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# Progress Report
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# Application of Diffraction Gratings to Measurement of Strain of Dental Materials

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U.S. DEPARTMENT OF COMMERCE
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# Application of Diffraction Gratings to Measurement of Strain of Dental Materials


## Abstract

A system for measurement of strain by means of diffraction gratings ruled on small specimens has been applied to dental materials. The method gives a rapid response to stress, is effective over very short gage lengths, and does not require that a measuring device be attached to the specimen. Gratings with a spacing of approximately 16,000 lines per inch are ruled on polished surfaces on the specimen. When the specimen is stressed, this spacing is changed resulting in a change in the angle of diffraction of a beam of light directed onto the grating. The change in angle of diffraction is measured by means of photomultiplier tubes behind "V" slits, and from the change in angle, the magnitude of the strain is calculated.


## 1. Introduction

In determining the properties of dental materials it is desirable but often difficult to make measurements on specimens comparable in size to the dental restorations made from these materials. The measurement of stress-strain relationships presents a problem since the dimensions of many commonly used strain gages are not adaptable to small specimens. Recent developments in resistance strain gages have made possible measurements over relatively short gage lengths. As an alternative method of measuring strain which might permit the use of even shorter gage lengths, it was decided to investigate the feasibility of diffraction gratings ruled directly on the specimen.

If a number of closely spaced, equidistant parallel lines are ruled on a specimen, they will act as a diffraction grating. Light striking the grating will be diffracted through an angle that is dependent on the distance between adjacent lines. Thus, when the specimen is strained the change in the angle of diffraction can be used as an indication of the magnitude of the strain.

The diffraction gage method offers several advantages. Depending upon the quality of the grating that can be ruled on the specimen, the gage length can be quite small, of the order of a few tenths of a millimeter. Attachment of any strain measuring device to the specimen during testing is eliminated. If a photoelectric method is used for measurement of the change in angle of diffraction, rapid changes in strain can be recorded with no difficulty.

Probably because of the difficulty of ruling gratings, little use has been made of diffraction gratings for measuring strain. Dr. James Bell and associates at Johns Hopkins University have, however, developed the method very completely in the course of their investigations of dynamic plastic strain [1,2]. The specimens used in the work of Bell et al were rods approximately one inch in diameter and from four to seven inches long. A diffraction grating was cut similar to a screw thread about the circumference of the specimen by use of an extremely accurate lathe [3]. These specimens were then dynamically stressed by impacting them with a known force and the resulting strains observed as they passed along the rod. Bell reports that he has been able to measure strains from a few micro-inches per inch up to 10% with an accuracy of better than 5% using gage lengths ranging from 0.001 of an inch to 0.030 inch [4,5].

Filbey [6] has described a method for using photomultiplier tubes for measuring rapidly the change in angle of light diffracted from a strained grating The success of these investigators prompted the application of the diffraction grating strain gage to dental materials.

## 2. Method

### 2.1 General Description

The method employed involved (1) ruling a grating on the specimen; (2) load ng the specimen in compression; (3) directing a beam of monochromatic light perpendicularly onto the grating on the specimen, and (4) utilizing photomultiplier tubes to measure the changes in angle of diffraction as indicated by changes in signal as the diffracted beams moved along V slits in front of the tubes.

### 2.2 Determination of Strain

The system used for determining strain is shown schematically in Figure 1. Strain is calculated from measurement of the distance, h, which the diffracted image of the source moves on the surface of the V slit on the face of the photomultiplier tube. The relationship between h and the strain, $\epsilon$, is derived as follows:

For light normally incident on a grating

$$d = \frac{n\lambda}{\sin\theta}$$

where

d = distance between adjacent grating lines
$\theta$ = angle of diffraction
n = order of diffraction
$\lambda$ = wave length of incident light

When the specimen is strained

$$d' = \frac{n\lambda}{\sin(\theta + \Delta\theta)}$$

$$\epsilon = \frac{d' - d}{d} = \frac{\frac{n\lambda}{\sin(\theta+\Delta\theta)} - \frac{n\lambda}{\sin\theta}}{\frac{n\lambda}{\sin\theta}}$$

$$\epsilon = \frac{\sin\theta - \sin(\theta+\Delta\theta)}{\sin(\theta+\Delta\theta)}$$

$$\epsilon = \frac{\sin\theta - \sin\theta\cos\Delta\theta - \cos\theta\sin\Delta\theta}{\sin\theta\cos\Delta\theta + \cos\theta\sin\Delta\theta}$$

Since $\Delta\theta$ is small, assume:

$$\cos\Delta\theta = 1$$

$$\sin\Delta\theta = \frac{h}{R}$$

where R is the distance from the grating to the face of the photomultiplier tube (when condensing lens are introduced as in Figure 6, R becomes the distance from lens to tube.)

$$\epsilon = \frac{-\frac{h}{R}\cos\theta}{\sin\theta + \frac{h}{R}\cos\theta}$$

$$\epsilon = -\frac{h}{R\tan\theta + h}$$

## 2.3 Material Used

The materials used in this investigation were aluminum (2014 - T4 alloy), yellow brass (Federal Spec. QQ - B - 626a), gold alloy (Gold alloy medium, Baker and Co.), and a dental amalgam alloy (Speyer alloy, extra fine). The aluminum and brass were used because they were easy to polish and presented a good surface on which to develop the technique of producing a grating. In addition, they provided a check on the experimental values obtained since values for the modulus of elasticity of both are available [7]. The gold alloy and the dental amalgam were selected to demonstrate that the technique could be used on the metallic materials commonly associated with dentistry.

