# NATIONAL BUREAU OF STANDARDS REPORT

8507

Progress Report

on

Early Strength, Flow and Dimensional Changes Obtained on Amalgam Prepared With a Standardized Mechanical Technic

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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# Early Strength, Flow and Dimensional Changes Obtained on Amalgam Prepared With a Standardized Mechanical Technic

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## Abstract

The rate of hardening of five amalgams was nearly linear with time during the first hours, as shown by the increase in compressive strength or the decrease in flow. There were large differences in strength and in flow among the alloys. The data indicate that an early flow test should be given consideration as a method of specifying the setting time of amalgam. The magnitude of initial shrinkage during hardening appears to be directly related to strength and flow. The time of maximum shrinkage occurs at about the same time for all five amalgams but there are large differences in the dimensional changes which occur during the hardening of the five amalgams. The relation of the dilatometric and flow data is linear up to the time of maximum shrinkage. Specimens were prepared by an all-mechanical method so that observations could begin within three minutes from the end of trituration.


## 1. Introduction

The rate of hardening of an amalgam determines the time during which it is sufficiently plastic for packing, the time during which it can be carved, and the early strength, flow and dimensional change. In the literature there are no data on physical properties determined on specimens from 3 to 19 minutes old [1-8] except for some setting changes by Souder and Peters [9] and a preliminary report on flow by Kimmel [10]. Most of the available information on the physical properties of amalgam was obtained on specimens prepared manually. Ware and Docking [11] developed an all-mechanical procedure for trituration and for condensing the amalgam. Their data were more reproducible when obtained on mechanically prepared than on manually prepared specimens.

In this investigation specimens 3 to 60 minutes old, prepared by an all-mechanical procedure, were used to obtain values for compressive strength and flow. Also, an initial observation on setting changes was obtained 5 minutes after the end of trituration.

## 2. Procedures

The same alloys as those in a recent paper on compressive strength [12] were used in the present study (Table 1).

The flow and strength specimens were cylinders 4 ± 0.01 mm in diameter and 8 ± 0.4 mm long. The setting change specimens (about 9 mm long) were made in the same mold except the plunger had a 90° inverted cone depression on one end.

The time for preparing specimens by the all-mechanical procedure [12] was shortened from 4 minutes to 1-1/2 minutes by decreasing the time of load application during packing of the specimen. This had no effect on the strength.

The standardized procedure in making all specimens follows:

1. To make each specimen, 0.60 ± 0.01 gm of alloy and 0.90 ± 0.03 gm of mercury were triturated with a low-energy mechanical amalgamator* controlled by an electric stop clock to within 1/4 second.

2. The time of trituration (without pestle) was the minimum time that would develop the maximum or near maximum 24-hour strength [12]. Other trituration times were used in developing some of the data, as will be noted subsequently.

3. After trituration the entire mass of mixed amalgam was placed in the mold [12] and the following schedule was observed:

* S. S. White Amalgamator #1

| | | Min-Sec |
|---|---|---|
| (a) | End of trituration | 0 - 00 |
| (b) | Place mix in mold, apply 150 kg/cm$^2$ (2,100 psi) at | 0 - 30 |
| (c) | Remove pressure at | 1 - 30 |
| (d) | Brush away mercury and eject specimen at | 1 - 45 |

4. All specimens were made, stored and tested at 23°C. (73°F).

The age of the specimen, when compressive strength was determined, and the cross-head speed are shown in Figure 1.

A series of flow determinations were made by initially applying a 50-kg/cm$^2$ stress on specimens that were 3, 6, 9, 12 and 15 minutes old as calculated from the end of trituration. Values for one amalgam were also obtained when the initial stress was placed on specimens 30 and 60 minutes old. To determine flow, a fiducial reading was taken at the time of load application and other readings were taken at 2, 4, 5, 8, 10, 15, 20, 30, 45 and 60 minutes, thereafter. From these readings the flow or percentage of shortening in length was calculated. The effects of the mercury-alloy ratio and the length of trituration on the early flow of one amalgam were determined.

Dimensional changes during hardening were measured in an interferometer. The schedule for making observations follows.

| | Time minutes |
|---|---|
| End of trituration | 0 |
| Every 5 minutes | 5 - 30 |
| Every 10 minutes | 30 - 60 |
| Every 15 minutes | 60 - 90 |
| Every 30 minutes | 90 - 180 |
| Final observation | 24 hours |

In the literature, most dimensional change data are based on the 15-minute fiducial reading. In this investigation, either a 5- or 15-minute observation was used as the fiducial or zero reading for dimensional change.

The residual mercury content of the specimens was estimated by the following procedure.

The weights of alloy, expressed mercury and specimen were determined on an analytical balance. The residual mercury in the specimen was then calculated by the equation:

$$\%Hg = \frac{A-(B-C)}{A} \times 100$$

where A = weight of specimen

B = weight of alloy used in making the specimen

C = weight of tin and silver in expressed mercury which is obtained by multiplying the weight of expressed mercury by 0.012 as the impurity in mercury expressed from amalgam is about 1.1 percent tin and 0.1 percent silver [13].

## 3. Results and Discussion

Graphs of the compressive strengths (average of five specimens) in relation to age of the specimens (3 to 60 minutes) in Figure 1 are nearly linear and show the rates of hardening of the five amalgams. Amalgams No. 1 and 2 show the same rate of hardening but are slightly different in strengths. Amalgams No. 3, 4 and 5 show a slower rate of hardening and lower compressive strengths than amalgams No. 1 and 2.

The absence of abrupt changes in slope in these curves indicates that the reaction is continuous at a nearly constant speed during the first hour.

The only variable in the preparation of the specimens of the different alloys was the time of trituration (Fig. 1). These are the minimum times at which maximum or near maximum strength was obtained with 24-hour specimens.

