve the chemical stability of these alloys which come in contact with oral and body fluids. The packing characteristics of the gallium (eutectic)-palladium alloys and of spherical powders in general are somewhat different from those of the usual commercial alloys. They are easily packed by inserting the entire mass into a cavity and condensing it as a unit. During the biological studies a number of gallium fillings were placed in rat molars and no unusual difficulties were encountered in the manipulation of these alloys. Further clinical studies would be required, however, before any extensive use of this alloy could be contemplated. It would also be necessary to evaluate the clinical significance of the higher compressive strength and the lower tensile strength of the gallium-tin-palladium alloy as well as the clinical effects resulting from its improved high-temperature strength, its greater wettability toward the tooth structure, and the improved matching between the thermal expansion coefficients for gallium-tin-palladium alloys and human teeth.

SUMMARY

The use of spherical particles provided an excellent method of studying the effect of particle size on the mechanical properties of dental amalgam and of palladium-gallium-tin alloy. Particle size affected the plasticity of the mixed mass, setting time, dimensional changes during setting and compression, shear and tensile strength. Mixing time also affected these properties but to a lesser extent. Amalgams made from particles approximately 20 to 40 μ in diameter had better properties than those made from either larger or smaller particles. Palladium alloys exhibited better properties when made from smaller particles or from mixtures of 40 μ size with small particles. With the exception of the tensile strength of palladium alloys, compressive shear, and tensile strengths of both amalgam and palladium alloys made from optimum particle sizes were equal or superior to strengths of the commercial alloys used for comparison.

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TABLE 1

SEPARATION OF SPHERICAL PARTICLES

SIZE	METHOD	U.S. SIEVE NUMBERS & FLOW RATES		PARTICLE DIAMETER (MICRONS)	AVERAGE DIAMETER (MICRONS)	% Ag OF TOTAL (Wt %)	% Pd OF TOTAL (Wt %)
		Ag	Pd				
11	SIEVING	—	R-50 mesh	297*	—	—	6.7
10	SIEVING	—	P-50 R-70	210 - 297	253.5	—	10.6
9	SIEVING	—	P-70 R-100	149 - 210	179.5	—	10.8
8	SIEVING	P-100 mesh R-140	P-100 R-140	105 - 149	127.0	5.4	22.9
7	SIEVING	P-140 R-200	P-140 R-200	74 - 105	89.5	5.1	17.0
6	SIEVING & ELUTRIATION	P-200 R-8.68 ml/sec	P-200 R-11.00 ml/sec	50 - 74	62.0	8.0	18.2
5	ELUTRIATION	P-8.68 R-3.88	P-11.00 R-5.08	30 - 50	40.0	18.0	5.1
4	ELUTRIATION	P-3.88 R- 1.55	P-5.08 R-2.00	15 - 30	22.5	26.6	4.3
3	ELUTRIATION	P- 1.55 R-0.82	P-2.00 R- 1.02	8 - 15	11.5	11.9	1.3
2	ELUTRIATION	P-0.82 R-0.52	P- 1.02 R-0.68	4 - 8	6.0	11.2	0.6
1	ELUTRIATION	P-0.52	P-0.68	0* - 4	2.0	9.3	
						LOST 4.5	LOST 2.4

P - PASSED thru the numbered sieve or flowed into the collecting tube.

R - RETAINED on the numbered sieve or remained in the column or flask.

TABLE 2

EFFECT OF TRITURATION TIME ON SETTING EXPANSION

ALLOY	PARTICLE SIZE	20 SECONDS MIX	180 SECONDS MIX
AMALGAM			
3	8 - 15μ	- 13.0μ	- 18.0μ
4	15-30	- 12.0	- 13.5
6	50-74	+ 24.0	+ 41.0
$Pd-Ga_E$			
3	8 - 15μ	+ 290μ	+ 136μ
4	15-30	+ 421	+ 189
6	50-74	+ 509	+ 230

- CONTRACTION

+ EXPANSION

TABLE 3

Comparison of Properties of Amalgam and Palladium-Gallium-Tin Alloys

Properties	Amalgam			Pd-Ga_E	
	Commercial	Spherical Type			
24 Hr. Strength	psi	size	psi	size	psi
Compressive	⊙44,600**	4	46,200	4,1*	61,000
Shear	18,000	4	21,600	4,1*	21,000
Tensile	6,200	4	7,700	4,1*	4,300
5 Day Setting Change	⊙0.83μ**	4	-2.75μ**	4,1**	104μ

* 2/3 of size 4 mixed with 1/3 of 1-2 microns precipitated powder. (by weight)

** Data from Demaree and Taylor (7). ⊙These are averages of three commercial alloys.

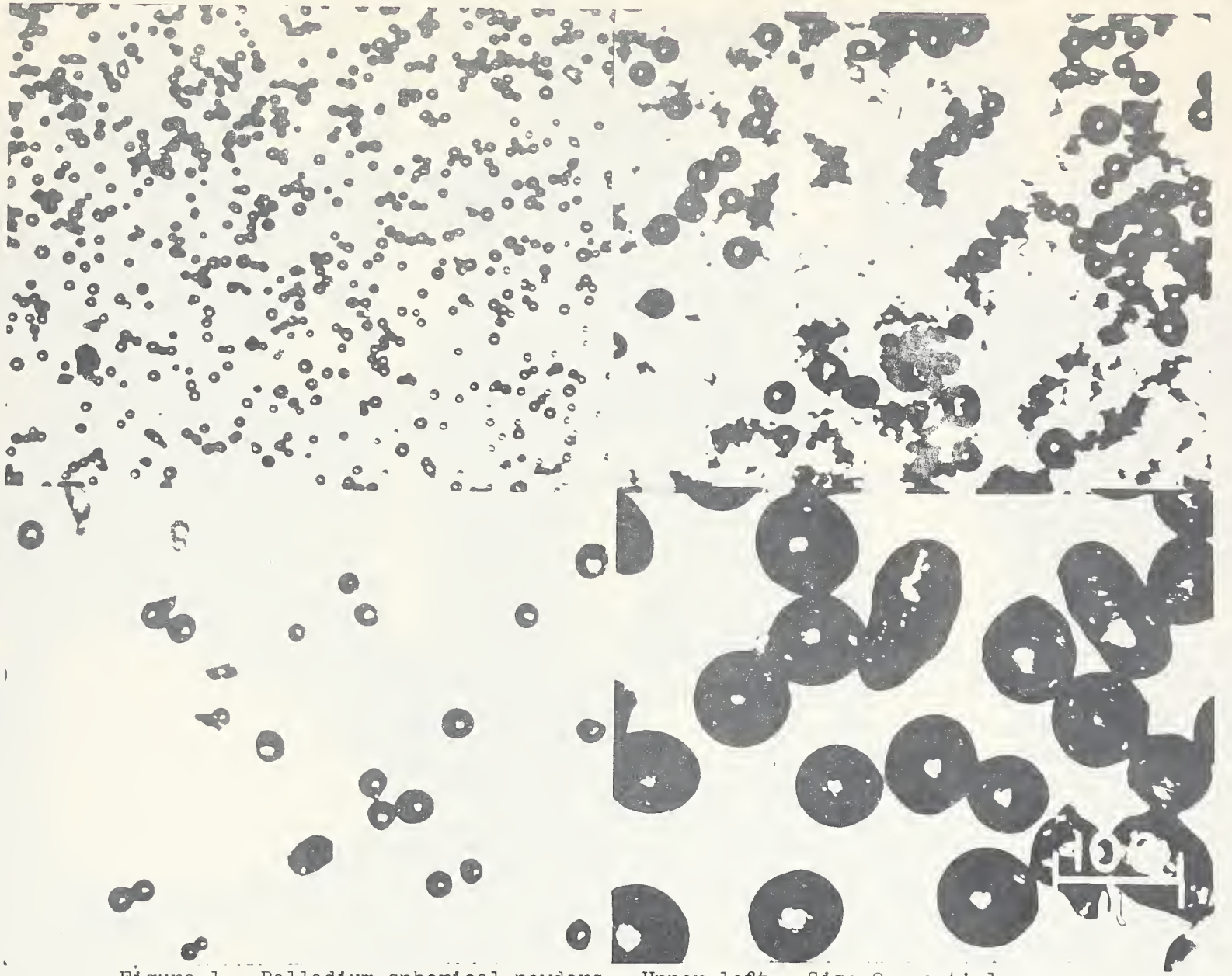

Figure 1. Palladium spherical powders. Upper left: Size 2, particle diameter range of 4 to 8 μ. Upper right: Size 4, a particle diameter range of 15 to 30 μ, mixed with a powder of diameter of 1 to 2 μ. Lower left: Size 4, particle diameter range of 15 to 30 μ. Lower right: Size 6, particle diameter range of 50 to 74 μ.

