# NATIONAL BUREAU OF STANDARDS REPORT

7708

Progress Report

on

Effect of Rate of Loading,
Time of Trituration and
Test Temperature on the Compressive
Strength Values of Dental Amalgam

by

Harold J. Caul
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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

# THE NATIONAL BUREAU OF STANDARDS

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A complete listing of the Bureau's publications can be found in National Bureau of Standards Circular 460, Publications of the National Bureau of Standards, 1901 to June 1947 ($1.25), and the Supplement to National Bureau of Standards Circular 460, July 1947 to June 1957 ($1.50), and Miscellaneous Publication 240, July 1957 to June 1960 (Includes Titles of Papers Published in Outside Journals 1950 to 1959) ($2.25); available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C.

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U. S. DEPARTMENT OF COMMERCE
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# Effect of Rate of Loading, Time of Trituration and Test Temperature on the Compressive Strength Values of Dental Amalgam


## Abstract

Factors affecting the compressive strength values of dental amalgams were investigated. Values for five amalgams increased with increase in rates of loading. When testing times were more than one minute, machines applying a constant stress rate gave higher values than did those applying a constant strain rate. Differences between testing machine characteristics prevented close agreement on strength values obtained by four laboratories. Fast loading rates, causing fracture in less than one minute, produced strength values less dependent on type of loading machine than did slower rates of either stress or strain application. An optimum trituration time defined as the minimum time necessary to produce maximum or near maximum strength was observed for each amalgam. As the testing temperature was increased from 23 to 60°C, compressive strength decreased about 50 percent.

## 1. Introduction

The strength of any dental filling material is an important and pertinent property as dental restorations are subjected to rather high stresses. Values for compressive strength have been used extensively in the general evaluation of dental amalgam and for testing specific effects of composition, treatment of the alloy, and variations of methods in preparing the specimens[1-17]. These reports show need for standardization of procedures.

Ware and Docking [14] developed a system of preparing specimens mechanically that greatly increased the reproducibility of compressive strength values as shown by small standard deviations. Increasing the head speed of a testing machine has been shown to increase the compressive strength values from 30 to 50 percent [12, 17]. Gray [1] showed the effect of temperature variations between 22 and 98°C (72 and 208°F) and the effect of time of trituration on compressive strength but used impractical methods in preparing specimens.

In the present study the effects of variations in loading rate were investigated. A difference is demonstrated between the compressive strength obtained by loading the specimens at a constant stress rate (Olsen machine, pendulum type) and at a constant strain rate ( Instron machine employing a load cell). The effects of different testing machines were further shown by cooperative tests among four laboratories. This study also shows that an optimum minimum trituration time for the development of the compressive strength of an individual amalgam can be determined by noting the change in compressive strength with increase in time of trituration. Data are presented on the large decrease in strength of amalgam as the testing temperature is increased.

## 2. Procedure and Results

### 2.1 Specimen Preparation

Specimens of the five alloys (Table 1) were cylinders 4 ± 0.01 mm (0.158 ± 0.0004 in.) in diameter and 8 ± 0.4 mm (0.315 ± 0.016 in.) long. All specimens were made and stored at 23°C (73°F). To make the specimens, 0.6 ± 0.01 gram of alloy and 0.92 ± 0.03 gram of mercury were triturated in an S. S. White mechanical amalgamator controlled by an electric clock to within 1/4 second (Fig. 1). The rate of trituration with this amalgamator allows a wide spread in the time scale.

After trituration the entire mass of mixed amalgam was placed in the mold and a pressure of 150 kg/cm$^2$ (2,100 psi) was applied by a weight through an arbor press fitted with ball bearings to reduce friction (Fig. 1). The procedure and time schedule used in preparing the specimens are outlined in Table 2.

The surfaces of the ends of the specimens were imparted by the ends of the plungers (C in Fig. 1) and the customary carving of one end of the specimen to adjust its length was avoided. Time during which the pressure is applied (Table 2) was reduced to as little as one minute with no effect on the strength. Different amounts of alloy (0.50 to 0.65 gram) and proportional amounts of mercury varied the specimen length from 6.7 to 9.0 mm (0.264 to 0.354 in.) with no change in compressive strength. This procedure not only avoids carving one end of the specimen, which may disturb the surface hardening, but also removes the mercury quickly.

The values for strength are averages of six specimens unless otherwise noted. The coefficients of variation indicate the reproducibility of the measurements.

## 2.2 Strength Testing Machines

Two strength testing machines were used in the Dental Research Section of the National Bureau of Standards. One, a Tinius Olsen machine, employs pendulum loading, while the other, an Instron machine, employs an electric-strain-gauge load cell and operates at constant rates of strain.

Data for comparing these two machines were collected with three-week-old specimens. The rate of loading of the Tinius Olsen machine was adjusted to 100 lb/min (45.5 kg) resulting in a constant stress rate of 5,100 psi/min (351 kg/cm$^2$). At this rate the specimen failed in about eight minutes by shattering. The rate of loading of the Instron testing machine was adjusted to a rate of strain that caused failure of the specimens in approximately the same time. The head speed was 0.002 in./min (0.005 cm/min). At this speed the specimens did not shatter but the stress went through a maximum and then began to decrease as the

specimen became barrel shaped. With both machines the stress rate and the strain rate were obtained by recording the load and the strain every 30 seconds until failure. The load was read directly from the machines. The strain was obtained by noting the change in distance between the platens with a dial gauge.

Stress and strain rates on single specimens are shown in Figure 2 for the Tinius Olsen machine and in Figure 3 for the Instron machine. One of these specimens (Fig. 2) shattered in the Tinius Olsen machine at 44,700 psi (3,140 kg/cm$^2$) and the other specimen (Fig. 3) failed in the Instron machine after sustaining a maximum stress of 40,600 psi (2,850 kg/cm$^2$). These failures for single specimens present typical patterns in which the strain rate was constant in the Instron machine but not in the Tinius Olsen machine. The stress rate was nearly constant until failure in the Tinius Olsen machine but not in the Instron machine where the stress rate became zero at failure. With the Tinius Olsen machine the strain rate was relatively constant up to stresses of about 25,000 psi (1,760 kg/cm$^2$) (Fig. 2), and then the strain rapidly increased until the specimen shattered. Since strain was calculated from the change in length of the specimen and not read directly from strain gauges attached to it, the strain data include effects caused by slight roughness and unparallelism of the ends of the specimen.

The data show that conditions for obtaining values for compressive strength cannot be prescribed by specifying a time interval in which the specimen is to be broken unless a constant stress rate, a constant strain rate, or a particular type of testing machine is also specified.

## 2.3 Rate of Loading

The head speed of the Instron machine used in this report ranged from 0.002 to 0.2 in./min (0.005 to 0.51 cm/min). These speeds required from 6 3/4 minutes to 4 seconds, respectively, to fail a specimen (Table 3). At head speeds of 0.002 to 0.05 in./min (0.005 to 0.127 cm/min)

reproducibility was very good as the coefficient of variation was never more than 1.7 percent. Also at these rates values from 42,000 to 63,000 psi (2,950 to 4,430 $kg/cm^2$) were obtained depending on the speed used. At head speeds greater than 0.05 in./min (0.127 cm/min ) the coefficient of variation increased, that is, the reproducibility was less precise. The compressive strength remained relatively constant at high head speeds (0.05 through 0.2 in./min). At these very high strain rates the strength is more dependent upon the elastic properties of amalgam and dependence upon its plastic properties (flow) is reduced.

Experiments with the Tinius Olsen machine seem to confirm this. At stress rates (47,600 psi/min or greater) where the loading times were 1 1/4 minutes or less, values were relatively constant (Table 4). With the Tinius Olsen machine it was impractical to reduce the loading time below 38 seconds. If the data in Tables 3 and 4 concerning loading time and compressive strength are examined it becomes evident that at loading times of more than one minute a constant stress rate results in a higher compressive strength than a constant strain rate.

These data indicate that a more meaningful value for compressive strength can be obtained on testing machines with different characteristics if high rates of loading are used.