## 2.4 Preparation of Ruling Blocks

Due to the configuration of our ruling engine it was convenient to prepare gratings on rectilinear blocks of material approximately 8 mm by 4 mm by 40 mm. The surfaces of the aluminum, yellow brass and gold ruling blocks were prepared using standard metallographic polishing procedures. Absolutely scratch free surfaces were not necessary and surfaces sufficient for a diffraction grating were readily produced.

The preparation of a ruling block of dental amalgam alloy presented a more difficult problem. The standard mixing and packing procedures for dental amalgam alloys prohibited the construction of one large block. By changing these procedures perhaps a large block similar to those of the aluminum could have been made, but this would have left doubt about the similarity of the material to that used clinically. It was desirable when ruling a grating to have the cutting instrument pass over a smooth surface and not pass from one material to another. This meant that an amalgam surface should be available throughout the entire length of the ruled line. Actually, the specimens to be used for the test procedure were small rectangular units cut from the ruling blocks after the grating had been produced. To develop the ruling blocks of dental amalgam alloy an acrylic mold was made into which the alloy could be packed. The acrylic block (Fig. 2) had six small rectangular depressions arranged side by side in the center of the mold. The entire center area (running lengthwise) was depressed approximately one millimeter. This permitted the packing of one small rectangular block at a time and by simply bridging across the depressed surface of the dividing acrylic the blocks were connected. This thin layer of amalgam formed the bridge between the small blocks thus keeping the continuity of surface needed for ruling purposes. After the amalgam alloy had hardened, the acrylic sides were cut from the mold block leaving only the alloy surface to be polished.

Dental amalgam alloy being a nonhomogeneous substance produced problems both in polishing and ruling a grating. The polishing procedure finally adopted used aluminum oxide abrasive in succeedingly finer sizes from five microns to one-tenth micron in conjunction with slow running cloth covered polishing wheels under a constant flow of water. A final light buffing with magnesium oxide abrasive and water produced the desired surface. Overpolishing at any stage produced a surface so irregular that it was necessary to return to the fine metallographic paper and start the final polish all over again.

## 2.5 Production of Gratings

Because quality grating production was in itself, such a large problem, no attempt was made to produce error free units. Diffraction gratings that provided a well defined green line proved to be sufficient for the purpose of measuring strain. It was anticipated that the aluminum and brass would rule best so initial efforts were with these materials. Amalgam being a multiphase material was expected to be a difficult, if not an impossible surface on which to develop a grating and therefore was not attempted until after some experience had been gained on other materials.

The ruling engine, Figure 3, used to produce the diffraction gratings was built at the National Bureau of Standards in the early 1920's. Although not used for many years it was found to be in good condition and with minor repairs was made operable.

To rule a grating, the specimen on a platform is moved continuously along ways by an extremely accurate screw. The grating lines are ruled by a diamond stylus (Fig. 4) which is drawn across the specimen and then lifted from the surface and returned to its original position to be again drawn across the specimen for the next line. The spacing of the ruled lines is controlled by gears which relate the frequency of ruling to the rate of rotation of the screw which moves the ruling block along the ways. For this study the line spacing was 16230 lines per inch (a line to line space of 1.56 μ). For most specimens a total of 320 lines were ruled at a rate of approximately 6 lines per minute.

One of the most critical factors affecting the quality of the ruled grating is the positioning of the diamond stylus. The cutting edge must be in line with the direction of travel of the stylus. Additionally the cutting edge should be tilted slightly (at an angle of about three degrees) so that the diamond is slicing rather than plowing. The load on the stylus should be adjusted to obtain optimum results with each material ruled. Loads of 1.5 to 2 grams produced the best results with the materials ruled in this work. Use of a libricant on the surface to be ruled is common practice. Penetrating oil (Fed. Sac VV-P-216) was found to be satisfactory for this purpose.