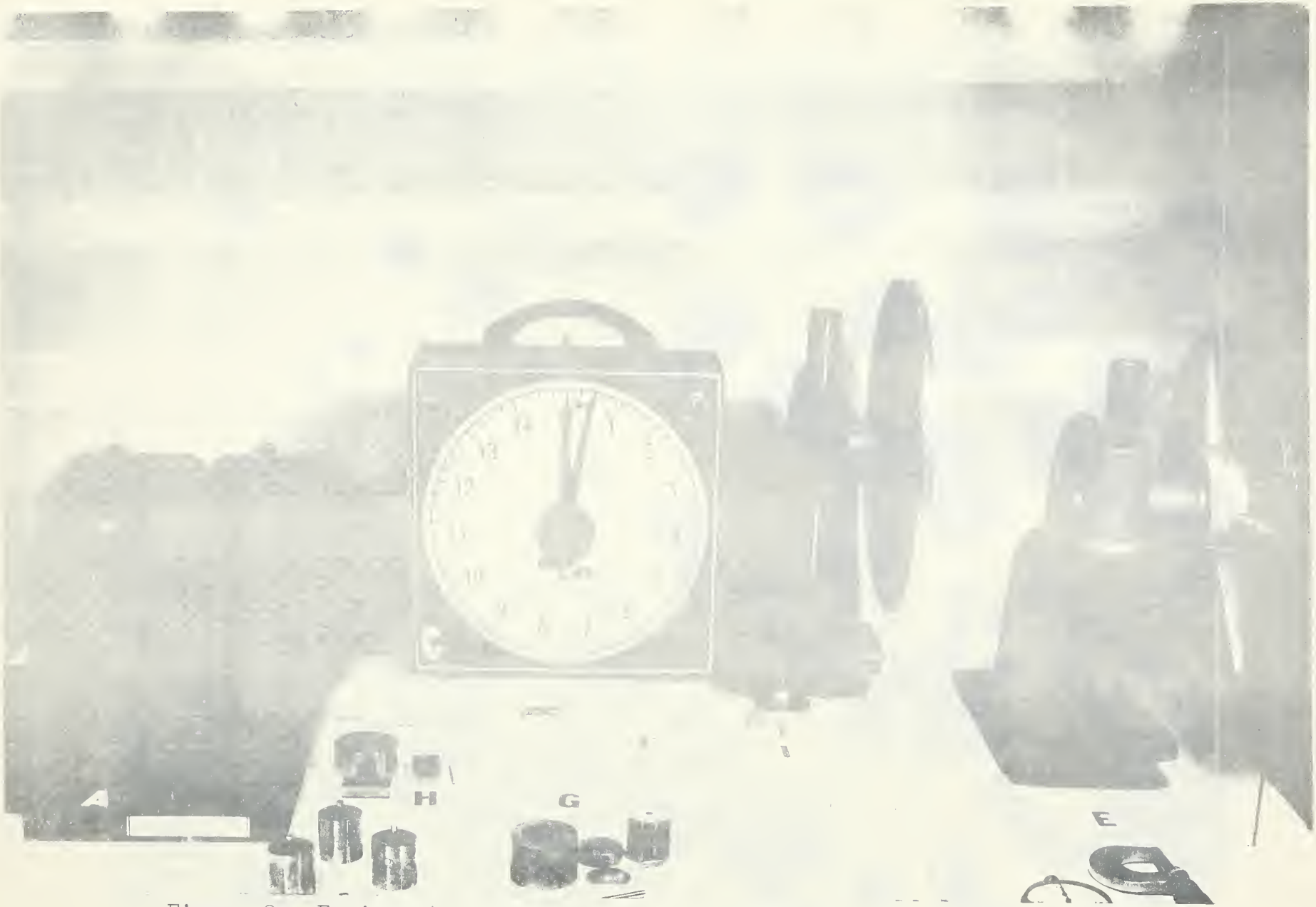

Figure 2. Equipment used for preparing specimens. A. Gelatin capsules and tray; B. Automatic amalgamator; C. Timing device; D. Compressive specimen mold; E. Bench press with assembled mold; F. Microburet; G. Teflon capsule and delivery plunger for mixing palladium-tin-gallium alloy; H. Tensile specimen mold; I. Assembled tensile mold; J. Shear specimen molds.

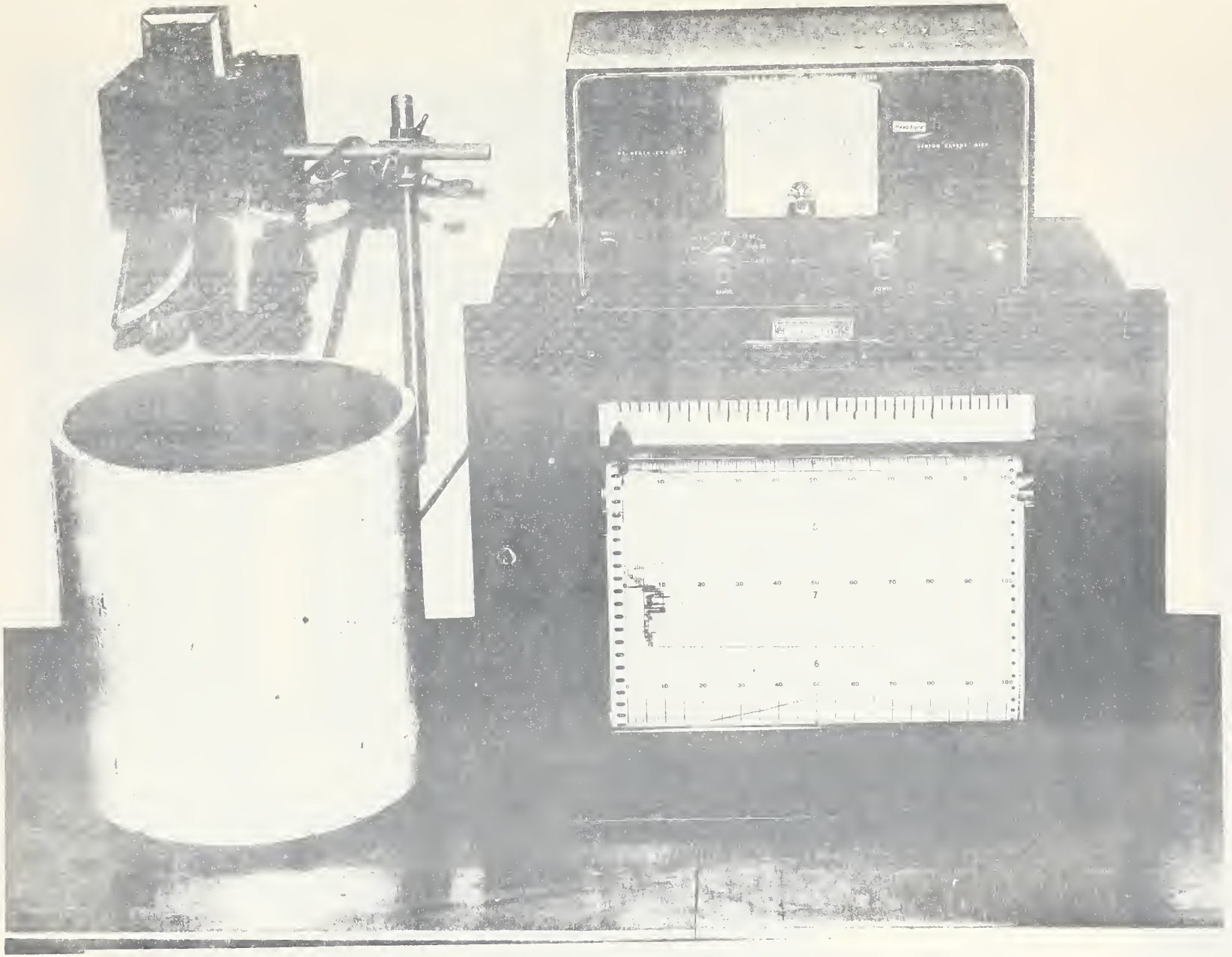

Figure 3. Centrifugal force apparatus. The apparatus used for tensile strength testing consists of a steel enclosure (left), containing the rotor, a photo cell (centered above the enclosure), a mirror (left of the photo-cell), a potentiometer recorder (right) and a frequency meter (upper right).

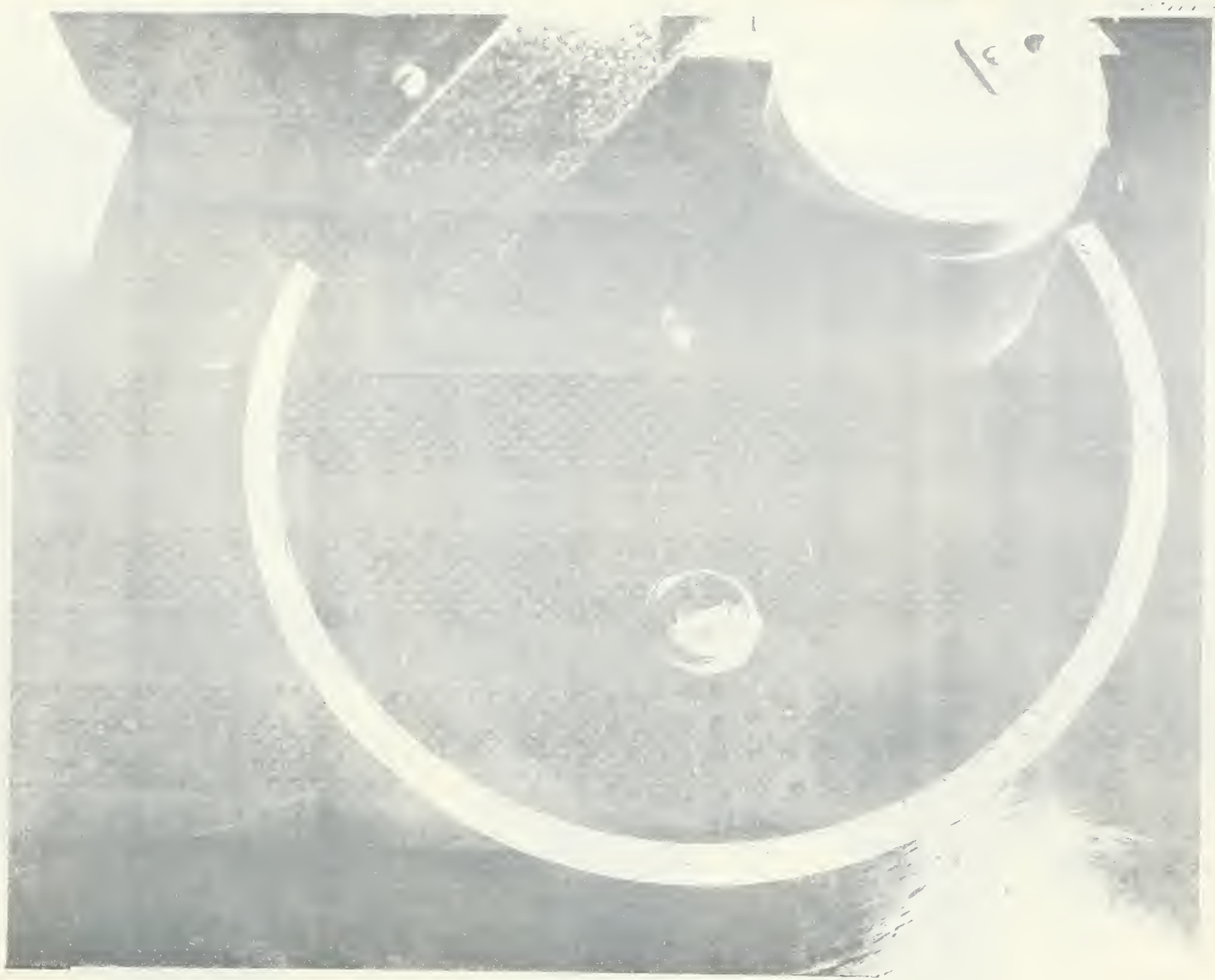

Figure 4. Top view of centrifugal force apparatus. The tensile specimen is placed in the dumb-bell shaped depression in the rotor near the center of the picture.