## 2.4 Cooperative Testing

Randomly selected amalgam specimens prepared at the National Bureau of Standards, as in Table 2, were tested by each of the laboratories listed in Table 5 when the specimens were about two months old. These laboratories were instructed to test any five of the specimens in less than one minute each and to test the remaining five specimens in approximately 6 minutes each. Table 6 which lists the name and type of each machine shows the wide variety of machines used to obtain the data.

The results of these tests are shown in Figure 4 and Table 6. If complete homogeneity of the specimens was assured, the observed values in Figure 4 should fall on a horizontal or a vertical line if the characteristics of the several machines had been completely eliminated at either the slow or fast rates of loading, respectively. The ideal situation would be one point on the graph which would mean complete homogeneity among the specimens and identical operating characteristics among the testing machines at all loading rates. The variation in compressive strength caused by variation in the loading rates (Table 6) confirms the observation of others [12, 16].

The average coefficient of variation for the slow loading rate was 1.2 percent and for the fast rate was 2.2 percent. These data in Figure 4 and Table 6 show that the range of average values obtained at high loading rates [5,700 psi (410 kg/cm$^2$)] was not much less than that obtained at low loading rates [6,500 psi (460 kg/cm$^2$)].

## 2.5 Trituration Time

The data in Figure 5 show variation in compressive strength when the time of trituration extends over the range of 5 or 10 seconds to 120 seconds. In all cases the compressive strength is much lower at 5 or 10 seconds trituration than for longer periods of trituration. The strength appears to increase and finally arrives at a plateau for 20 to 80 seconds trituration. The optimum time of trituration on the basis of compressive strength is taken as the minimum time required to develop maximum or near maximum strength.

Use of the metal pestle has little effect on the results. Typical results for two of the five alloys are shown in Figure 6.

## 2.6 The Effect of Temperature

Gray's data [1] showed a linear decline in compressive strength of 1,130 psi/ °C (79.4 kg/cm$^2$/ °C) from 23°C (73°F) to 70°C (158°F). The strength at 70°C (158°F) was about 32 percent of the strength at 23°C (73°F). He used a packing pressure of about 7,250 psi (510 kg/cm$^2$) for 8 minutes

in preparing the specimens so his values for compressive strength were very high.

Nelsen et al [18] reported that 60°C (140°F) is approximately the highest temperature at which four ounces of hot liquids could be consumed in a few seconds. With these data as guides, an instrument was devised for measuring the compressive strength of amalgam at temperatures from 23°C (73°F) to nearly 70°C (158°F) (Fig. 7). The inner core of the furnace (Fig. 8) was a thick cylinder of copper to provide a large heat capacity and uniform temperature in the core of the furnace. The copper core weighed 1.8 kg (4.0 lb), was 14.6 cm (5 3/4 in.) long, and had a 4.4 cm (1 3/4 in.) outside diameter and a 1.4 cm (9/16 in.) inside diameter.

Figure 9 shows the position of the thermocouple during the test. The furnace was positioned on the machine with the plywood spacer (Fig. 10), with the ends of the platens about 8 millimeters apart. After the furnace was brought to the desired temperature [± 2°C (± 3 1/2°F)], the spacer was removed exposing the ends of the platens (Fig. 8) and the specimen was inserted quickly. An amalgam specimen, in which a thermocouple was inserted, reached furnace temperature within two minutes. In actual testing, three minutes were allowed for the specimen to reach the furnace temperature.

The relationship between compressive strength and testing temperature for the five alloys is represented by a family of curves (Fig. 11) having similar negative slopes with decreasing strength at a rate of approximately 700 pounds per square inch per degree centigrade as the temperature increases. Average values and the relative strength at various temperatures are given in Table 7. At 60°C (140°F), an estimated maximum temperature that amalgam restorations may reach, the strength is 50 percent of that at room temperature. Even at body temperature [37°C (98.6°F)] a 15 percent loss in strength has occurred.

## 3. Summary and Conclusions

The compressive strength values of five widely used amalgams were influenced by how the testing machine applied the load. Strengths were higher on machines that applied a constant stress rate than on those applying a constant strain rate when the testing time was greater than one minute.

Four research laboratories determined the compressive strength of two-month-old amalgam specimens prepared at the National Bureau of Standards. When about six minutes were required to fail the specimens, the range among the laboratories was from 49,500 to 56,000 psi (3,480 to 3,940 $kg/cm^2$). When the testing time was shortened to about one minute, the range was from 59,900 to 65,600 psi (4,210 to 4,610 $kg/cm^2$). These higher values which are obtained at fast loading times confirm the experience of several investigators [12, 17].

The optimum time for triturating each amalgam on the basis of strength was taken as the minimum time to develop maximum or near maximum compressive strength. The data indicating that under-trituration of amalgam can be expected to produce a weak alloy should be given careful consideration by clinicians.

The compressive strength of amalgam at 60°C (140°F) is roughly 50 percent of its strength at 23°C (73°F). The relation is linear as Gray previously showed [1].

TABLE 1

| Brand | Batch Number | Manufacturer |
|---|---|---|
| Aristaloy | 540548 | Baker Dental Division Englehard 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 Company |
| Twentieth Century Micro Non Zinc | 30H61 | L. D. Caulk Company |

TABLE 2

Specimen Preparation

| Procedure | Time | |
|---|---|---|
| | Min. | - Sec. |
| End of Trituration | 0 | 00 |
| Place mix in mold and apply 150 $Kg/cm^2$ (2,100 lbs./sq. in.) at | 0 | 30 |
| Remove pressure at | 4 | 00 |
| Eject specimen at | 4 | 15 |

TABLE 3

Effect of Strain Rate on Compressive Strength (Instron Machine)

| Head Speed | Loading Time* | Strain Rate | 24 Hour Compressive Strength | Coefficient of Variation | Remarks |
|---|---|---|---|---|---|
| In./Min. | Min.-Sec. | In./In./Min. | PSI | % | |
| 0.002 | 6 3/4 | 0.0067 | 43,500 | 0.9 | No specimen shattered |
| 0.002 | 6 3/4 | 0.0067 | 42,000 | 1.0 | " " |
| 0.005 | 2 3/4 | 0.017 | 51,000 | 0.0 | " " |
| 0.005 | 2 3/4 | 0.017 | 50,100 | 1.4 | " " |
| 0.01 | 1 - 31 | 0.033 | 54,000 | 0.4 | " " |
| 0.01 | 1 - 30 | 0.032 | 53,800 | 1.7 | " " |
| 0.02 | 0 - 46 | 0.067 | 58,700 | 0.0 | " " |
| 0.05 | 0 - 18 | 0.16 | 62,900 | 1.0 | All specimens shattered |
| 0.1 | 0 - 08 | 0.31 | 61,000 | 2.5 | " " |
| 0.2 | 0 - 04 | 0.63 | 63,000 | 3.7 | " " |

* Loading time is the interval between the start of the test and the shattering of the specimen or until the load begins to decrease after reaching a maximum.

TABLE 4

Effect of Stress Rate on the Compressive Strength (Tinius Olsen Machine)

| Loading Time* | Stress Rate | 24 Hour Compressive Strength | Coefficient of Variation | Remarks |
|---|---|---|---|---|
| Min.-Sec. | PSI/Min. | PSI | % | |
| 8 - 16 | 6,100 | 50,800 | 0.8 | All specimens shattered |
| 4 - 57 | 10,000 | 49,900 | 1.2 | " " |
| 3 - 23 | 16,500 | 55,800 | 1.4 | " " |
| 1 - 16 | 47,600 | 60,500 | 2.5 | " " |
| 0 - 56 | 63,000 | 58,800 | 3.9 | " " |
| 0 - 45 | 79,300 | 59,500 | 1.8 | " " |
| 0 - 38 | 94,400 | 59,500 | 3.5 | " " |

* Loading Time is the interval between the start of the test and the shattering of the specimen.