To prevent irregularities in the gratings due to vibration during the ruling process, the table on which the engine was mounted was floated on air cushions. Also the drive system consisting of an electric motor with gear reduction box and pulleys and belt to the ruling engine were carefully selected and adjusted to produce minimum vibration. After a grating was cut on a ruling block, the block was sectioned into small specimens about 8 mm by 4 mm by 4 mm (Fig.5). Each specimen was placed in the light field and the pattern obtained from it observed. The specimens showing the best patterns were retained for use. Figure 6 shows a typical pattern as diffracted from an aluminum specimen onto a ground glass plate. The photograph clearly illustrates a low intensity, continuous spectrum background in the light of the mercury vapor lamp. The brightest lines seen are the most intense wavelengths emitted from this lamp and represent from left to right of the photograph the following colors and wavelengths: yellow (5770 A°), green (5461 A°), blue (4358 A°) and violet (4047 A°). In actual use very little of the spectrum was visible except for the main lines noted above. Filtering the light with a Kodak 77 A/58 glass filter eliminated nearly all of the light except the green line as seen in Figure 7.

As expected, better gratings were obtained on aluminum, brass, and gold than on dental amalgam alloy. The green line reflected from the latter was not as clearly defined as was the case with the other materials. All of the materials did exhibit a spectrum to a degree sufficient for measurement purposes.

## 2.6 Equipment

The optical part of the measuring equipment developed for use with the diffraction gratings is shown in Figure 8. A one hundred watt mercury vapor lamp (General Electric H 100 A4/T) was selected as the light source. The main disadvantage of the lamp was that its alternating current power supply produced a 120 cps variation in intensity of the light. The lamp was placed in a horizontal position in a closed cannister light shield through which air was forced for cooling purposes. On one side of the cannister was an adjustable slit which in effect acted as the source of light for the system. This light beam was collimated by an adjustable lens located

approximately 50 mm from the slit. The collimated light beam was directed onto the surface of the specimen so as to form a right angle. The determination that the light was normal was done very simply by observing where the direct reflection from the specimen surface struck the lens. When this direct reflection coincided with the light passing to the specimen the alignment was satisfactory. A filter (previously described) was placed between the lens and the specimen. This filter satisfactorily eliminated all light other than the 5461 A° green line.

Since the incident light was perpendicular to the grating surface the two first order images were symmetrically arranged at an angle on either side of the grating normal. For the 5461 A° mercury line and gratings with 16,230 lines per inch, $\theta_o$ was 24° 25′. A second order was seen at 44° 15′ on either side of the grating normal on those specimens having good quality rulings. The second order might also have been used for measurement purposes. It had the advantage of exhibiting more displacement under the same load but conversely it was a great deal less intense than the first order. Only the gratings on the aluminum and brass specimens were of sufficient quality to show a second order.

Spherical lenses were placed in the path of each first order beam to focus the light on the surface of the slit on the face of the photomultiplier tube. These lenses were placed at a distance equal to their focal length (one meter) in front of the measuring tubes.

Three photomultiplier tubes were used in the strain gage system. Two RCA 6199 tubes were used for measurement of light movement. They were end on tubes that exhibited a uniform response over the central portion of the grid. "V" slits were placed in front of the uniform area (Fig. 9). The quantity of light impinging on the phototube then depended on the location of the light on the "V" and changes in position of the light were seen as changes in signal response. Since these two tubes responded to any change in light intensity as if it were a change in light location, a third tube, an RCA 3819, was placed in the system to act as a monitor. A glass plate was used as a beam splitter to reflect part of the first order diffracted beam to the end surface of the monitor tube. This tube had a rectangular opening over the grid and thus was not sensitive to image movement and responded only to changes in overall light intensity. Changes such as this occurred due to variance within the light source, from movement of the grating to a slightly brighter or dimmer area of the light field or from deterioration of the grating during stress.

The electrical equipment used in conjunction with the measuring device consisted of the photomultiplier tubes, an amplifier, voltage supplies and a recording oscillograph. Each photomultiplier tube operated on one thousand volts direct current (supplied by a Regulated High Voltage Supply, Model No. 1603, Northeast Scientific Corporation). The voltage was supplied to each of the three phototubes via a junction box which also contained the amplifier. Amplification of the signal could be adjusted individually for each tube by the rheostats located on the side of the box. The individual adjustments proved to be a great help in balancing the tube responses. The amplifying system required additional power supplies of a positive and a negative one hundred fifty volts direct current. (These requirements were met by two Regulated Power Supplies Model No. 29, Lambda Electronics Corporation.)

The signal from each phototube was plotted automatically by a recording oscillograph (Honeywell Visicorder Model 906 C) which is a multichannel continuous recorder employing fluid damped galvanometers to provide rapid response to signal change. The actual recording was made on light sensitive paper by high intensity minute points of light reflected from mirrors attached to the galvanometers. Three galvanometers were used to record continuously the signals from the three photomultiplier tubes.

The loading of the specimens was done by means of an Instron Testing Machine utilizing compression cell CF with a range of 0 to 10,000 lbs. The head speed was 0.005 inch per minute. The time from initial to maximum load was about 3.5 seconds. The equipment is illustrated in Figure 10.