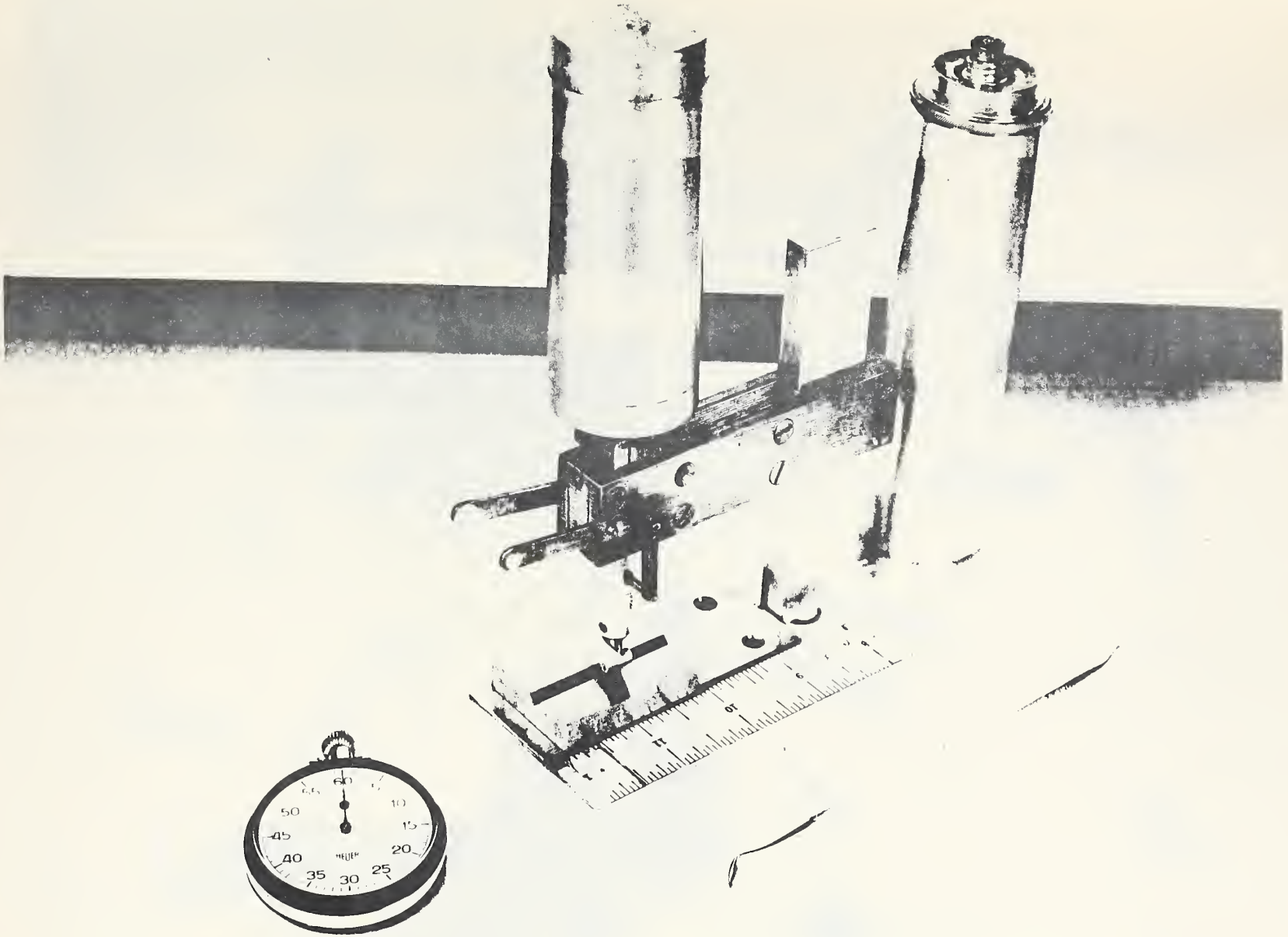

Figure 5. Initial setting time measuring device. The compressive-type cylindrical specimen is held by the V-shaped trough beneath the wedge shaped blade. The load on the blade is 1000 grams, total weight of the blade, holder, and cylinder above it containing lead shot. The load is applied and released by means of the handle (left center).

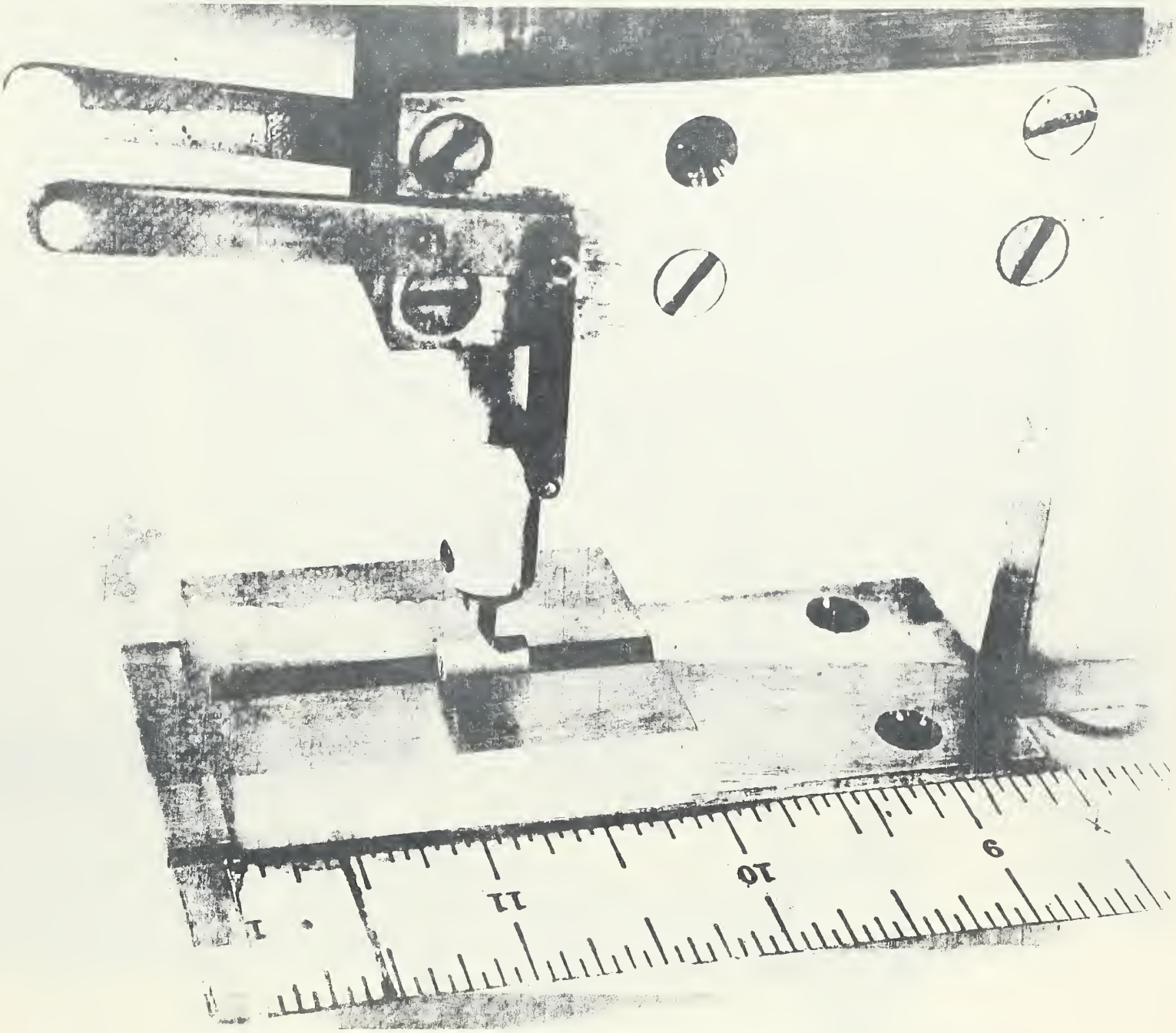

Figure 6. Blade resting on specimen in the initial setting time device.

Figure 7. Shear strength apparatus. A. Housing with a slot in the top to guide the blade; B. Blade; C. Blade positioning pins; D. Specimen, E. Specimen aligning rod; F. Frame; G. Set screws which contact the aligning rods.

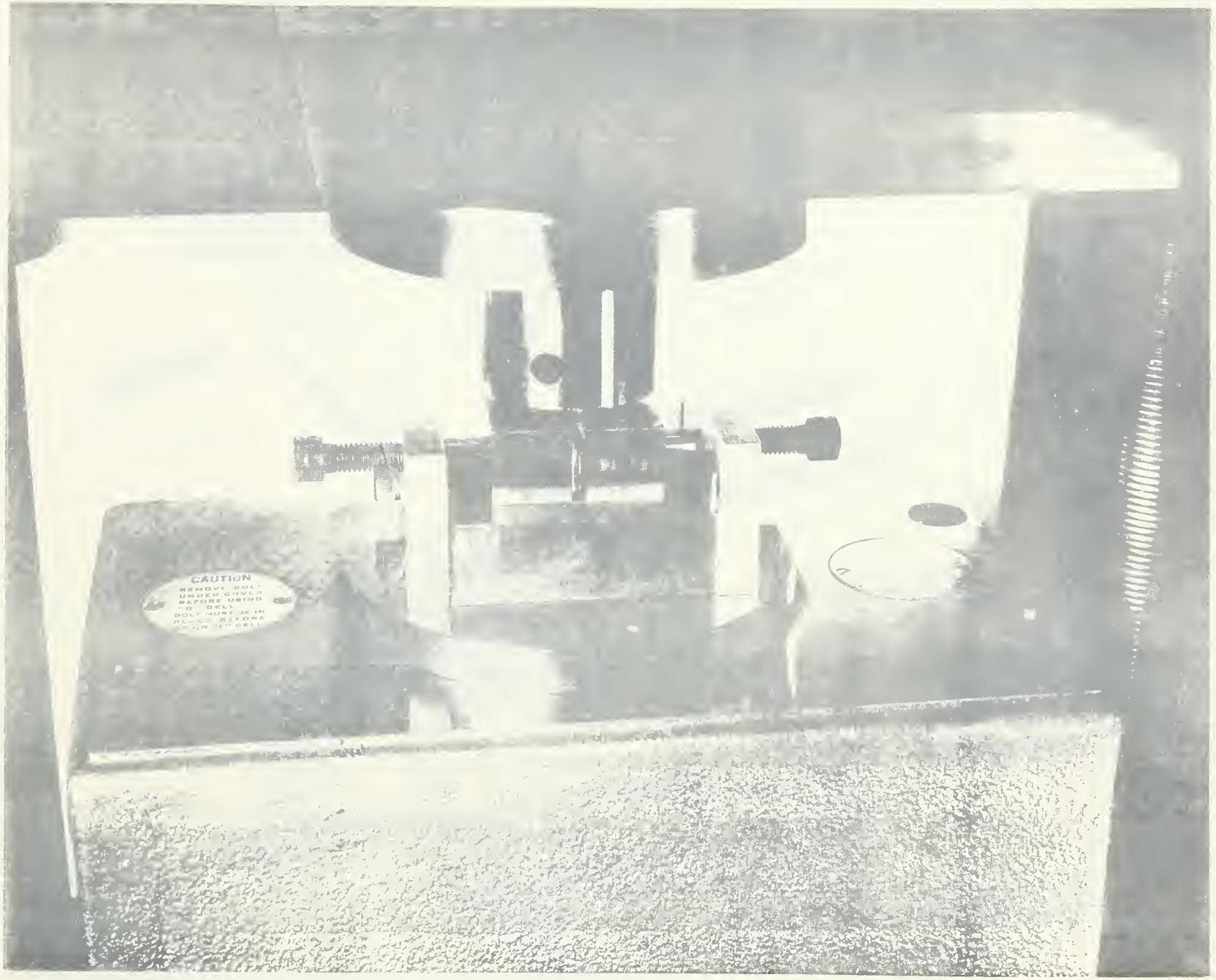

Figure 8. Shearing apparatus assembly in position on Instron testing machine.