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TABLE 5

Laboratories Cooperating in the Testing

| Code | Laboratory | Investigator |
|---|---|---|
| A | L. D. Caulk Company | W. Dean Kimmel |
| B | Marquette University | Gunnar Ryge |
| C | S. S. White Dental Manufacturing Co. | George M. Birch |
| D | National Bureau of Standards | Harold J. Caul |

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TABLE 6

Compressive Strength of Amalgam*
Determined by the Cooperating Laboratories

| Code | Testing Machine | | Slow Loading | | | Fast Loading | | |
|---|---|---|---|---|---|---|---|---|
| | Manufacturer | Type | Loading Time Min.-Sec. | C. S.† PSI | V.‡ % | Loading Time Min.-Sec. | C. S. PSI | V. % |
| A | Baldwin-Southwark | Hydraulic | 6-12 | 56,000 | 1.2 | 0-56 | 63,800 | 1.1 |
| B | Riehle | Screw | 3-48** | 55,500 | 2.0 | 0-43 | 59,900 | 4.0 |
| C | Olsen | Hydraulic with constant rate loading device | 6-32 | 51,500 | 0.6 | 0-47 | 65,600 | 2.0 |
| D | Instron | Electric-Strain-gauge load cell constant strain rate | 6-33 | 49,500 | 0.8 | 0-56 | 62,200 | 1.6 |
| Average | | | 5-46 | 53,100 | 1.2 | 0-50 | 62,900 | 2.2 |

* Two months old
** The slowest speed at which the machine could be operated
† Compressive Strength
‡ Coefficient of Variation

TABLE 7

The Average Effect of Temperature on 24 Hour Compressive Strength of Five Amalgams*

| Temperature | Compressive Strength | Relative Strength |
|---|---|---|
| °C | PSI | % |
| 23 | 58,200 | 100 |
| 30 | 53,500 | 92 |
| 37 | 49,500 | 85 |
| 40 | 47,800 | 82 |
| 50 | 41,100 | 71 |
| 60 | 28,100 | 48 |
| 65 | 18,700 | 32 |

* Calculated from Figure 11 with extrapolation.

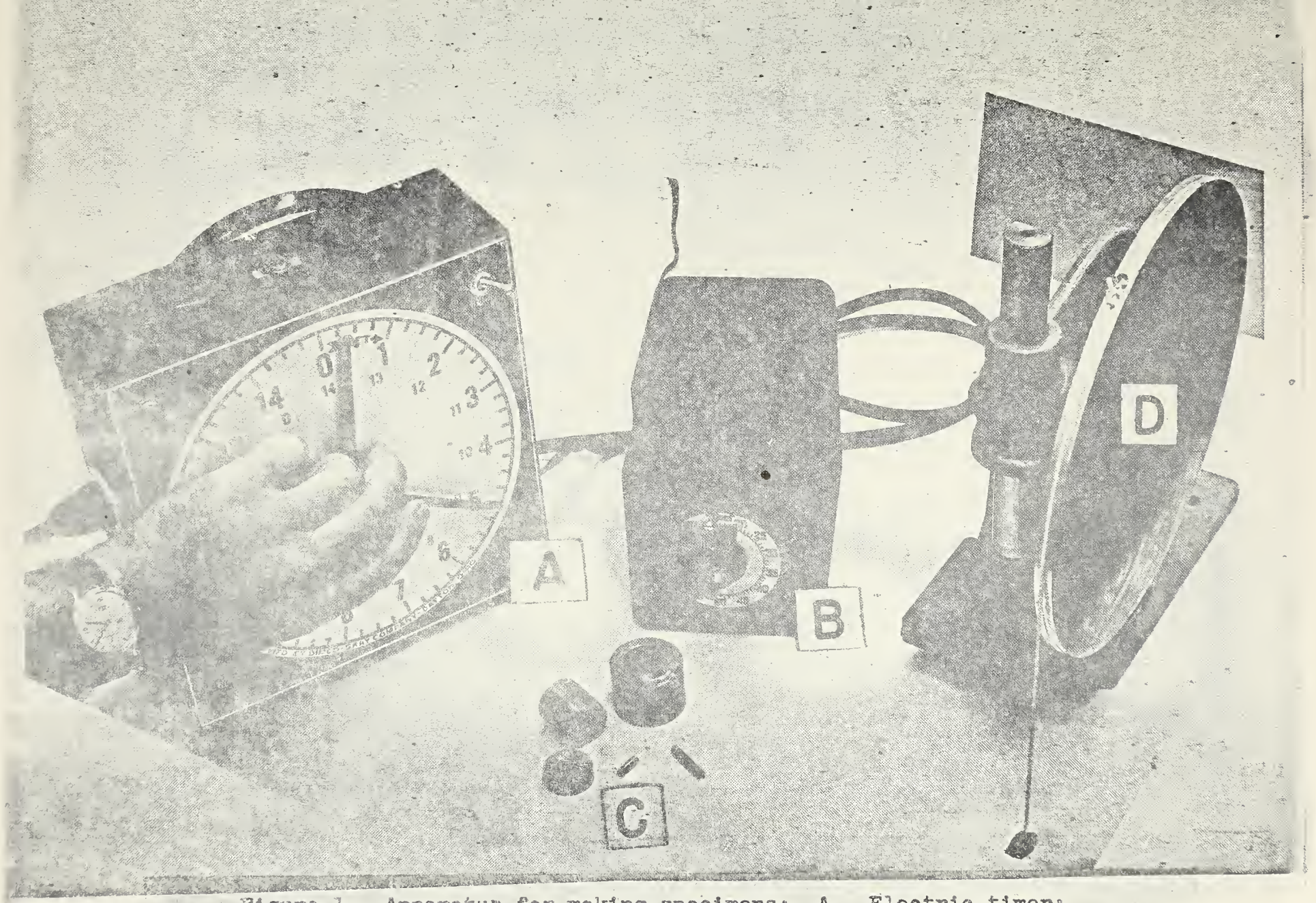


Figure 1. Apparatus for making specimens: A - Electric timer; B - Mechanical amalgamator; C - Mold; D - Arbor press (dead weight is suspended underneath).