## 2.7 Test Procedure

Following the removal of the specimen from the ruling block the ends of the specimen were machined parallel and at right angles to the sides. This was done to minimize the possibility of tilting as well as to assure the application of the load uniformly. The bright areas of the specimen to either side of the grating were covered with a nonreflecting black paint. The specimen was centered beneath the advancing platen of the testing machine and a small load applied to gain stability. The light was adjusted so that it was incident at right angles to the specimen and fully covered the grating.

The images were then noted on the surface of the photomultiplier tubes. Each tube was set so that the image beam struck the grid at a right angle. After rectangular apertures were placed on each tube, a test load was applied. This permitted the observance of the intensity variations during the application of the stress. The light was adjusted so that these variations were brought as close to zero as possible. When the intensity was uniform the "V" slits were placed on the two measuring tubes. These triangular apertures were oriented so the apex of each triangle pointed toward the center of the apparatus. With such an arrangement an increase in the angle of diffraction was shown as an increase in signal from both tubes. Tilting of the specimen on the other hand appeared as an increase on one tube and a decrease on the other. During test runs a combination of these two effects occurred. The influence of tilting on the measurements was eliminated by averaging the movement measured on the tubes.

Calibration was necessary in order to relate signal change observed on the recorder to the distance the light moved on the face of the photomultiplier tube. For this purpose each tube was moved through a known distance perpendicular to the diffracted beam and the change in signal recorded on the oscillograph tracing. The distance equivalent of signals obtained later when the specimen was strained was then calculated by comparison with the calibration tracings.

## 3. Results

The tests were carried out first on aluminum and brass to permit the accuracy of the apparatus to be checked against established values. Gold and dental amalgam specimens then were tested. A summary of the results obtained is given in Table 1.

Table 1

Comparison of Values of the Modulus of Elasticity

(Million PSI)

| Specimen | Source | | |
|---|---|---|---|
| | Diffraction Grating | Tuckerman System | Metals Handbook |
| Al | 9.1 ± 1.0* | 9.9 | 10.6 |
| Brass | 15.8 ± 1.3 | 16.8 | 15.0 |
| Au | 15.5 ± 1.0 | .. | 15.0 |
| Amalgam | 8.0 ± 0.3 | .. | .. |

* Standard Deviation

The summary table shows that all of the reference values except the Handbook value for the aluminum fell within one standard deviation of the experimentally determined modulus.

Figure 11 illustrates a stress-strain diagram for amalgam obtained from three runs on one specimen. The plot of the points obtained indicates a straight line or elastic relationship between stress and strain at this loading rate and within the range of 6,000 to 16,000 psi. The modulus of elasticity determined from the slope is $7.7 \times 10^6$ psi.

## 4. Discussion

The determinations on aluminum, brass and wrought gold alloy indicate that the apparatus developed is capable of determining strain to within approximately 10% or better over the ranges measured. The modulus of elasticity of dental amamgam of $8 \times 10^6$ is significantly higher than previously reported. Analysis of the previous [8,9,10,11] work indicates that the strain determinations made heretofore have been a combination of the elastic, the retarded elastic and the viscous deformation of the material. Since both the retarded elastic and viscous deformations are time dependent, the instantaneous elastic characteristics only are approached when rapid loading and recording techniques are used. The high value obtained for the modulus of elasticity of dental amalgam with this technique which required only 3.5 seconds for attainment of maximum load, indicated that flow of the material had been markedly reduced. This is further verified by the straight line relationship between stress and strain shown in Figure 11.

The system described in this paper has several sources of error that limit the accuracy of the measurements. The largest error is probably introduced when the oscillograph chart is interpreted. It is difficult to read the chart accurately to 0.25 division. This problem arises in both calibration and measurement runs and depending on the amount of deflection involved may vary from 1 to 5%. During calibration the measuring phototubes are moved through 0.125 inch. This movement could easily vary ± 0.001 inch thus producing another possible 1% error. The load determination as taken from the Instron also may vary on the order of 1%. Therefore, for a given test run the total experimental error may be expected to approximate 5 to 10%. However, by refinement of the apparatus it should be possible to reduce these errors. One shortcoming of the present equipment lies in the fact that strain is measured only on one side of the specimen while stress is determined on the basis of load applied to the entire specimen. If due to uneven contact between the specimen and the testing machine platens or for other reasons the specimen is not uniformly stressed an error will be introduced. This effect may account for some of the variations in results shown in Table 1.

## 5. Conclusions

A method that permits the measurement of rapidly changing strain on small specimens by means of a diffraction grating strain gage was developed. The system built measured strain to within 10% over a gage length of 0.5 mm. Tests conducted to determine the modulus of elasticity of dental amalgam alloy yielded a value of $8 \times 10^6$ psi. This figure indicates the modulus of elasticity of dental amalgam alloy to be significantly higher than previously reported. The diffraction grating strain gage is unique in that it provides a method for the quantitative determination of the elastic properties of some dental materials when the specimens approximate the size used in restorative dentistry.