Figure 9. Shearing apparatus blade and specimen after testing. The blade contains the middle portion of the specimen in the lower center. The holes above to the right and left are for blade aligning. Another sheared specimen in the foreground shows the relative sizes of the sheared portions.

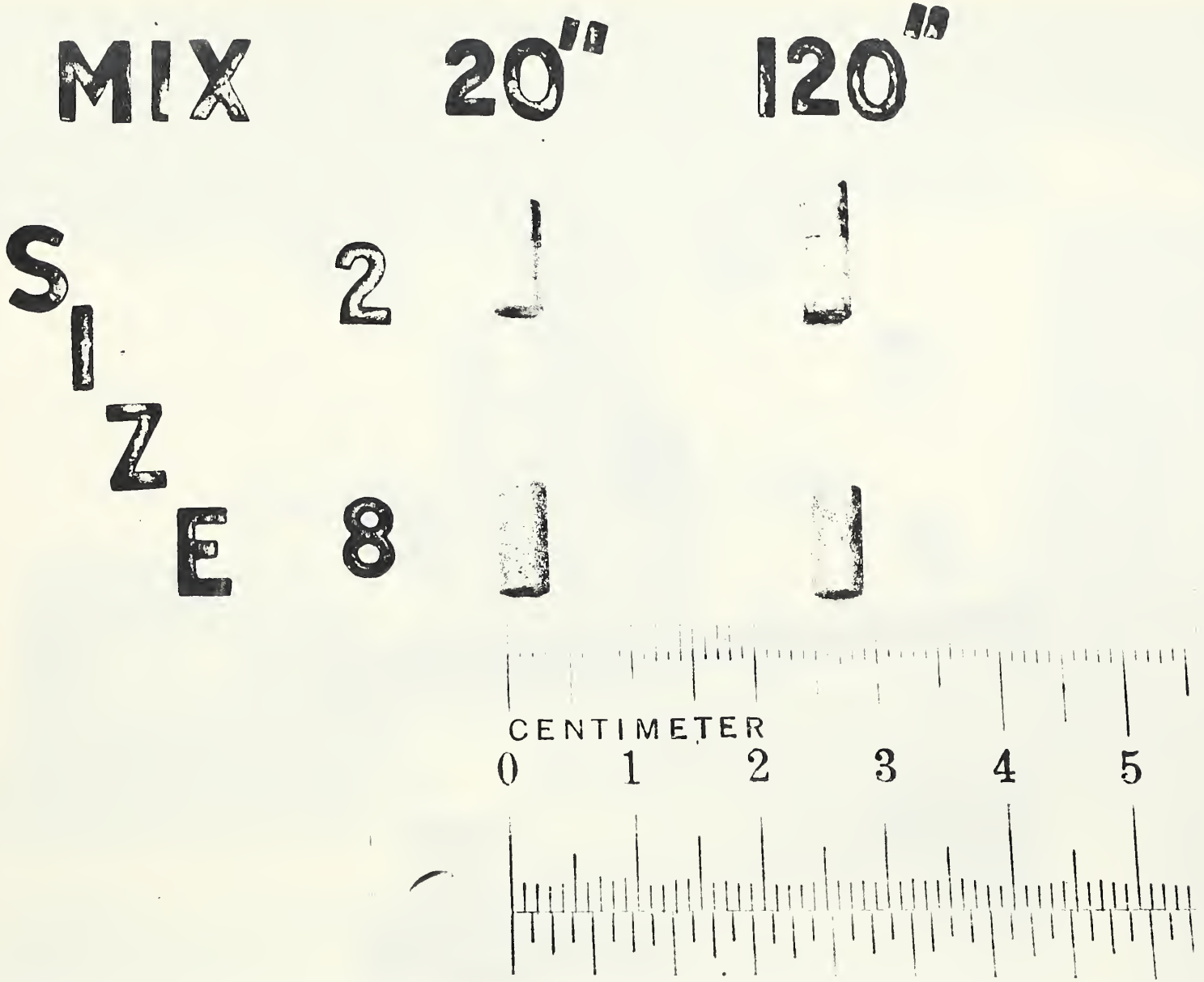

Figure 10. Amalgam compressive strength specimens. The specimen made from size 2 mixed for 20 seconds (upper left) was smooth in surface. The upper right specimen made from size 2 mixed for 120 seconds had surface voids and was longer. The specimen from size 8 (lower left) mixed for 20 seconds had rough pebbly surfaces. When mixed for 120 seconds the resulting specimen (lower right) was smoother and a little shorter in length.

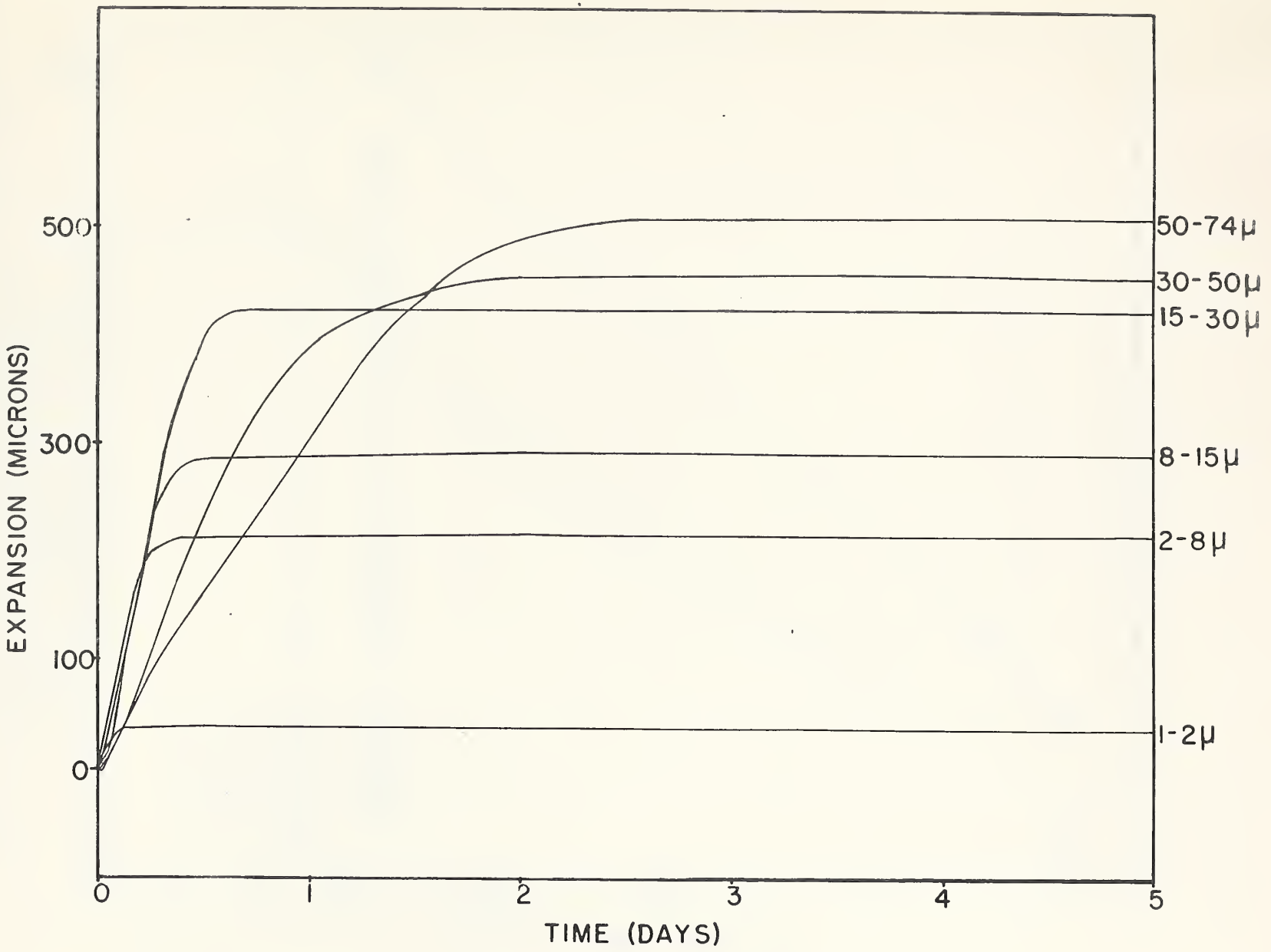

Figure 11. Setting expansion of palladium-gallium-tin alloys. The time of final set, indicated by the horizontal lines, increased with increasing particle size.