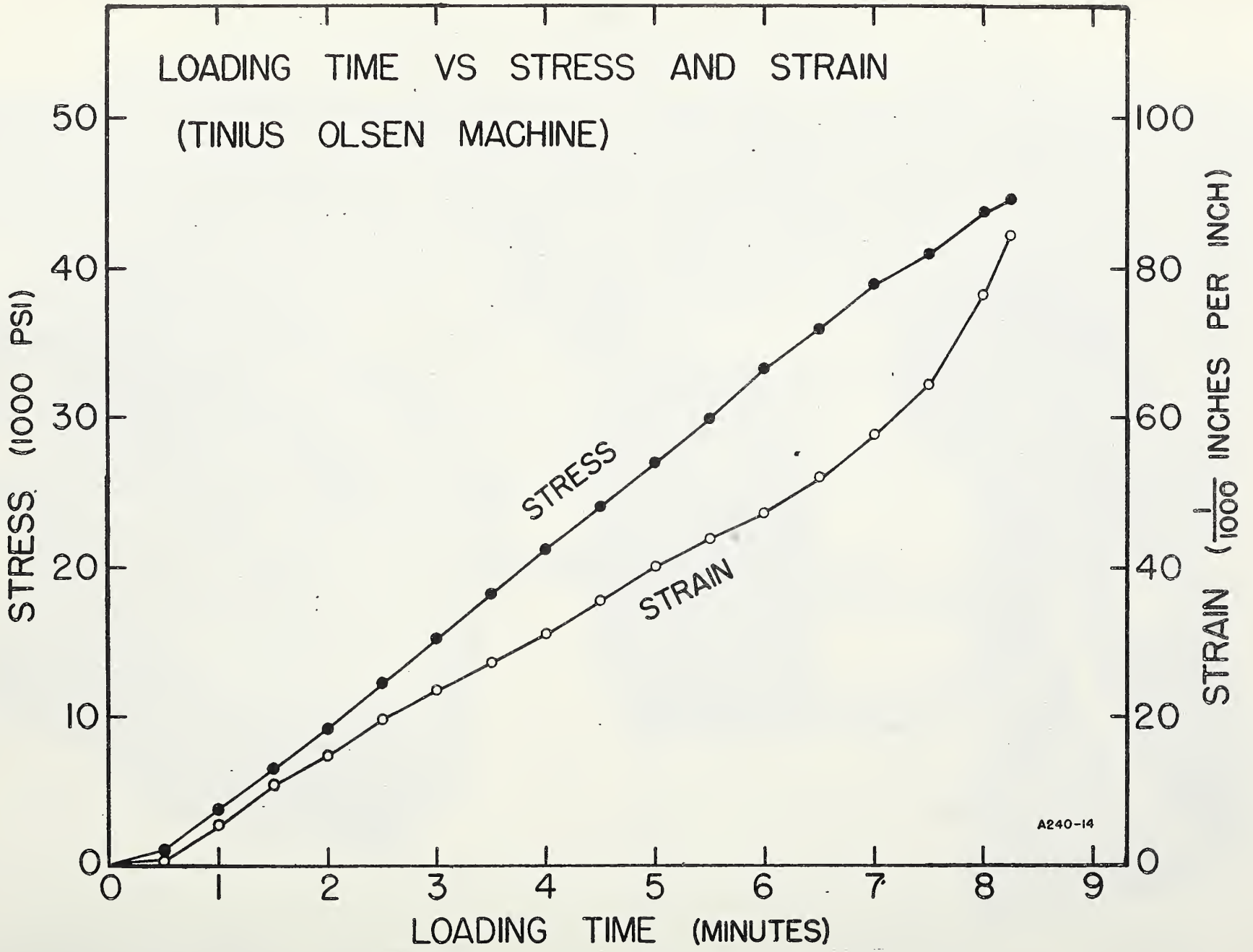


Figure 2. The relationship of stress and strain with respect to time with the Tinius Olsen machine.

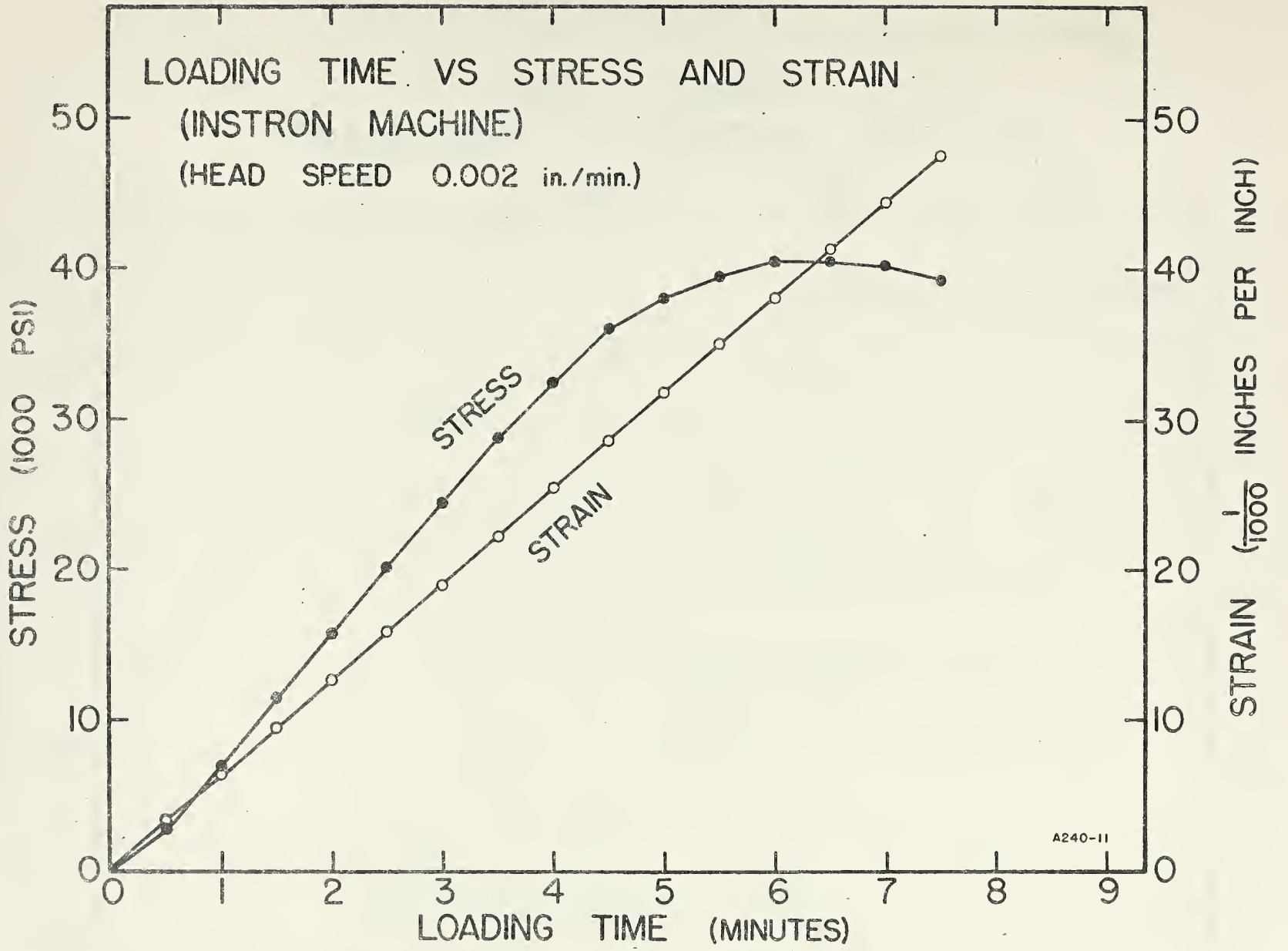


Figure 3. The relationship of stress and strain with respect to time with the Instron machine.