The effect of age of specimens on the flow of amalgam is shown in Figure 2. The load was applied at various times between 3 and 60 minutes from the end of trituration. For example, by applying the load when the specimen was 15 minutes old instead of 3 minutes old, the flow was reduced 50 percent (4% to 2%) on specimens stressed for 10 minutes. The data for each point is the average of two or three determinations.

Figure 3 shows the flow of amalgam specimens after 10 minutes of load application for various ages. These flow data show that the rate of hardening is very similar to that shown by the compressive strength data (Fig. 1). The numerical identification of the curves in Figure 3 is the same as that in Figure 1. As strength is increasing linearly with the time the flow is decreasing at a nearly linear rate (Fig. 3). The weakest alloy has the largest flow. These data indicate that the flow measurements are relatively more sensitive than the strength determination for evaluation of the hardening rate of the alloys tested at early age of specimens.

The determination of flow of amalgam is relatively simple in comparison with the determination of its ultimate compressive strength because a dead load is used for stressing the specimen. The dead load eliminates the difficulties of obtaining constant stress or strain rates required in the determination of compressive strength. The stress or strain rates govern the values obtained in tests for ultimate compressive strength of amalgam and are not easy to control on the variety of available testing machines [12,14]. Therefore, different laboratories should be able to agree better with each other's values for flow than for compressive strength.

The estimated values for mercury content of the five amalgams given in parentheses in Figure 3 and in Table 2 had ranges of 0.1 to 0.9 percent which indicate good reproducibility for the method. These values for mercury content are in accord with values which are expected to be found in well-condensed amalgam.

Increasing the trituration decreases the flow as shown in Figure 4. The minimum trituration time that will give a maximum 24-hour strength in alloy No. 2 is 20 seconds [12]. Increasing the trituration time to 80 seconds did not materially increase the 24-hour compressive strength of alloy No. 2 [12], but Figure 4 shows that the early rate of hardening is accelerated as manifested by its decrease in flow.

On 6-minute-old specimens, loaded for 10 minutes, an increase in the mercury-alloy ratio to as much as three had no effect on flow (Fig. 5). The average value was 2.5 percent ($\sigma = 0.1$) which is within the precision of the data. The flow at a ratio of four was slightly higher.

The effect of trituration on the 24-hour dimensional change during hardening (Fig.6) shows the expected result of decreasing dimensional change with increased trituration. The unexpected result is that all of the alloys have a zero setting change when triturated for 40 ± 10 seconds. In other words , when amalgam is triturated for less than the 30 seconds, under the experimental conditions of this paper, expansion occurs and when triturated for more than 50 seconds shrinkage takes place.

The standardized all-mechanical technic prescribed for the five amalgams uses a fixed weight of mercury and alloy, and the same mechanical mixing and packing procedure so there should be about the same amount of work done in preparing each specimen. These data on dimensional change show that it is feasible to prescribe a standardized technic including a fixed trituration time for the five amalgams even though their rate of hardening is different as the data on flow and compressive strength indicate.

Since the five amalgams became stiffer and stronger with time, but at different rates, it was thought that these effects could be detected in the data on dimensional changes with time. Seemingly, the time of occurrence of the maximum early shrinkage during hardening should occur earlier with the amalgam (No. 2) having the most rapid rate of hardening as shown by the data on early flow and strength. Any such assumption is hazardous because all of the physical and chemical changes occurring in the early hardening of amalgam are not well known and have not been definitely related to the shape of the curves of dimensional change during hardening.

To see if the foregoing correlation with respect to dimensional change existed, each of the five amalgams was triturated for 30 or 40 seconds. The results are in Figure 7.

A comparison of the five amalgams (Fig. 7) shows that a wide variation in the amount of shrinkage occurs and that the setting change curve reaches a minimum at 45 ± 10 minutes. Amalgam No. 5 with the greatest shrinkage has the largest flow while amalgam No. 1 with the least shrinkage has the smallest flow. Thus it would appear that the curves in Figure 7 reflect the rate of hardening.

Doubling the trituration time from 30 to 60 seconds with amalgam No. 3 (Fig. 8) and with amalgam No. 1 had very little effect on the time at which the maximum shrinkage occurred but nearly doubled the amount of shrinkage. On amalgams No. 2, 4 and 5 there was little effect on either the time at which the maximum shrinkage occurred or on its amount. When the trituration time was doubled, the curve for amalgam No. 1 showed a continuing shrinkage for as long as 24 hours when the readings were discontinued. The other curves were shaped similar to those in Figure 8.

Reference to Figures 7 and 8 shows that the time of maximum setting cannot be selected accurately. In Figure 7 the change from 30 to 90 minutes through the maximum shrinkage averages about 1 micron per centimeter with the largest change being 1.7 microns. These results indicate that there is a difference in the setting or reaction rate of the five alloys as shown by the dilation curves (Fig. 7 and 8).

Since both the data on early dimensional change during hardening and on early flow should delineate the rate of matrix formation, there should be a linear relation between the two sets of data. Plots of the relationship in Figure 9 show this to be true up to the time of maximum shrinkage.

## 4. Summary

An all-mechanical, standardized procedure for preparing test specimens was found suitable for five widely used amalgams employing 0.60 grams of alloy, 0.90 grams of mercury and 40-second trituration in a nylon capsule without a pestle in a low-energy mechanical amalgamator.

These amalgams became stiffer and stronger with time but at different rates according to data on flow and compressive strength obtained on specimens with ages as low as 3 minutes. The mercury-alloy ratio up to 3 did not appreciably affect flow. Data on dimensional change during hardening also reflected the different rates of hardening.

The early flow test is a convenient method of specifying the setting time of dental amalgam.