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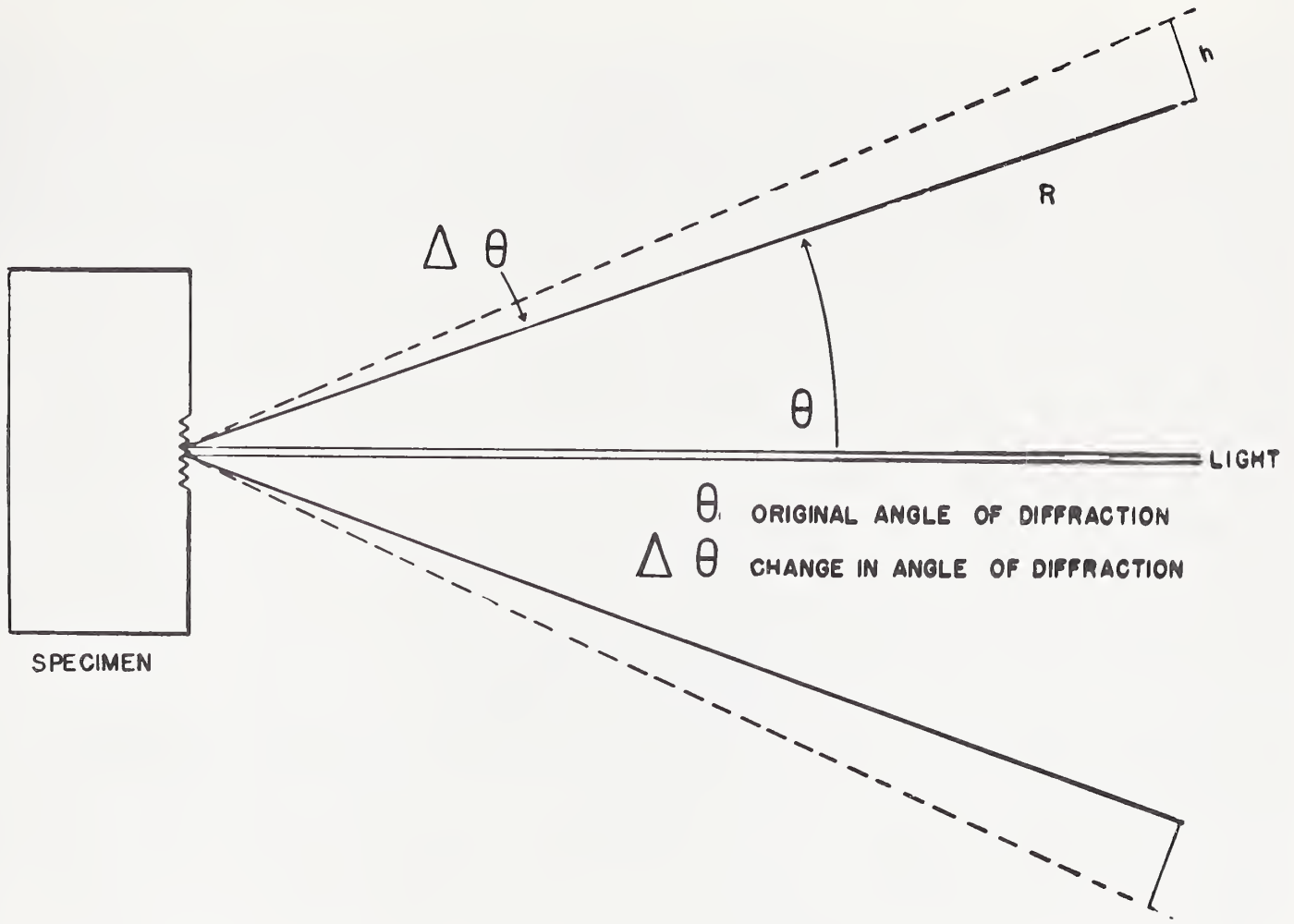


Figure 1. Change in angle of diffraction when specimen is strained by application of compressive stress.