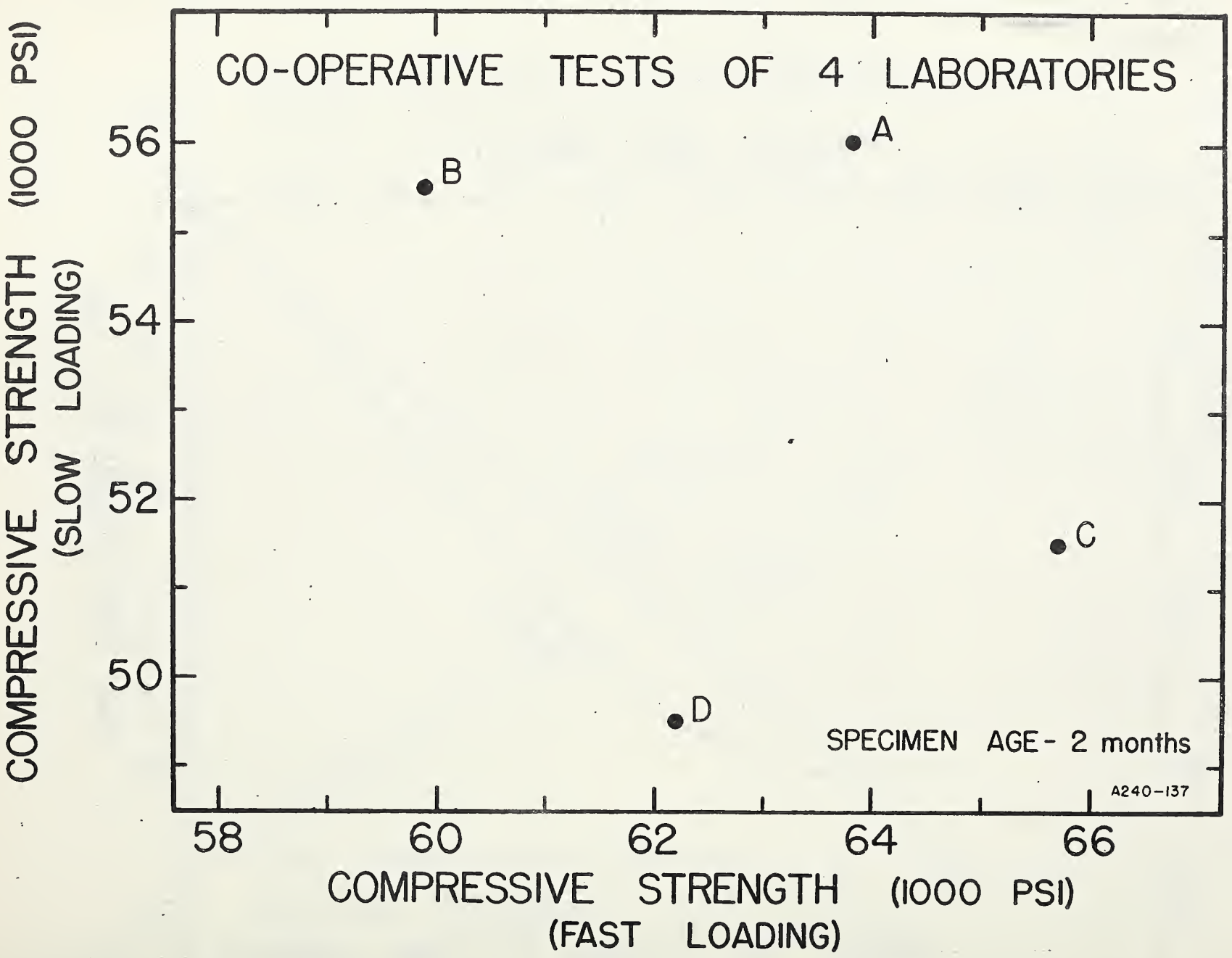


Figure 4. Compressive strength values of four laboratories for slow and fast rates of loading.

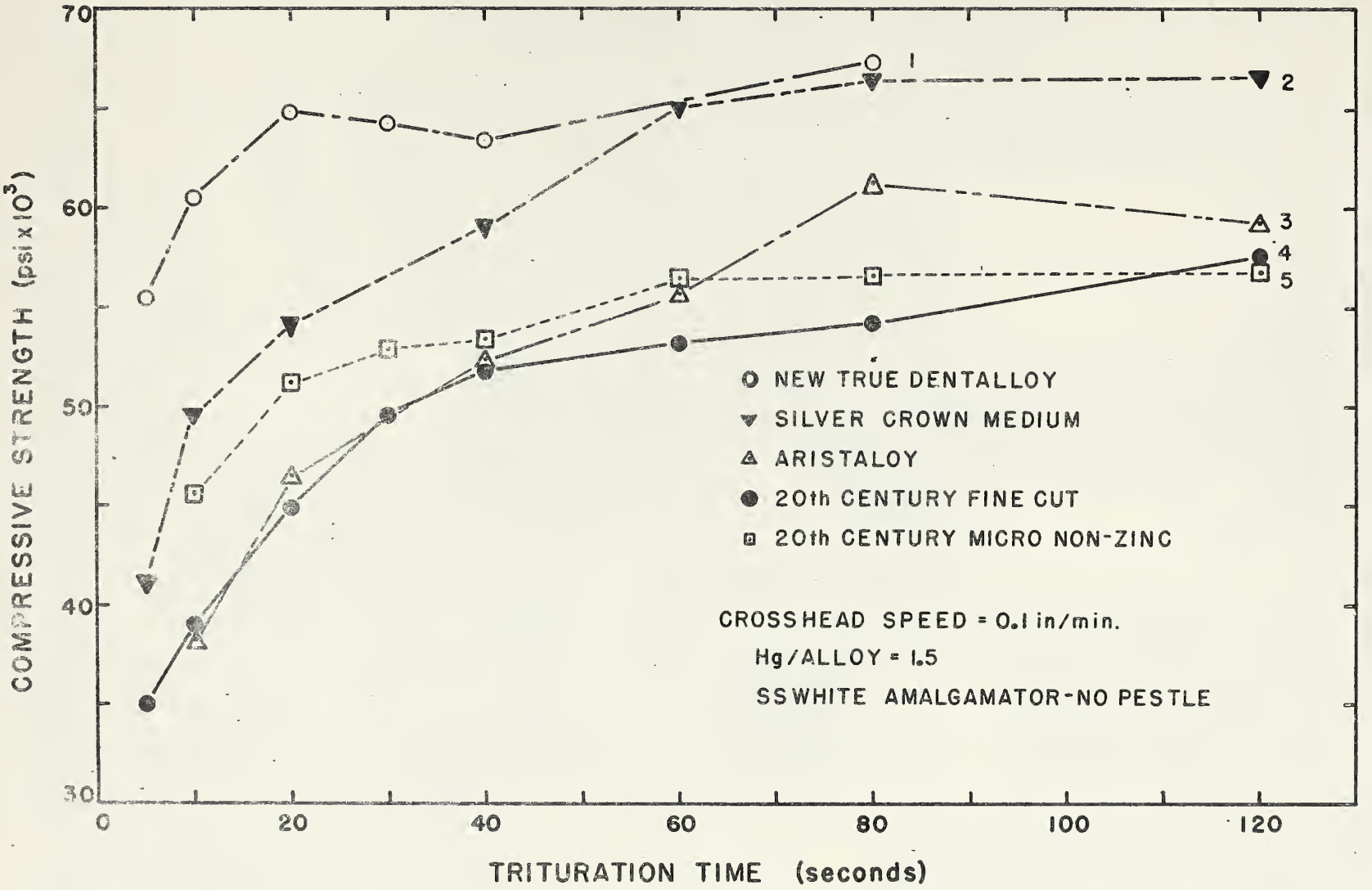


Figure 5. The effect of trituration time on compressive strength.