## 5. References

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Table 1

Commercial Dental Alloys Tested

| Brand | Batch Number | Manufacturer |
|---|---|---|
| Aristaloy | 540548 | Baker Dental Division Engelhard Industries, Inc. |
| New True Dentalloy | 17860358 | S. S. White Dental Manufacturing Co. |
| Silver Crown Medium | 511 | General Refineries, Inc. |
| Twentieth Century Fine Cut | 13G61F | L. D. Caulk Co. |
| Twentieth Century Micro Non Zinc | 30H61 | L. C. Caulk Co. |

Table 2

Estimated Mercury Content

| Amalgam | Percent Mercury: Individual determinations | Percent Mercury: Average |
|---|---|---|
| 1 | 48.9; 48.9; 48.6 | 49 |
| 2 | 47.8; 47.8; 48.2 | 48 |
| 3 | 48.9; 48.9; 49.0 | 49 |
| 4 | 50.0; 50.4; 50.4 | 50 |
| 5 | 45.7 46.6 45.7 | 46 |

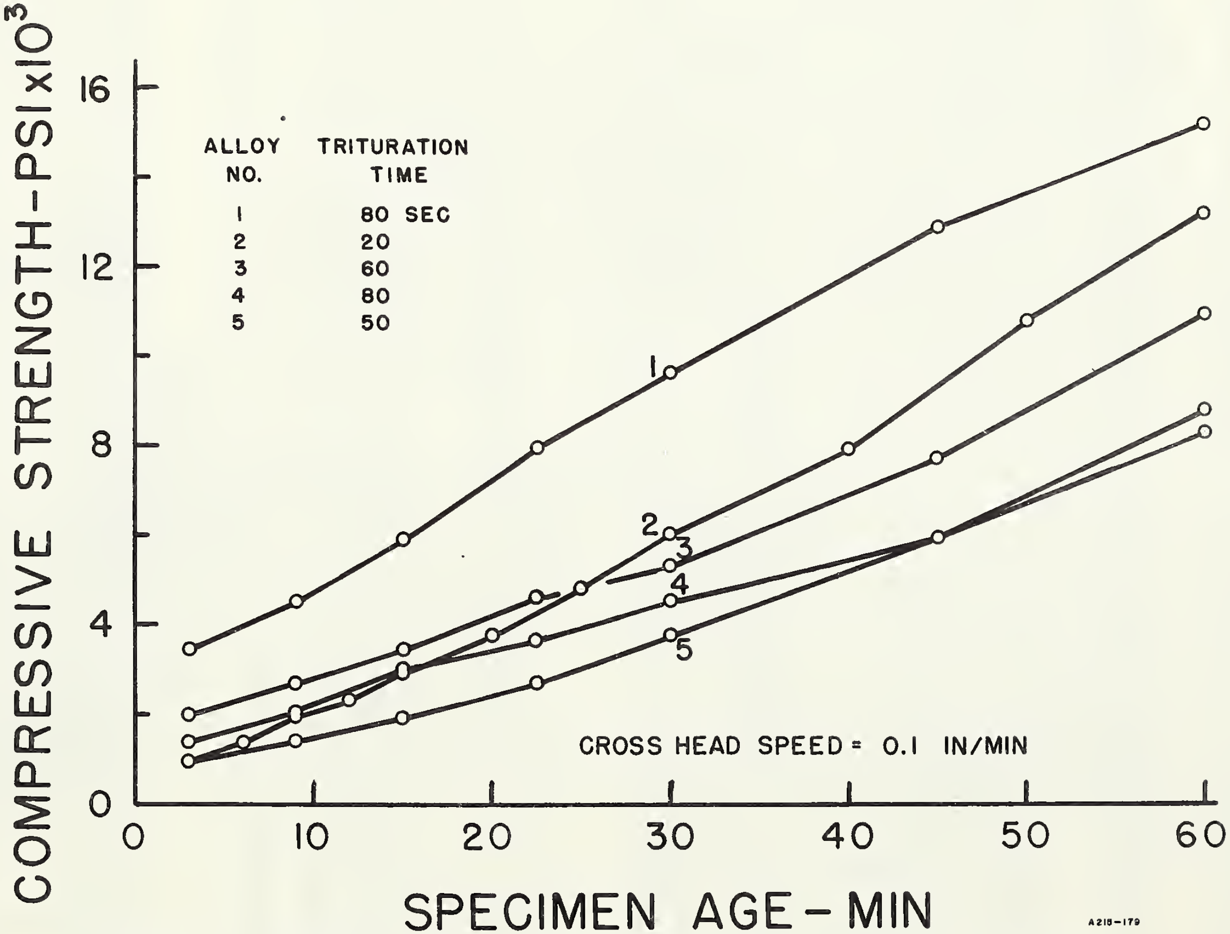


Figure 1. Relation between compressive strength and age of specimen. Age of specimen is the time from the end of trituration to the time the load is applied. The times of trituration given in the upper lefthand corner are the minimum times at which the approximate highest compressive strength at one day was attained. A Model 1, S. S. White amalgamator, and a nylon capsule without pestle were used. The numbers given to the amalgams are in sequence with decreasing compressive strength on 30-minute-old specimens.