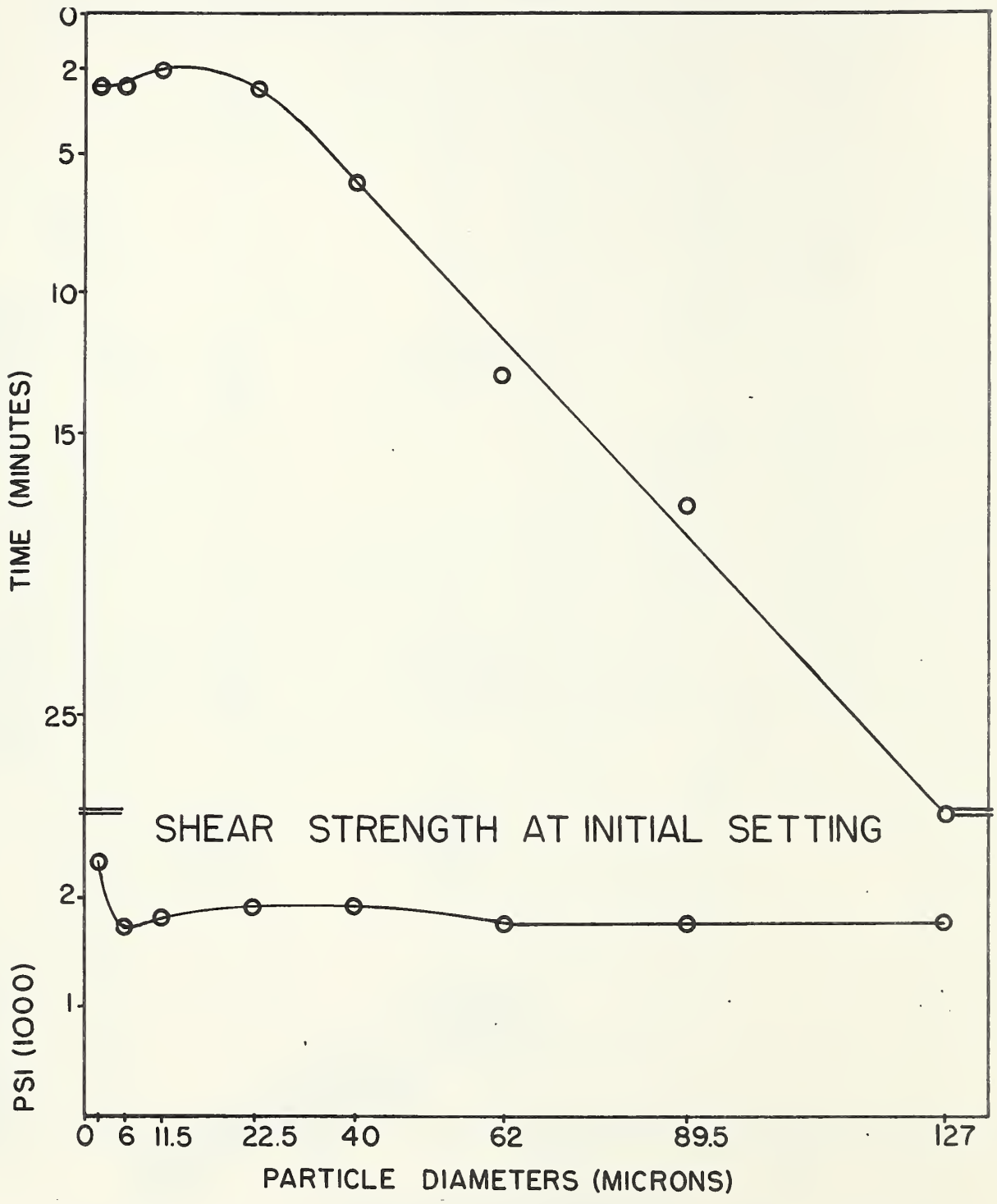

Figure 12. Initial setting time vs particle size (top) and shear at initial setting time (bottom) of amalgam. Each increment on the horizontal axis represents a particle size starting with size 1 to the left to size 8 at the far right.

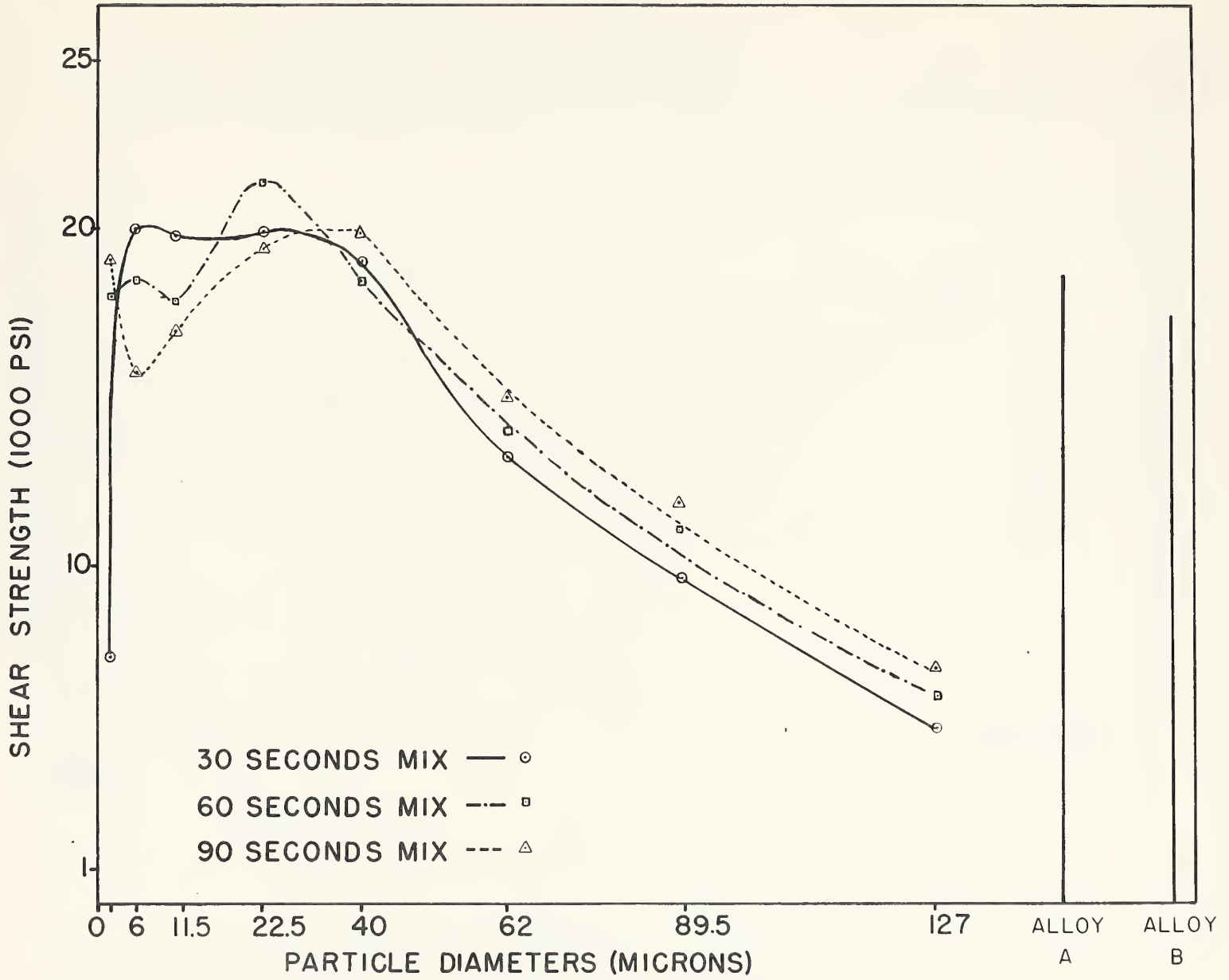

Figure 13. Effect of particle size and mixing time on the shear strength of amalgam. Values for two commercial alloys are shown for reference.

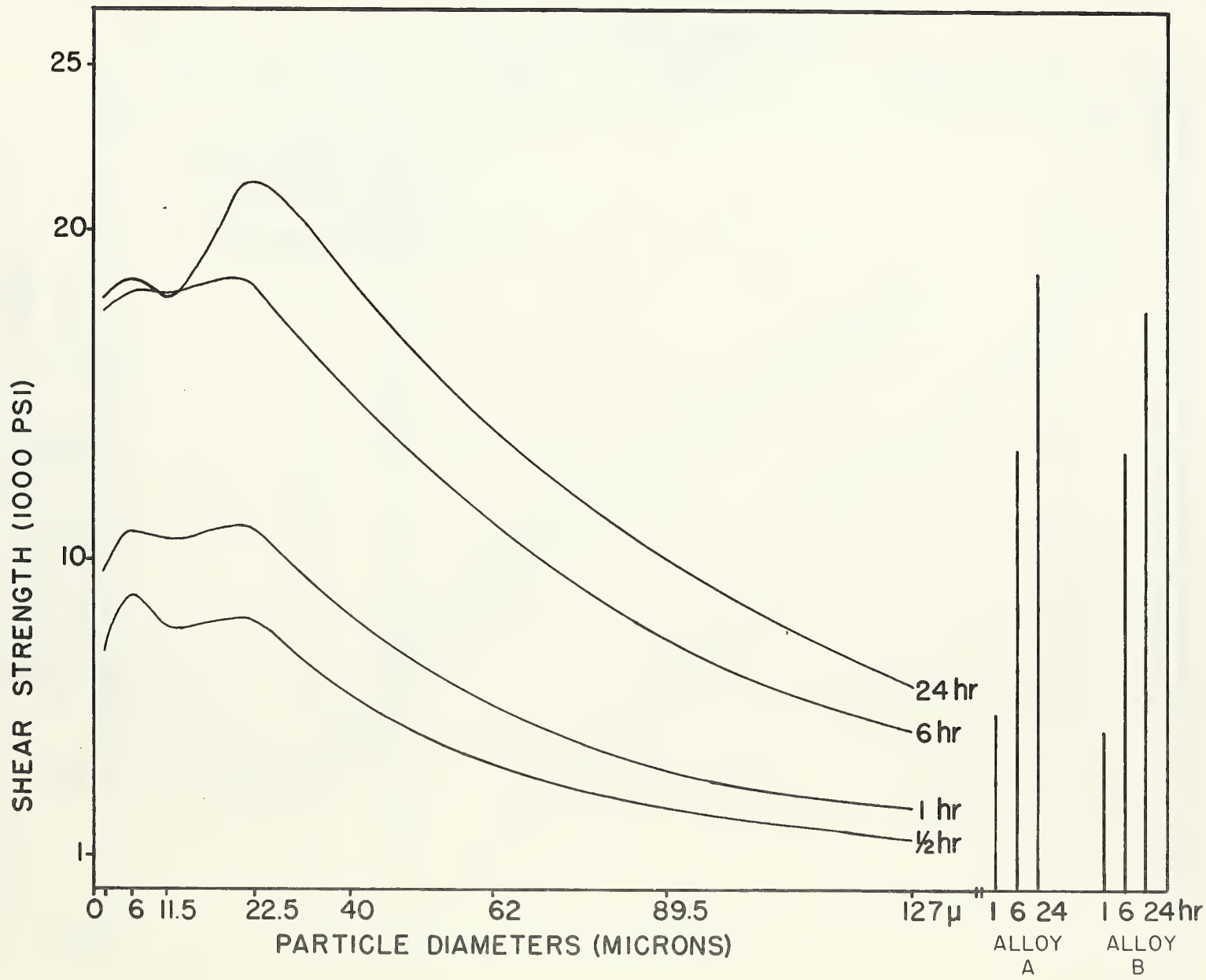

Figure 14. Effect of particle size and specimen age on shear strength of amalgam.