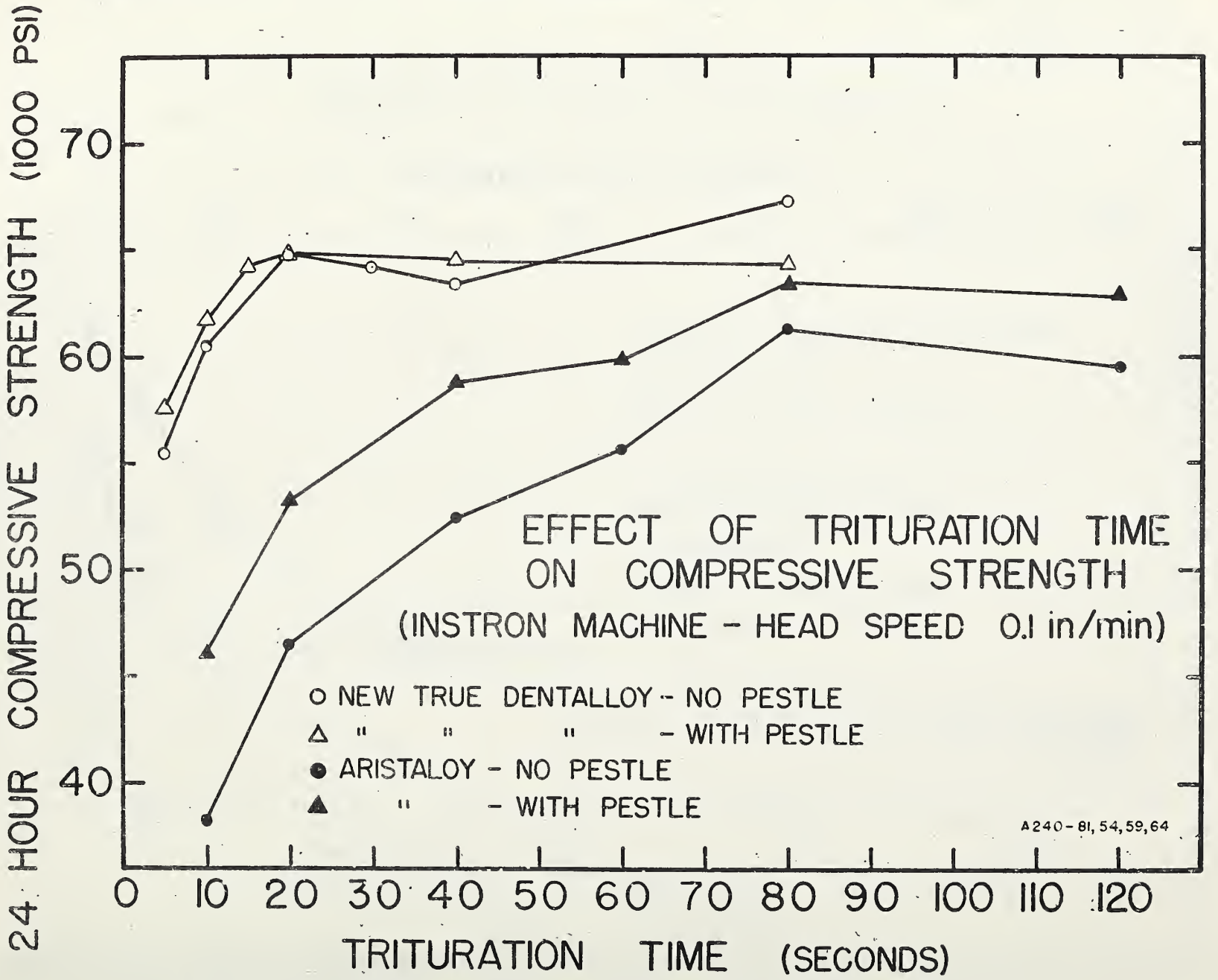


Figure 6. The effect of the steel pestle on trituration and strength.

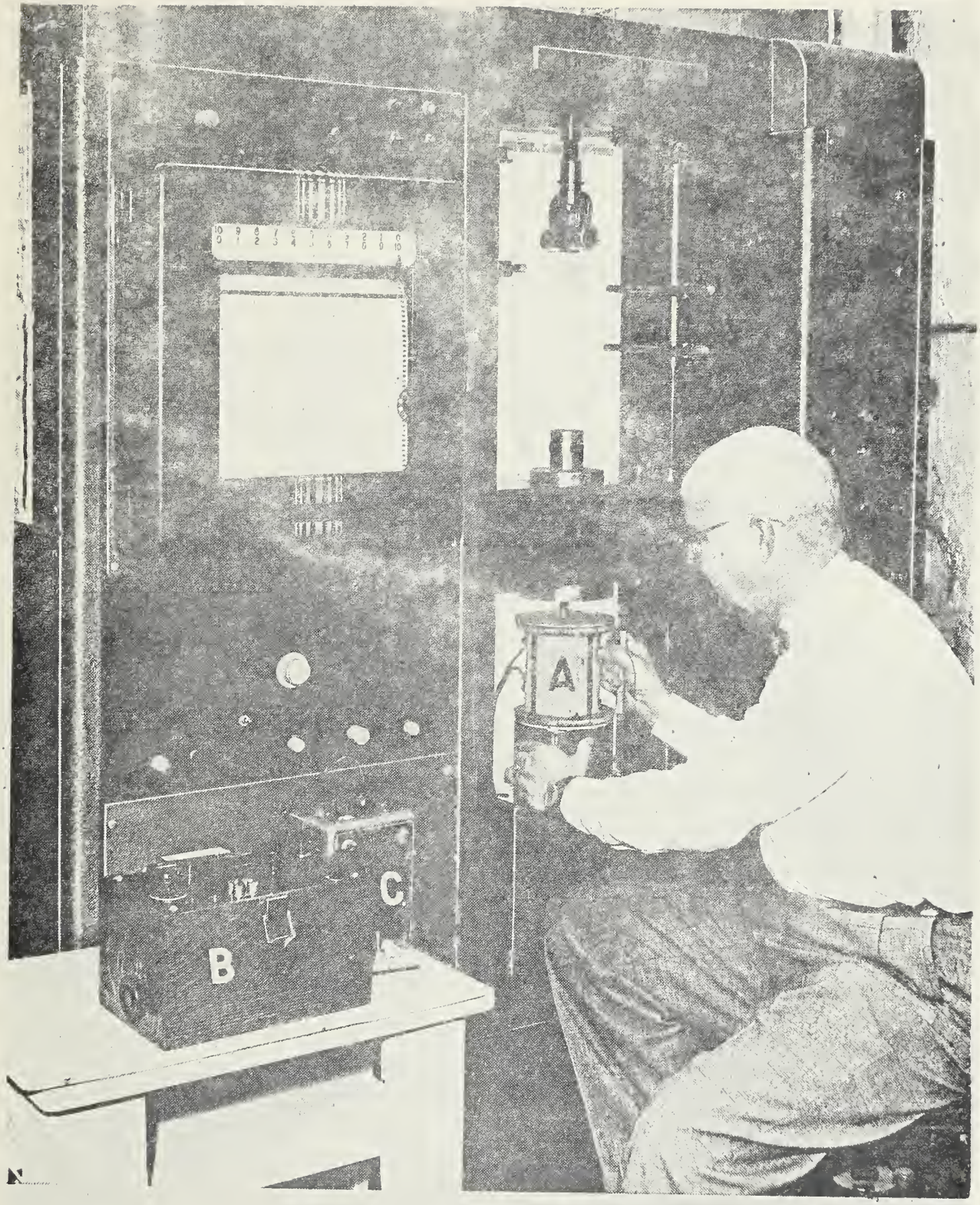


Figure 7. Instrument used in determining compressive strength of amalgam at temperatures up to 70°C.
A-Furnace (see Figures 8, 9, and 10) in position on platens of testing machine.
B-Potentiometer for determining the temperature of the thermocouple shown in Figure 10.
C-Voltage regulator for controlling temperature of the furnace.