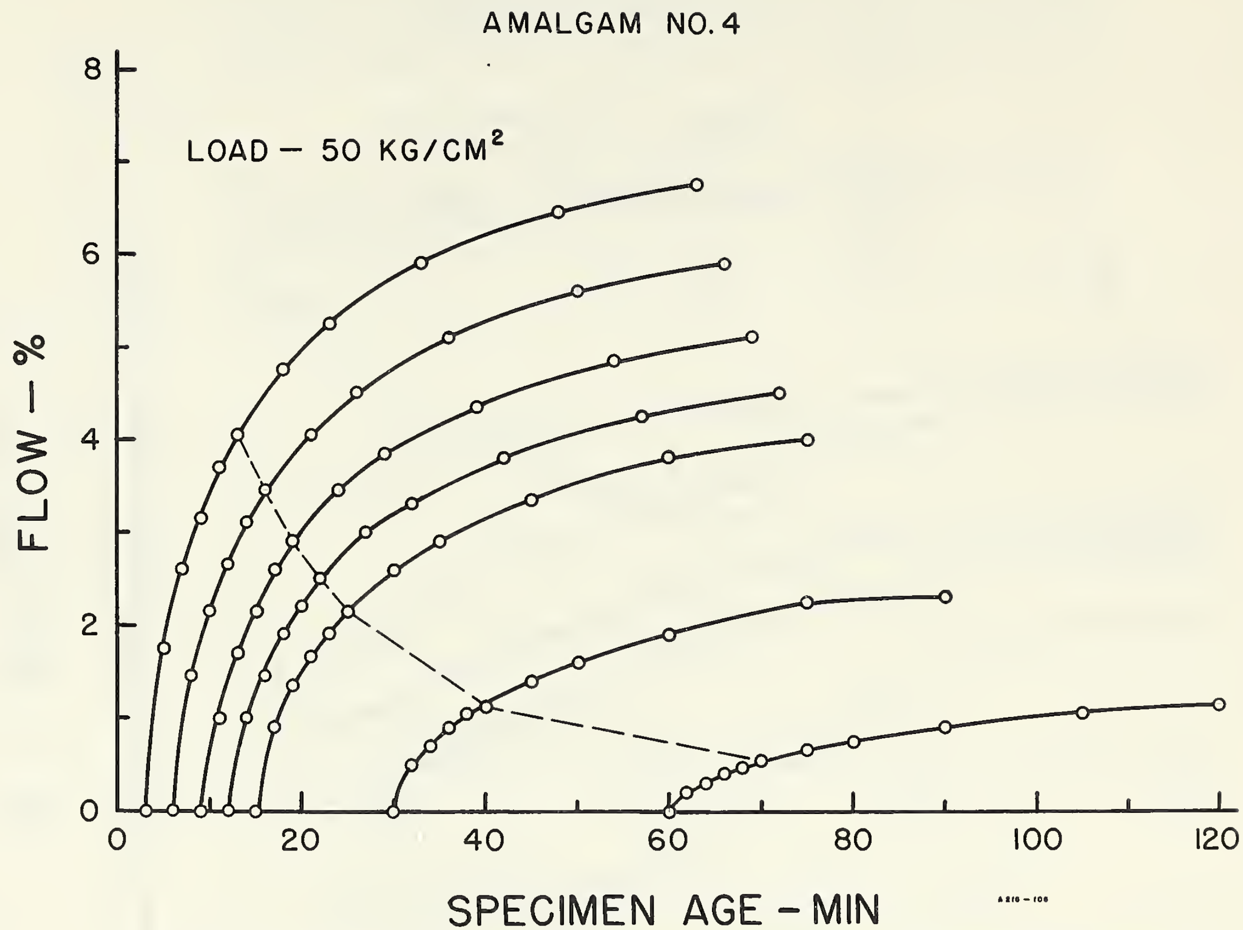


Figure 2. Family of curves showing the effect of the age of the specimen on flow of amalgam No. 4. Broken line connects points at 10 minutes after application of load.