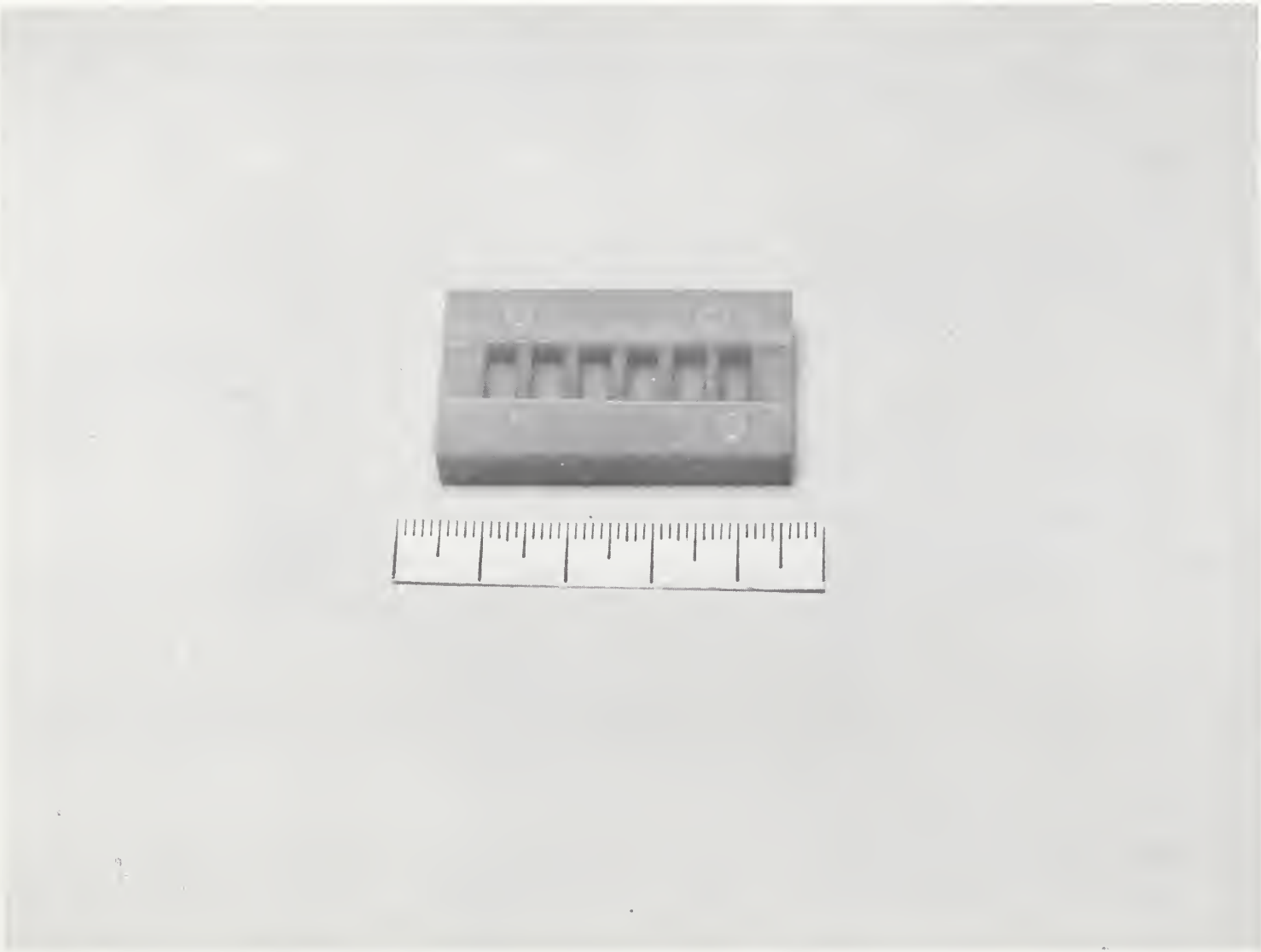

Figure 2. Acrylic mold used in preparation of dental amalgam specimens (scale in mm).