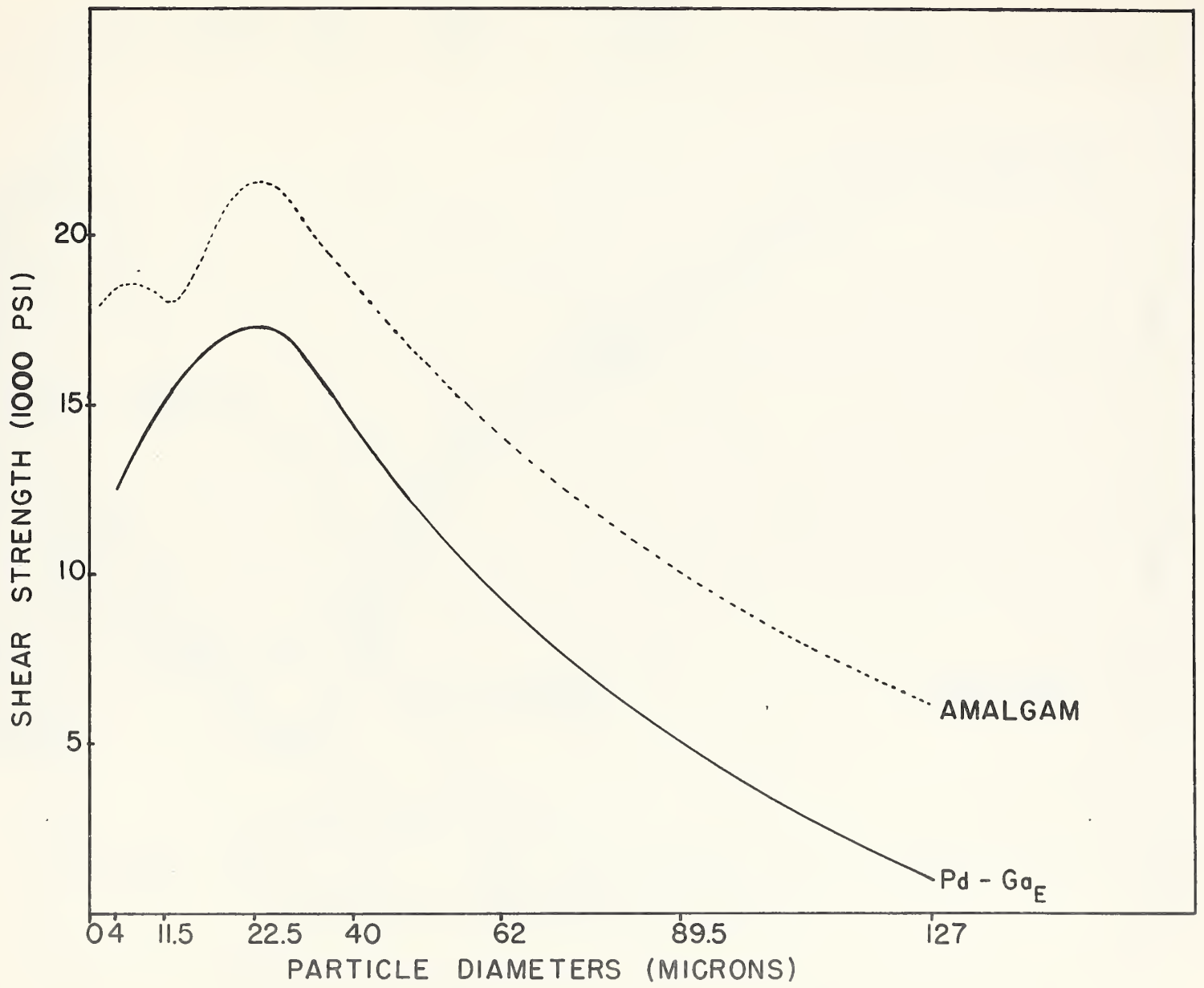

Figure 15. Comparison of shear strength of amalgam and Pd-Ga_E alloys at 24 hours.

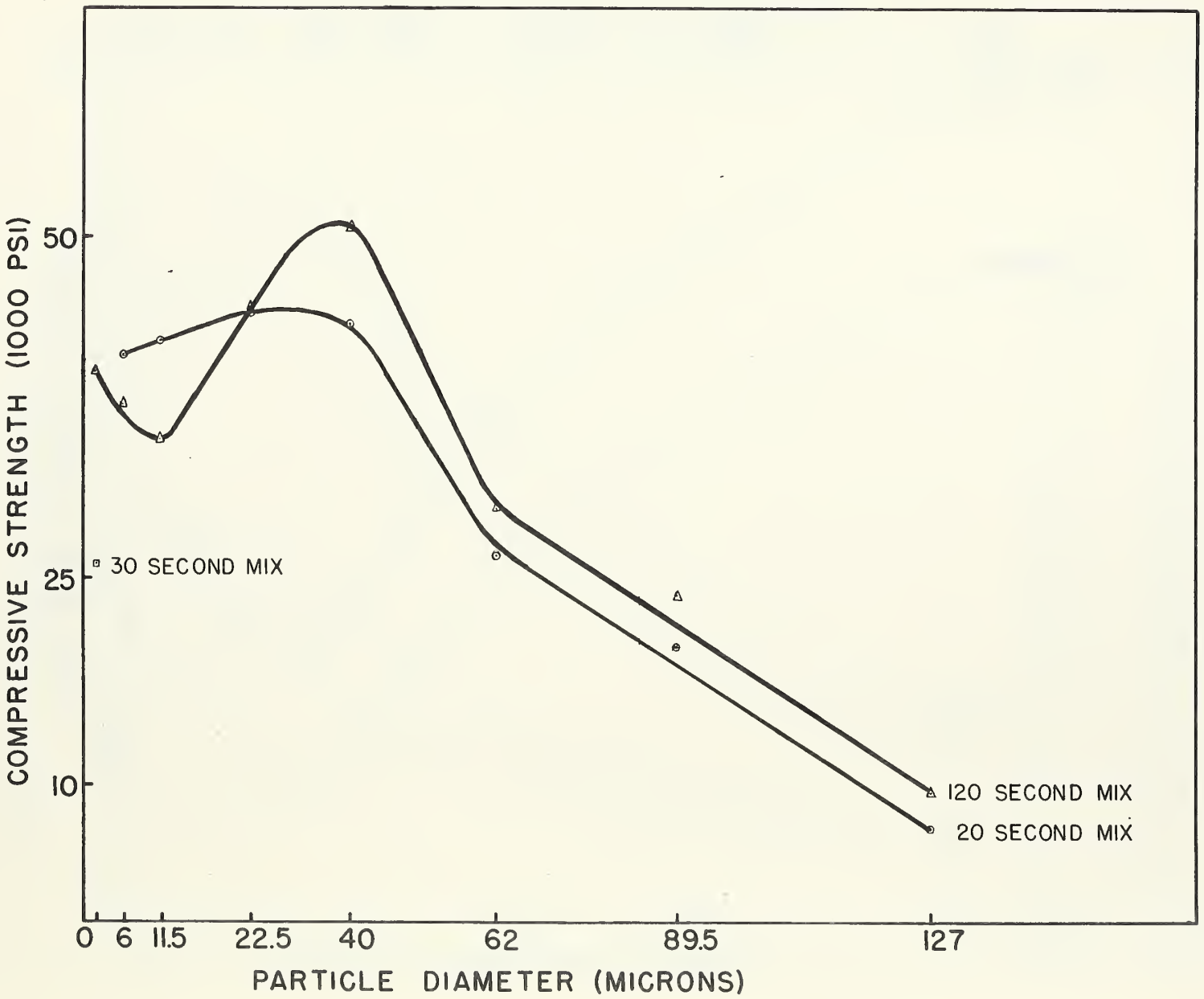

Figure 16. Effect of particle size and mixing time on compressive strength at 24 hours.

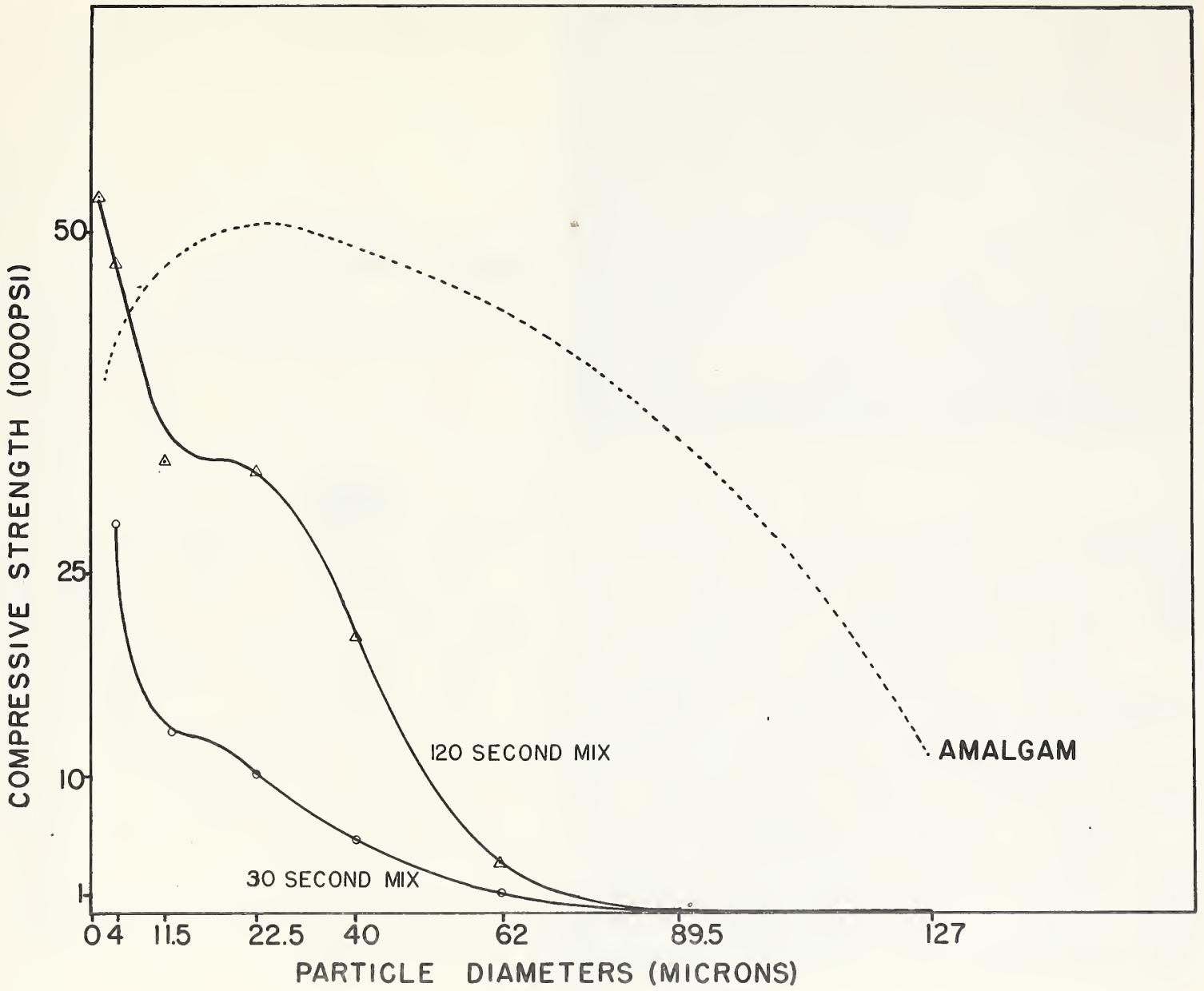

Figure 17. Comparison of compressive strength of amalgam and Pd-Ga_E alloys at 24 hours. Amalgam data are from Demaree and Taylor [7].