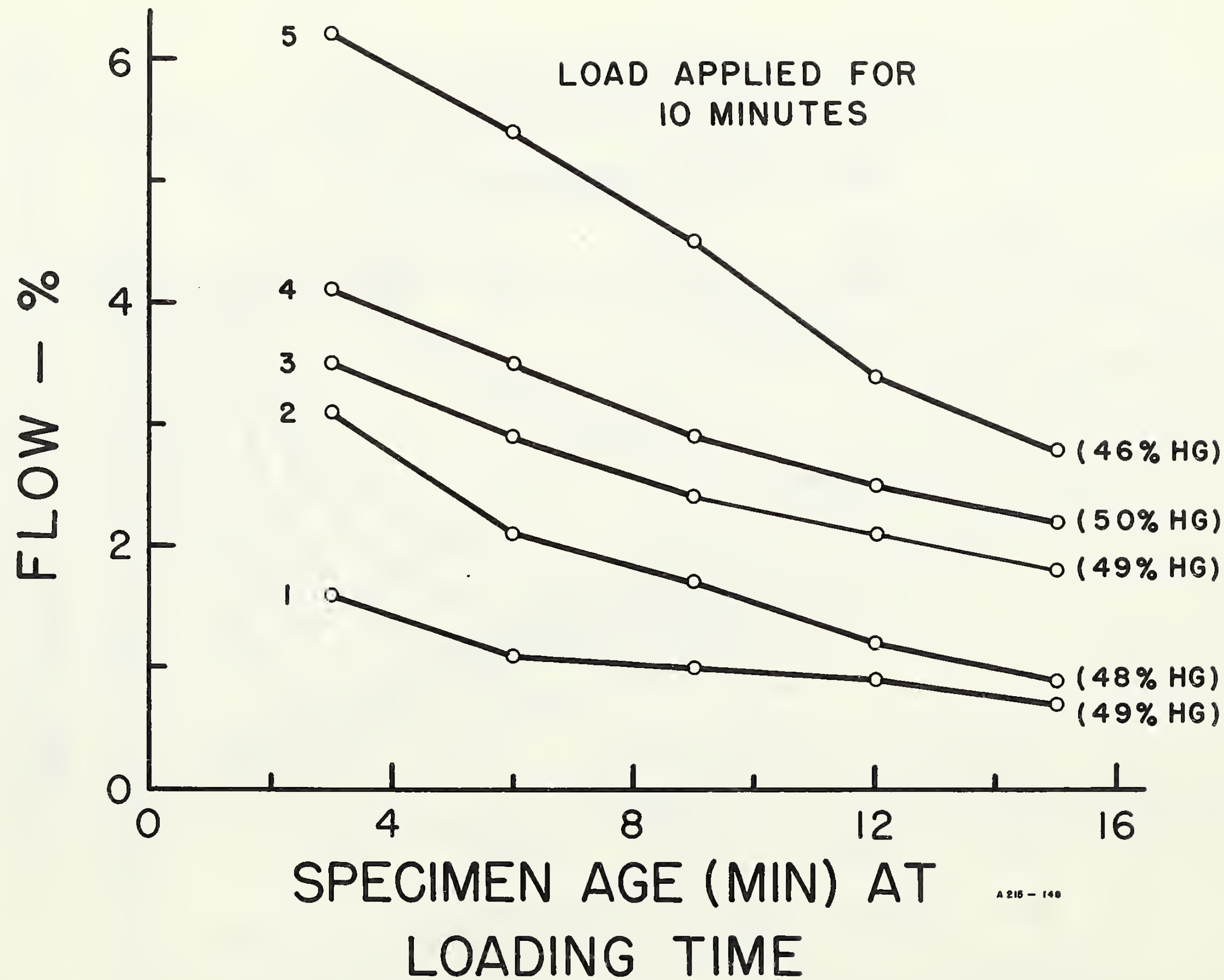


Figure 3. Relation between the age of specimen and flow for the five amalgams. The initial points on the left of the curves represent the amount of flow in percent which occurred when 3-minute-old specimens were subjected to a stress of 50 $kg/cm^2$ for 10 minutes; the second points for 6-minute-old specimens, and so on. The figures in parentheses at the right end of the curves give the calculated percentages of residual mercury in the specimens.

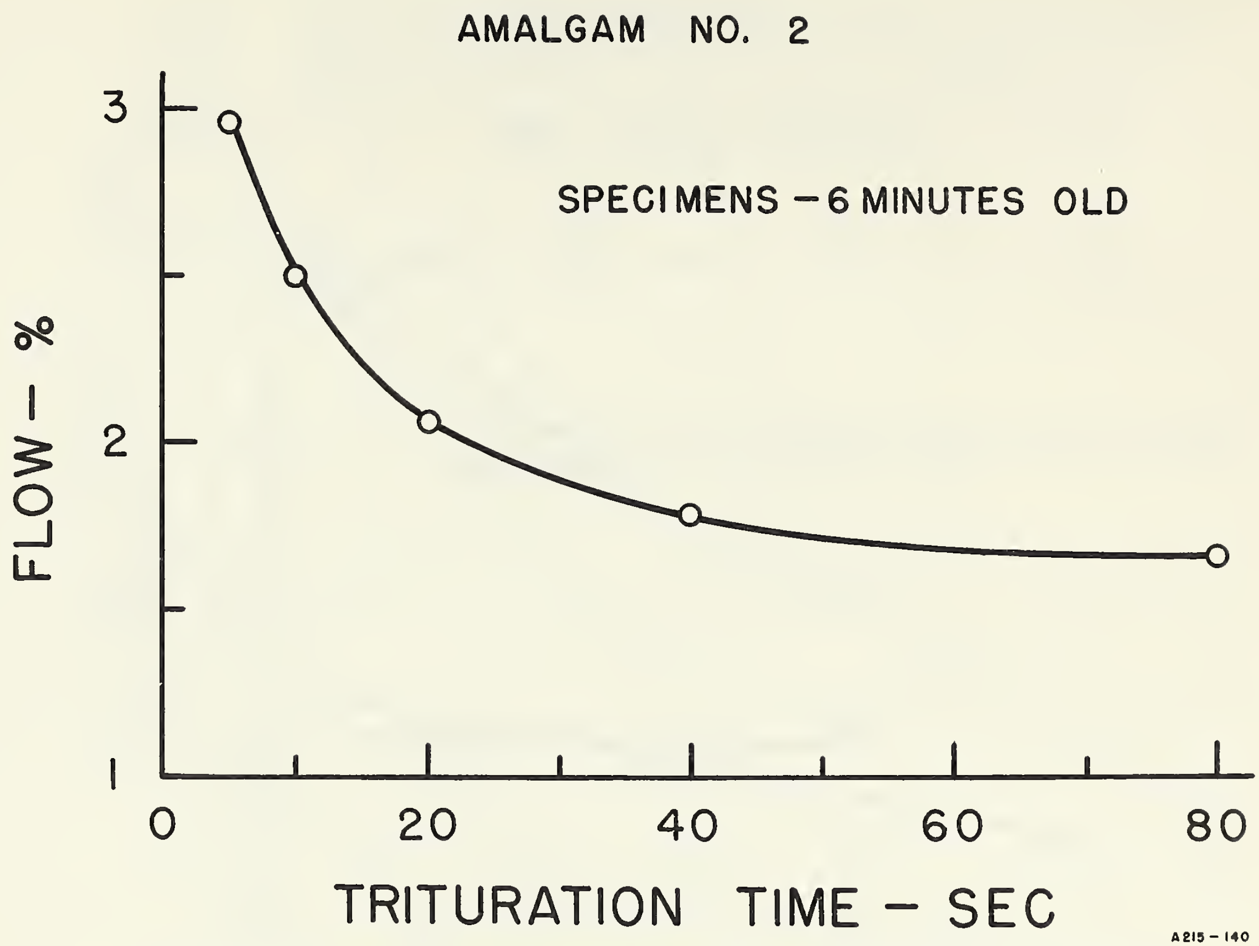


Figure 4. Effect of trituration time on flow of 6-minute-old specimens of amalgam No. 2. A 50-kg/cm$^2$ stress was applied for 10 minutes.