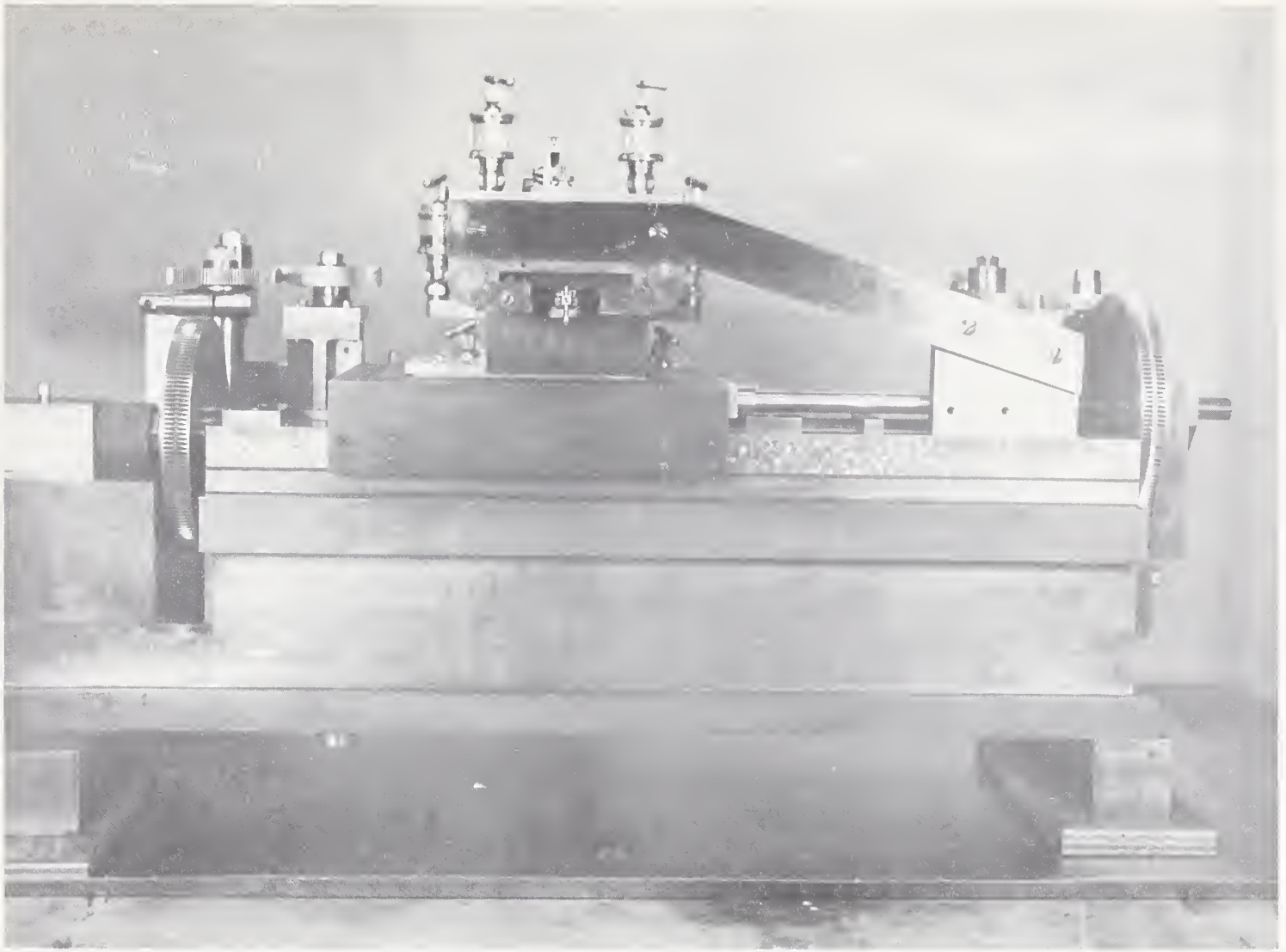

Figure 3. Ruling engine used to rule gratings on specimens.