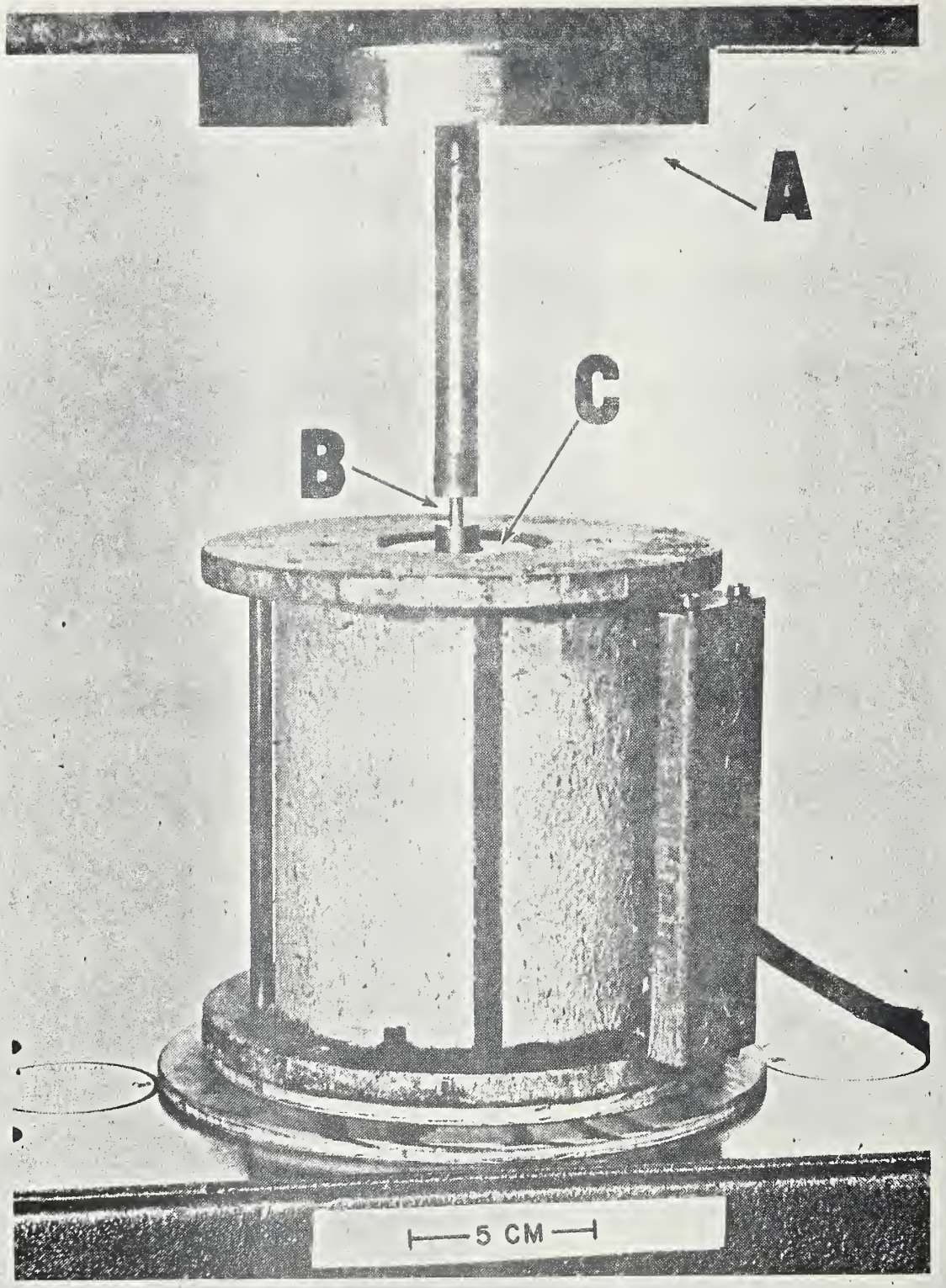


Figure 8. View of specimen between the platens. Furnace in the lowered positions showing specimen in place between the platens of the testing machine.
A-Thermocouple wires
B-Specimen
C-The inner core of the furnace is copper in order to supply a large heat capacity and a uniform temperature.

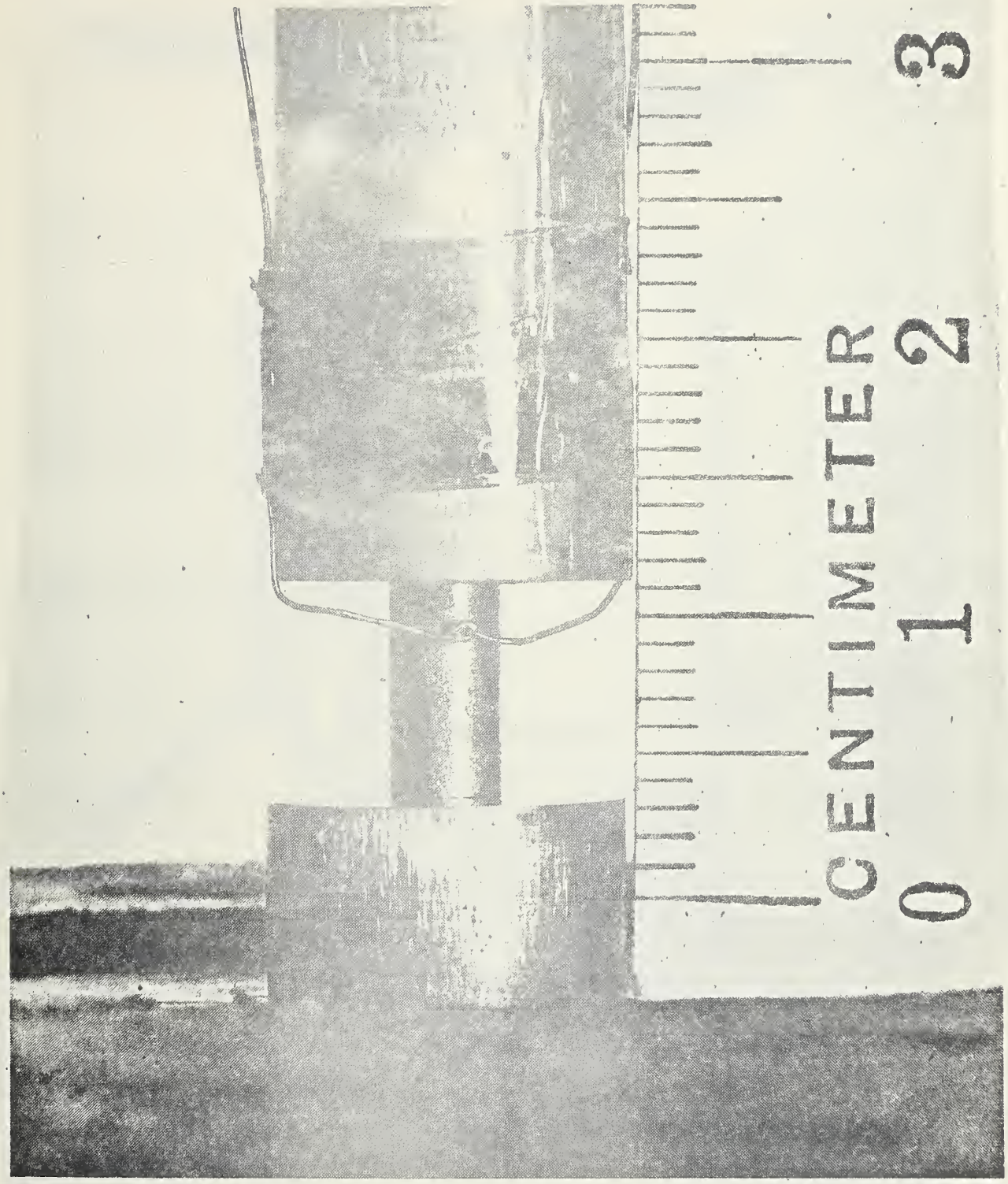


Figure 9. A close up of the specimen showing the thermocouple for measuring temperature.