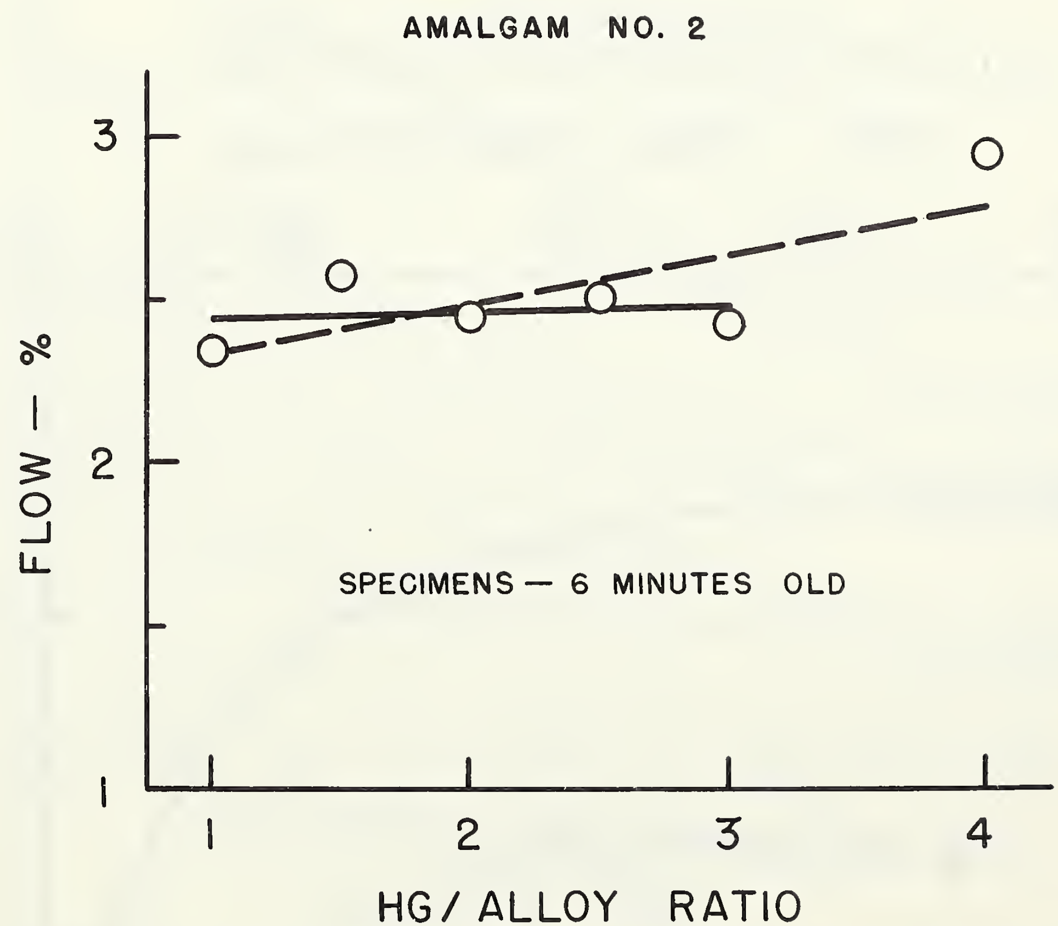


Figure 5. Effect of mercury-alloy ratio on flow. A 50-kg/cm$^2$ stress was applied for 10 minutes. The horizontal line represents the data for mercury-alloy ratios from 1 to 3, and the broken line, the 1 to 4 ratios. Both lines were fitted by the least squares method.