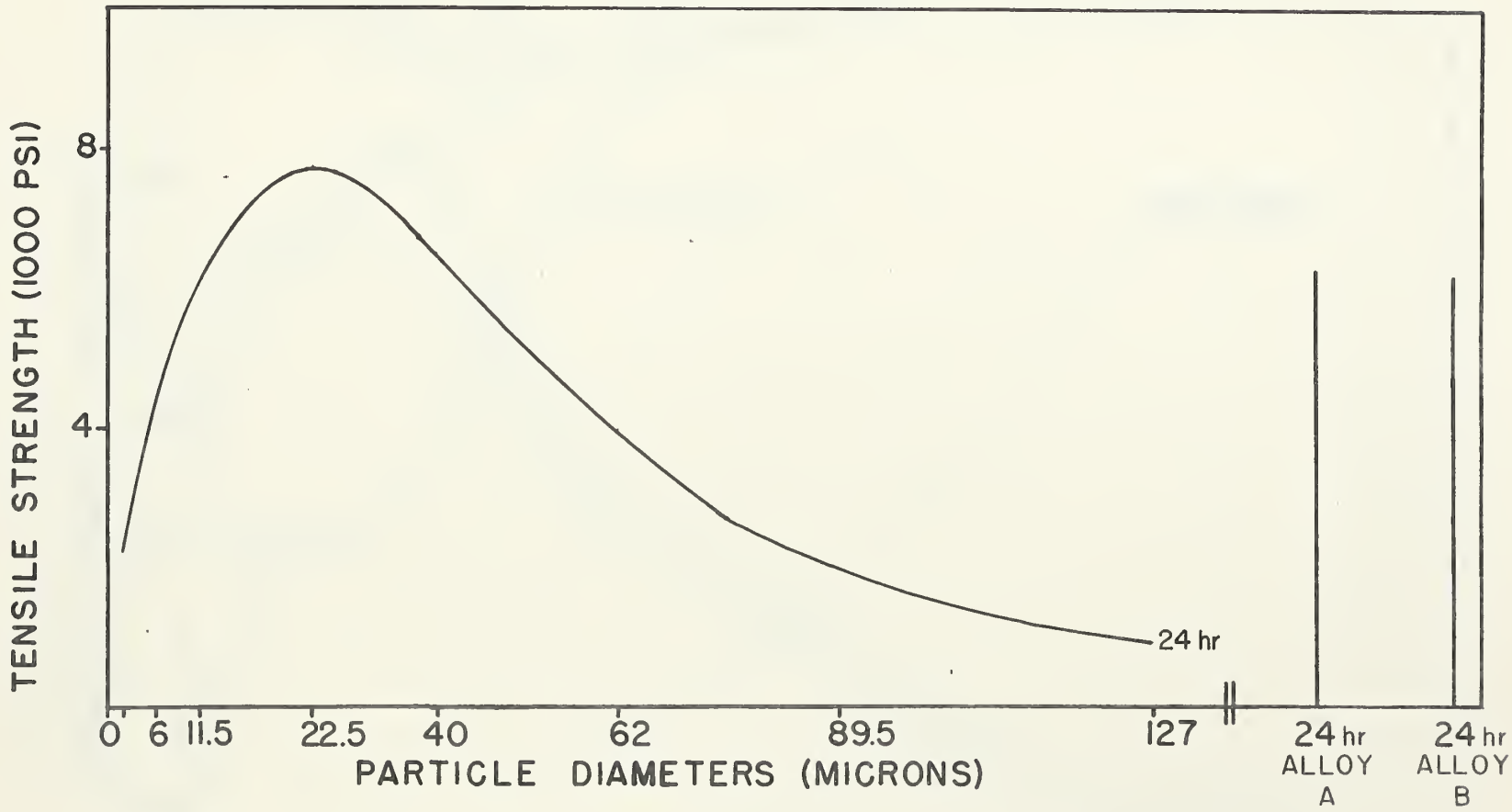

Figure 18. Effect of particle size on tensile strength of amalgam.

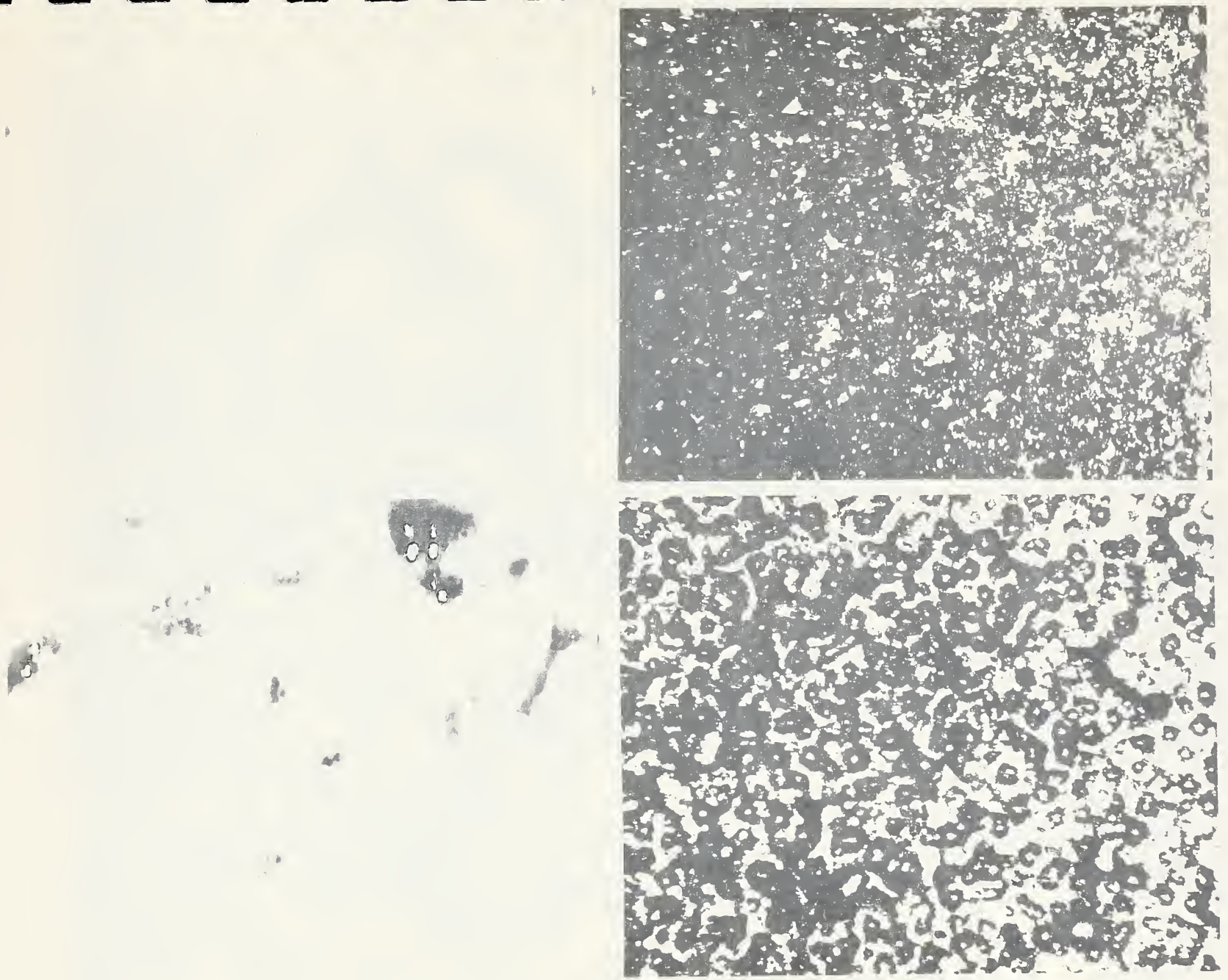

Figure 19. Smear of amalgam made from spherical particles five minutes after the end of trituration. Upper left - Size 8 (2/3 by wt) mixed with size 4 (1/3 by wt). Upper right - Size 2. Lower left - Size 8. Lower right - Size 4.