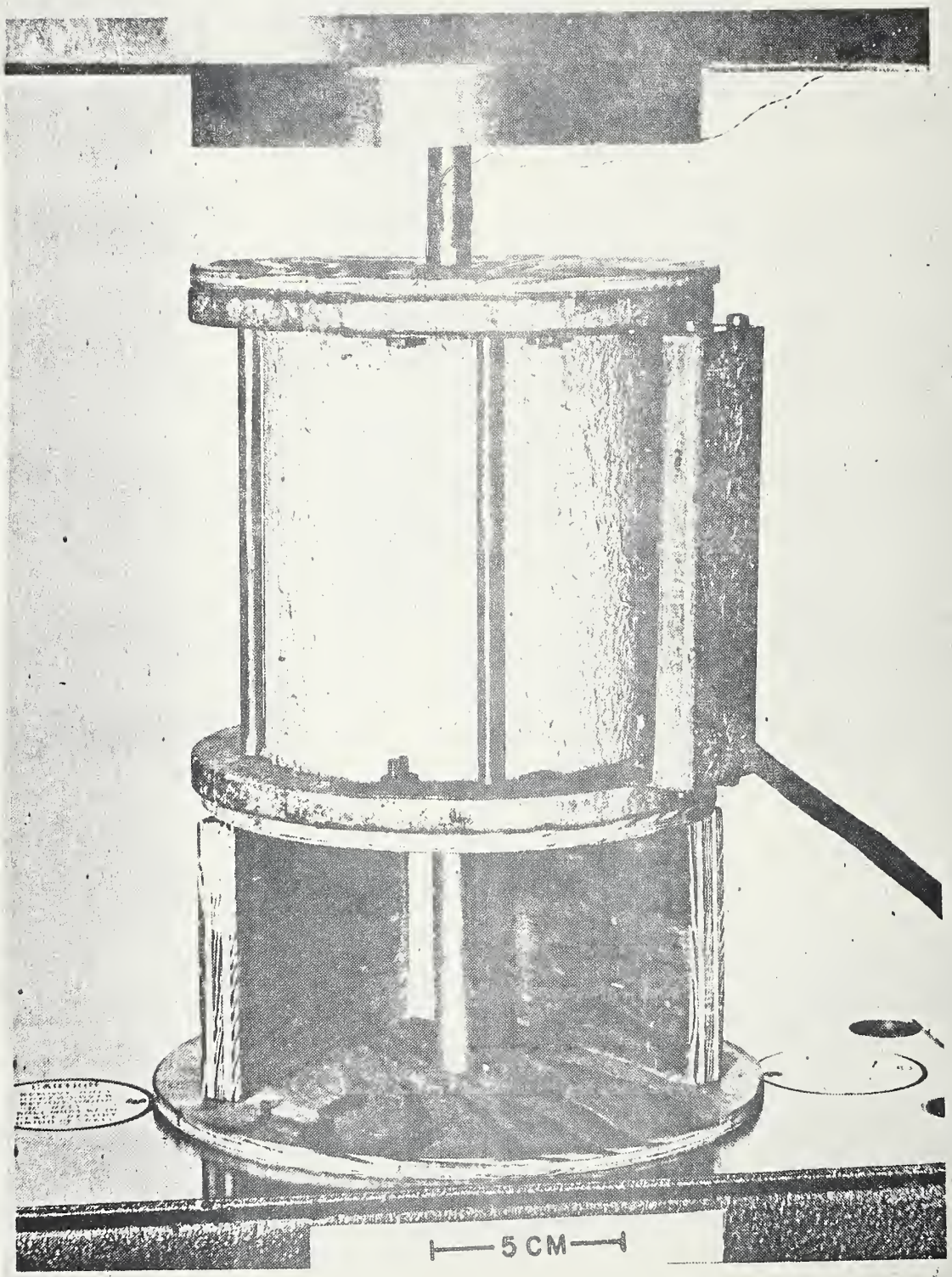


Figure 10. Furnace in position with specimen to be tested in the middle of the furnace.

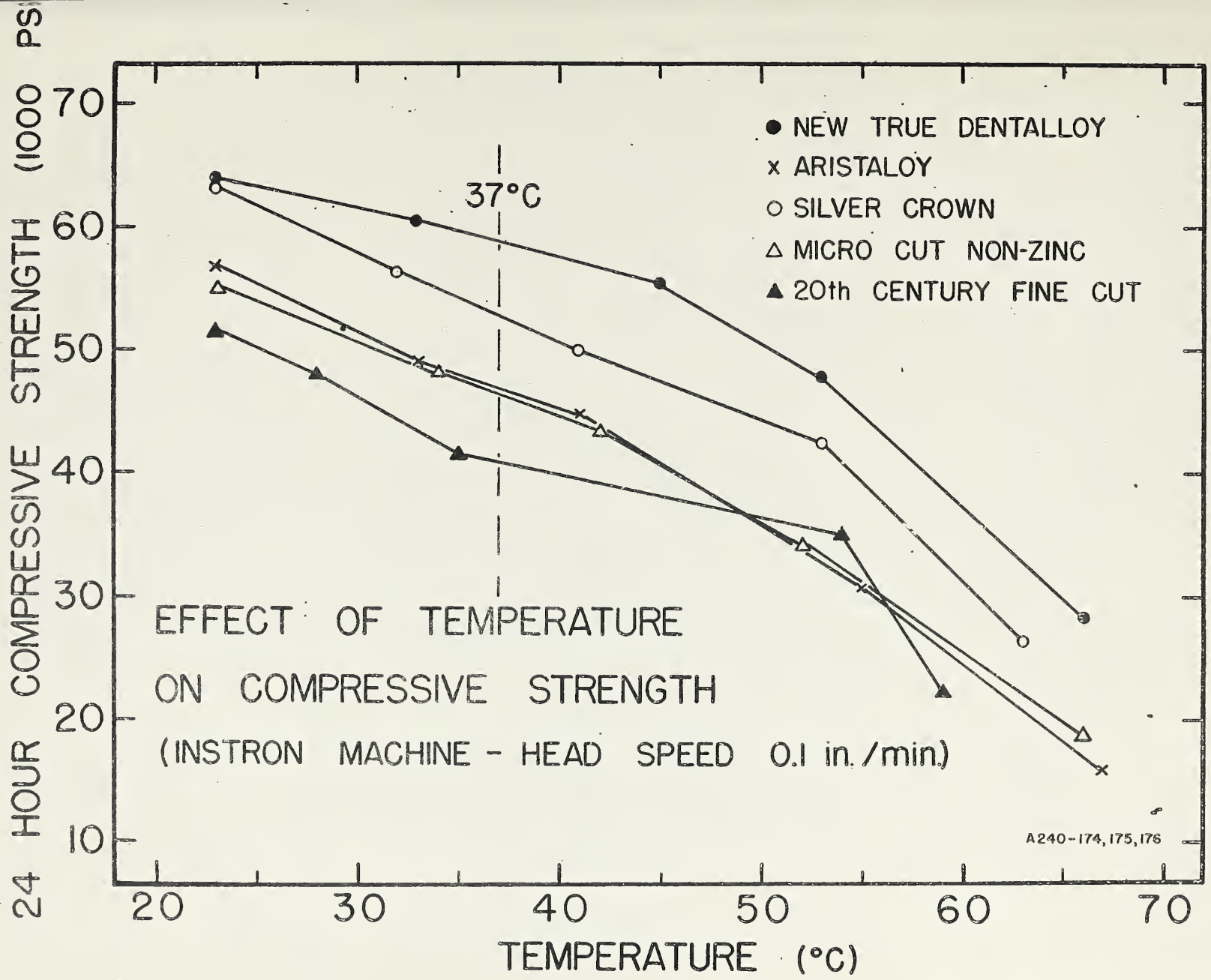


Figure 11. The effect of temperature on the compressive strength of amalgam.

U. S. DEPARTMENT OF COMMERCE
Luther H. Hodges, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

# THE NATIONAL BUREAU OF STANDARDS

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**Heat.** Temperature Physics. Heat Measurements. Cryogenic Physics. Equation of State. Statistical Physics.

**Radiation Physics.** X-ray. Radioactivity. Radiation Theory. High Energy Radiation. Radiological Equipment. Nucleonic Instrumentation. Neutron Physics.

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**Mechanics.** Sound. Pressure and Vacuum. Fluid Mechanics. Engineering Mechanics. Rheology. Combustion Controls.

**Polymers.** Macromolecules: Synthesis and Structure. Polymer Chemistry. Polymer Physics. Polymer Characterization. Polymer Evaluation and Testing. Applied Polymer Standards and Research. Dental Research.

**Metallurgy.** Engineering Metallurgy. Microscopy and Diffraction. Metal Reactions. Metal Physics. Electrolysis and Metal Deposition.

**Inorganic Solids.** Engineering Ceramics. Glass. Solid State Chemistry. Crystal Growth. Physical Properties. Crystallography.

**Building Research.** Structural Engineering. Fire Research. Mechanical Systems. Organic Building Materials. Codes and Safety Standards. Heat Transfer. Inorganic Building Materials. Metallic Building Materials.

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**Data Processing Systems.** Components and Techniques. Computer Technology. Measurements Automation. Engineering Applications. Systems Analysis.

**Atomic Physics.** Spectroscopy. Infrared Spectroscopy. Far Ultraviolet Physics. Solid State Physics. Electron Physics. Atomic Physics. Plasma Spectroscopy.

**Instrumentation.** Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

**Physical Chemistry.** Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Elementary Processes. Mass Spectrometry. Photochemistry and Radiation Chemistry.

**Office of Weights and Measures.**

## BOULDER, COLO.

**Cryogenic Engineering Laboratory.** Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

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**Ionosphere Research and Propagation.** Low Frequency and Very Low Frequency Research. Ionosphere Research. Prediction Services. Sun-Earth Relationships. Field Engineering. Radio Warning Services. Vertical Soundings Research.

**Radio Propagation Engineering.** Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

**Radio Systems.** Applied Electromagnetic Theory. High Frequency and Very High Frequency Research. Frequency Utilization. Modulation Research. Antenna Research. Radiodetermination.

**Upper Atmosphere and Space Physics.** Upper Atmosphere and Plasma Physics. High Latitude Ionosphere Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.

### RADIO STANDARDS LABORATORY

**Radio Physics.** Radio Broadcast Service. Radio and Microwave Materials. Atomic Frequency and Time-Interval Standards. Radio Plasma. Millimeter-Wave Research.

**Circuit Standards.** High Frequency Electrical Standards. High Frequency Calibration Services. High Frequency Impedance Standards. Microwave Calibration Services. Microwave Circuit Standards. Low Frequency Calibration Services.


NBS