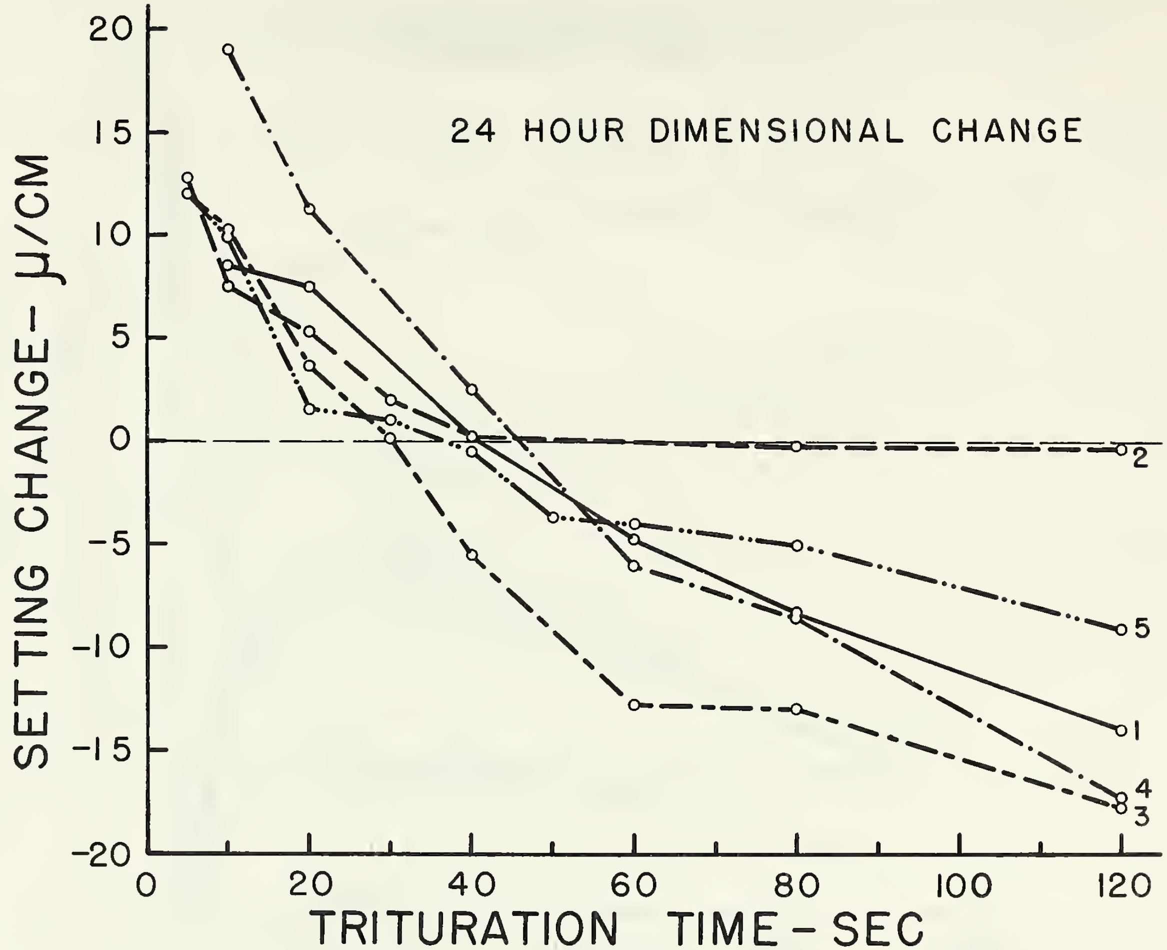


Figure 6. Effect of trituration time on 24-hour dimensional change. The dimensional change during 24 hours represents the difference in the length of the specimen (μ/cm) determined at 15 minutes after the end of the trituration and at 24 hours.

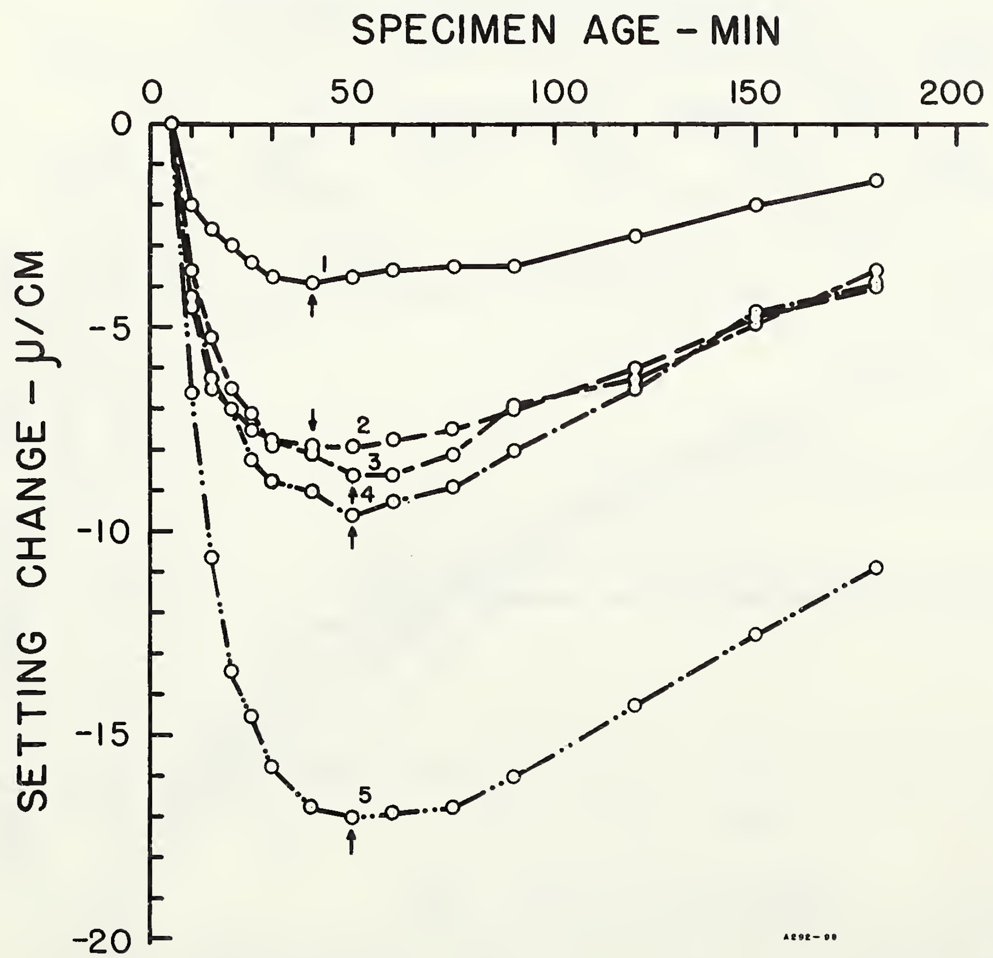


Figure 7. Dimensional setting change occurring during the first three hours. The fiducial reading was taken when the specimen was 5 minutes old. Arrows indicate time of maximum shrinkage.