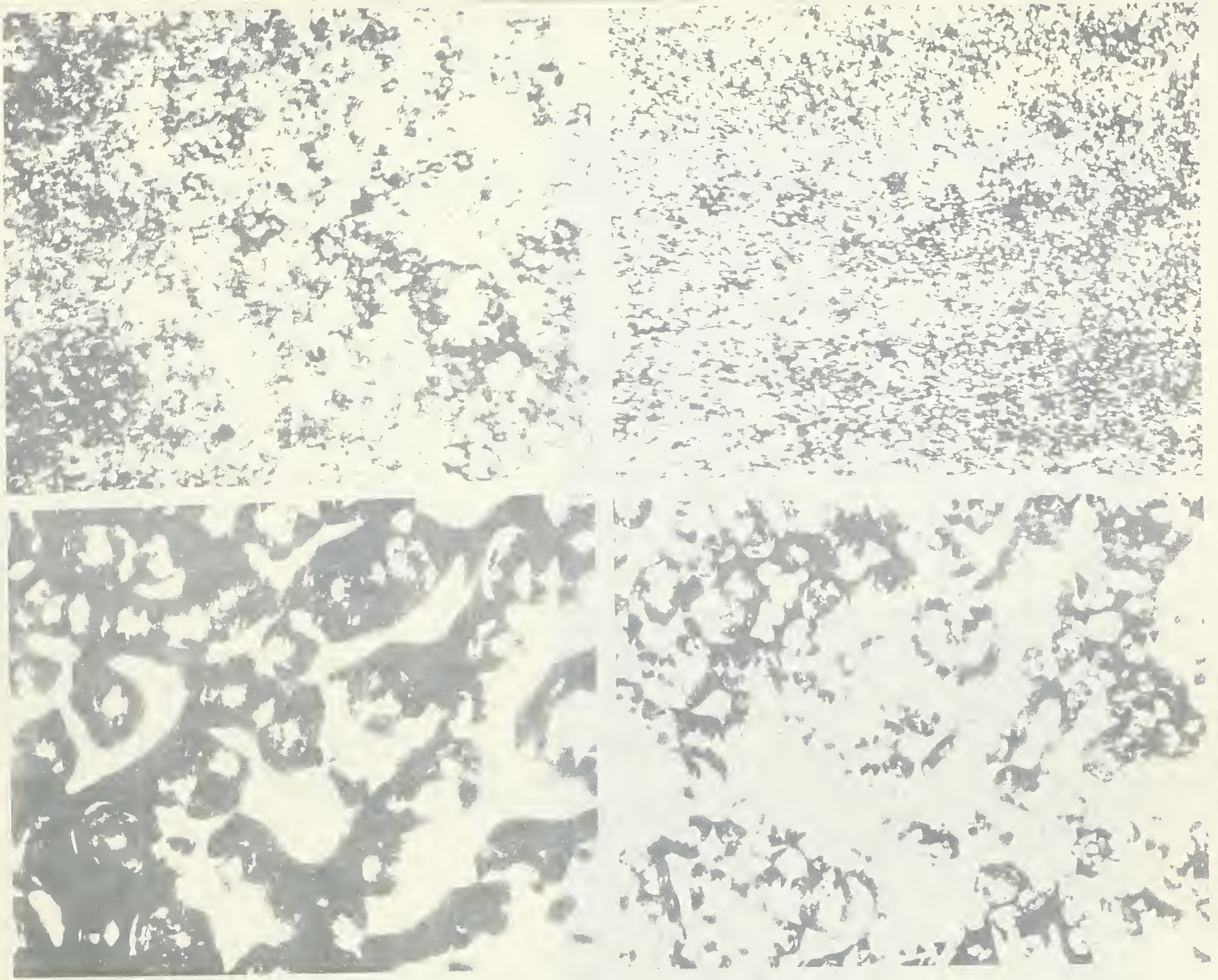

Figure 20. Surface of compressive-type specimen of palladium-gallium-tin alloy made from spherical particles 5 minutes after trituration. Upper left - Size 4 (2/3 by wt) mixed with 1-2 micron powder (1/3 by wt). Upper right - Size 2. Lower left - Size 6. Lower right - Size 4.

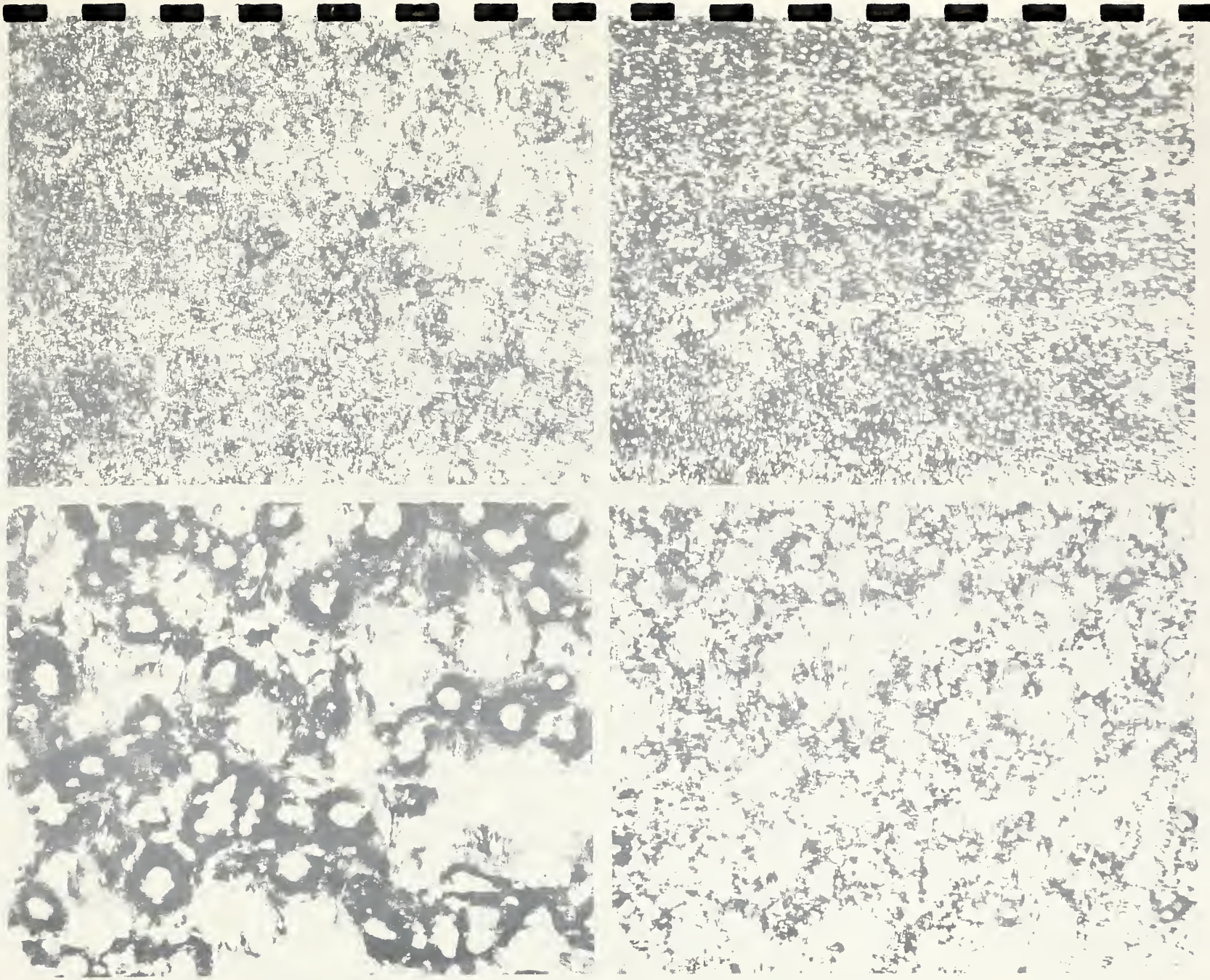

Figure 21. Surface of compressive-type specimen of palladium-gallium-tin alloy made from spherical particles 1 hour after trituration. Upper left - Size 4 (2/3 by wt) mixed with 1-2 micron powder (1/3 by wt). Upper right - Size 2. Lower left - Size 6. Lower right - Size 4.

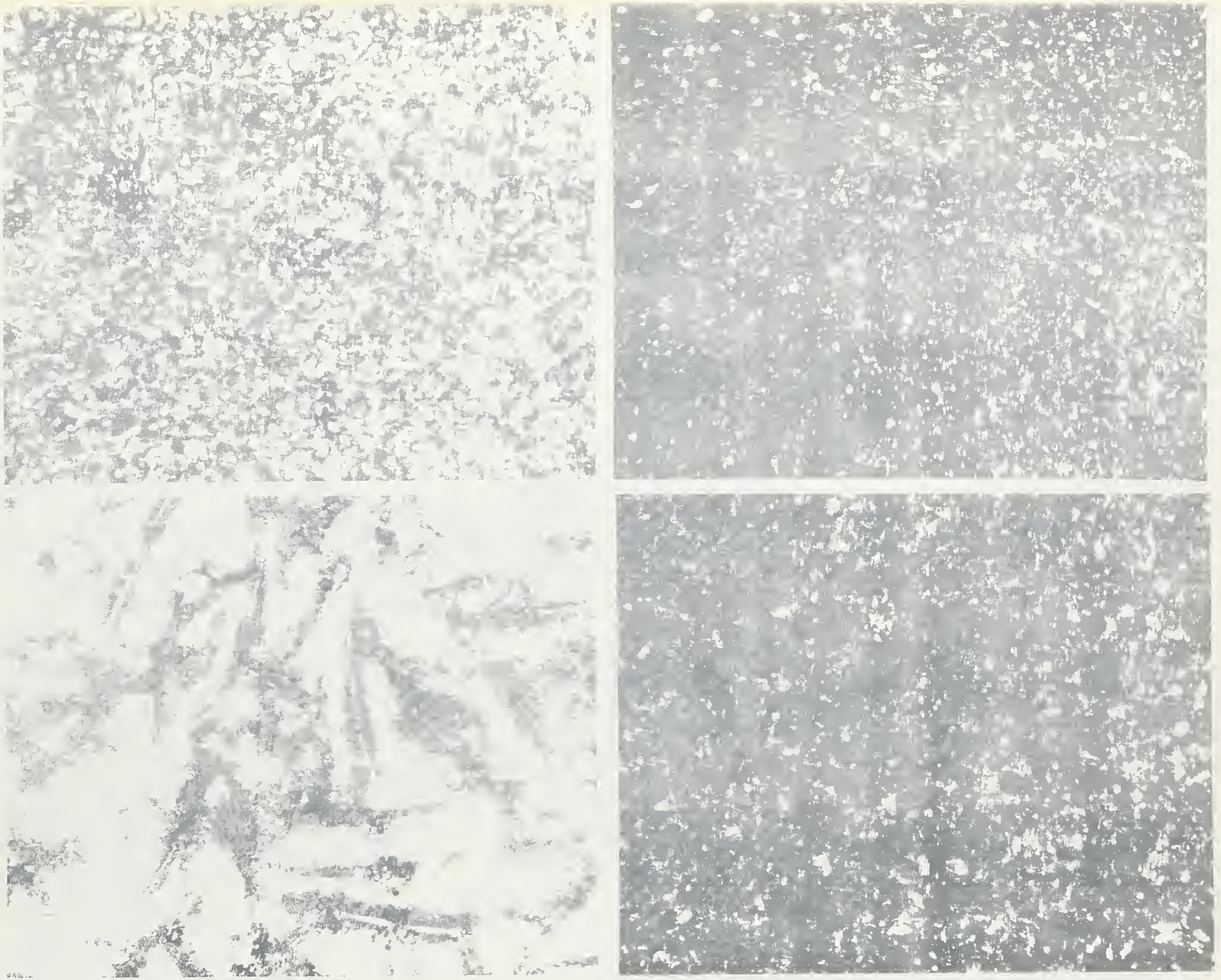

Figure 22. Smear of amalgam made from spherical particles 1 hour after the end of trituration. Upper left - Size 8 (2/3 by wt) mixed with size 4 (1/3 by wt). Upper right - Size 2. Lower left - Size 8. Lower right - Size 4.