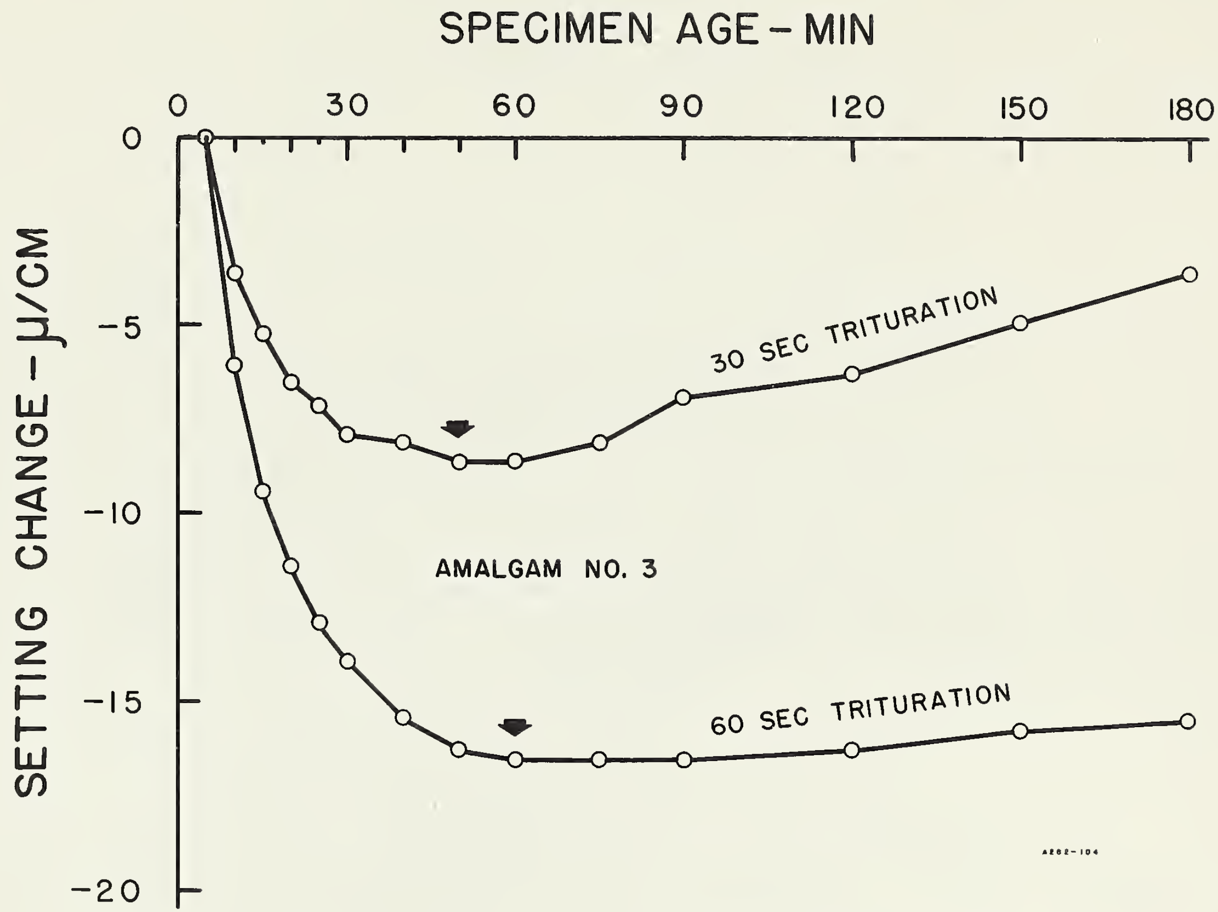


Figure 8. Effect of doubling the trituration time of amalgam No. 3 on setting change during the first three hours and on the time of maximum shrinkage (arrows).

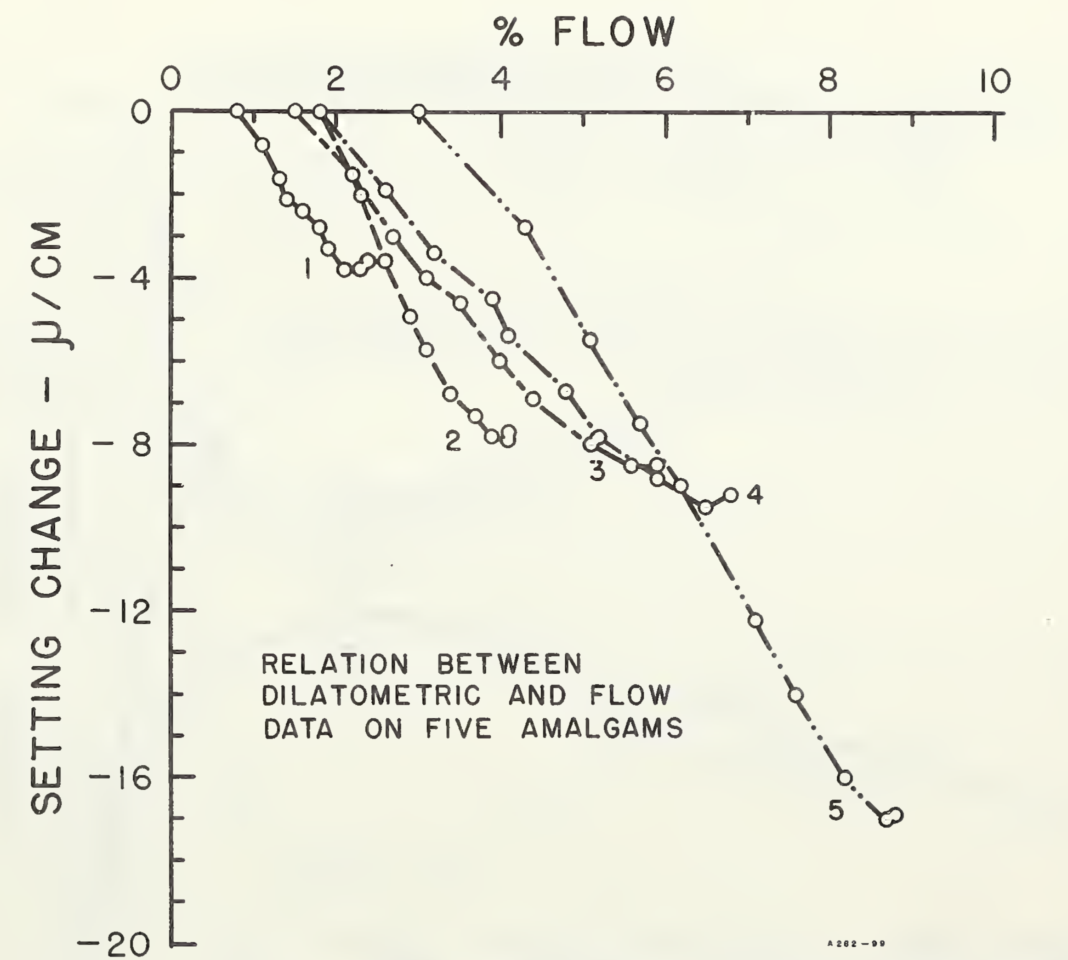


Figure 9. The relation between linear dimensional change occurring during the hardening of five dental amalgams and their flow under a pressure of 50 $kg/cm^2$. The individual points on the curves represent readings taken when the specimens were 5, 7, 9, 11, 13, 18, 23, 33, 48 and 63 minutes old. Ages of the specimens were reckoned from the end of trituration.

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