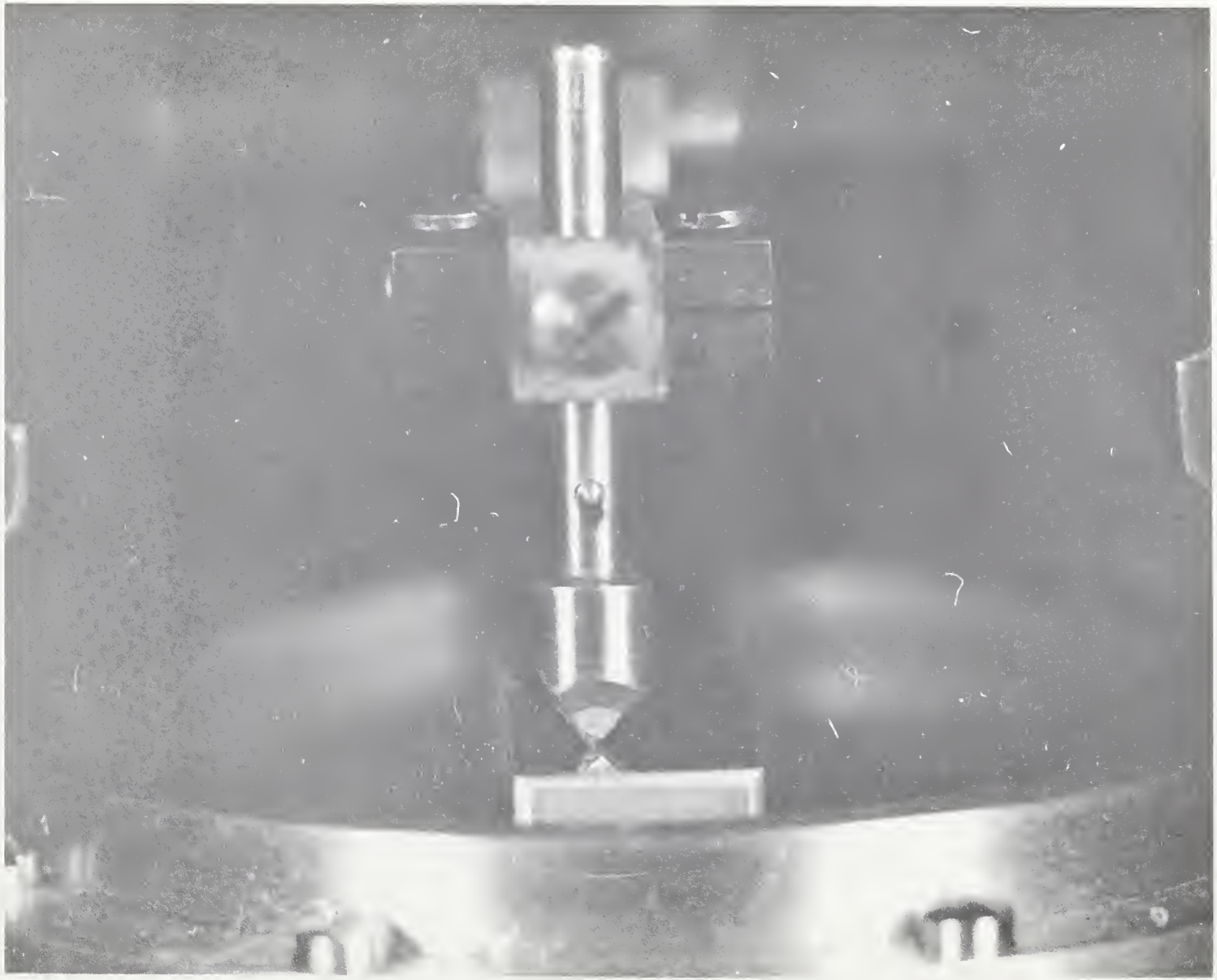

Figure 4. Diamond stylus of ruling engine in contact with aluminum specimen block.

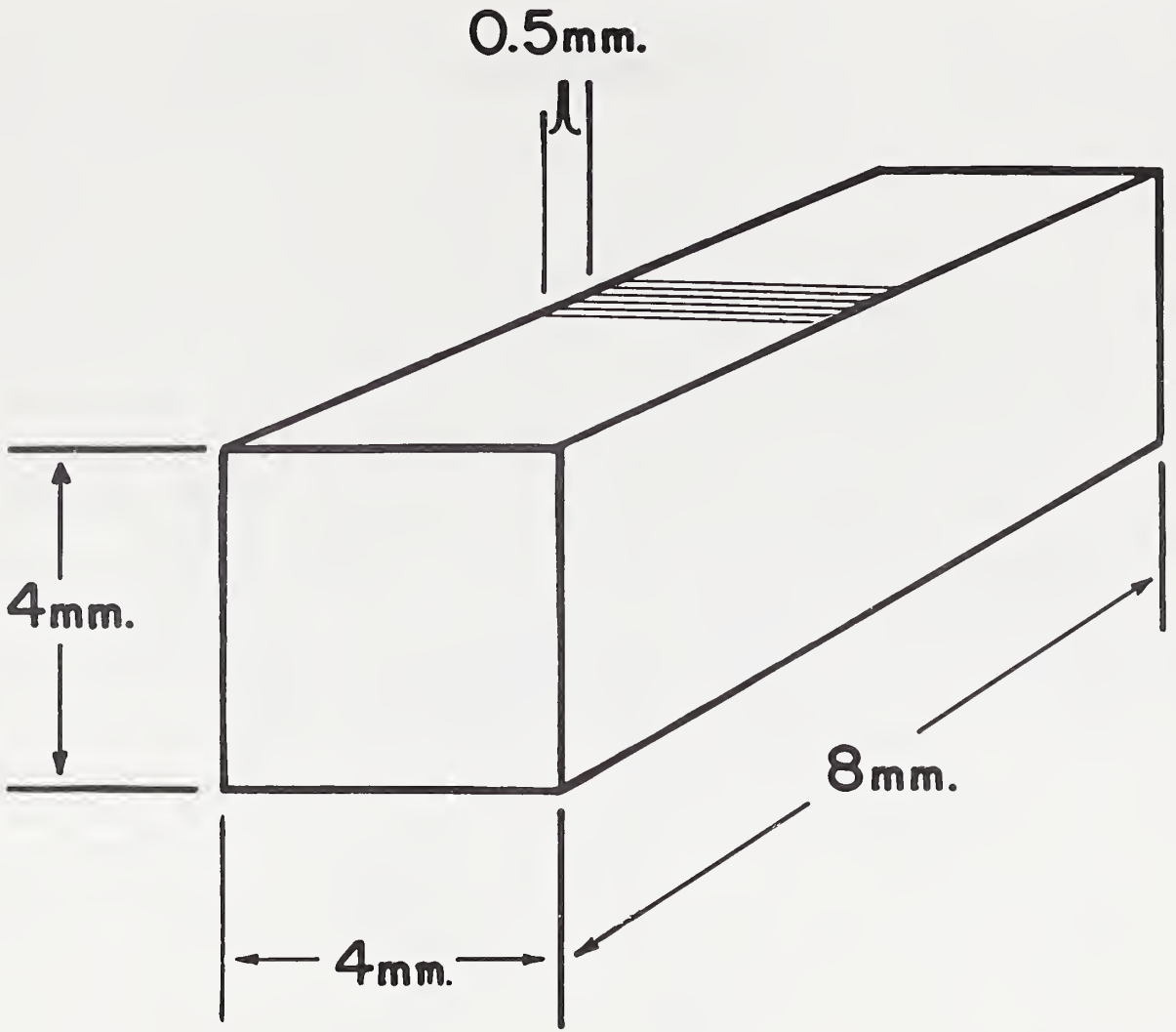


Figure 5. Drawing of typical compressive specimen.

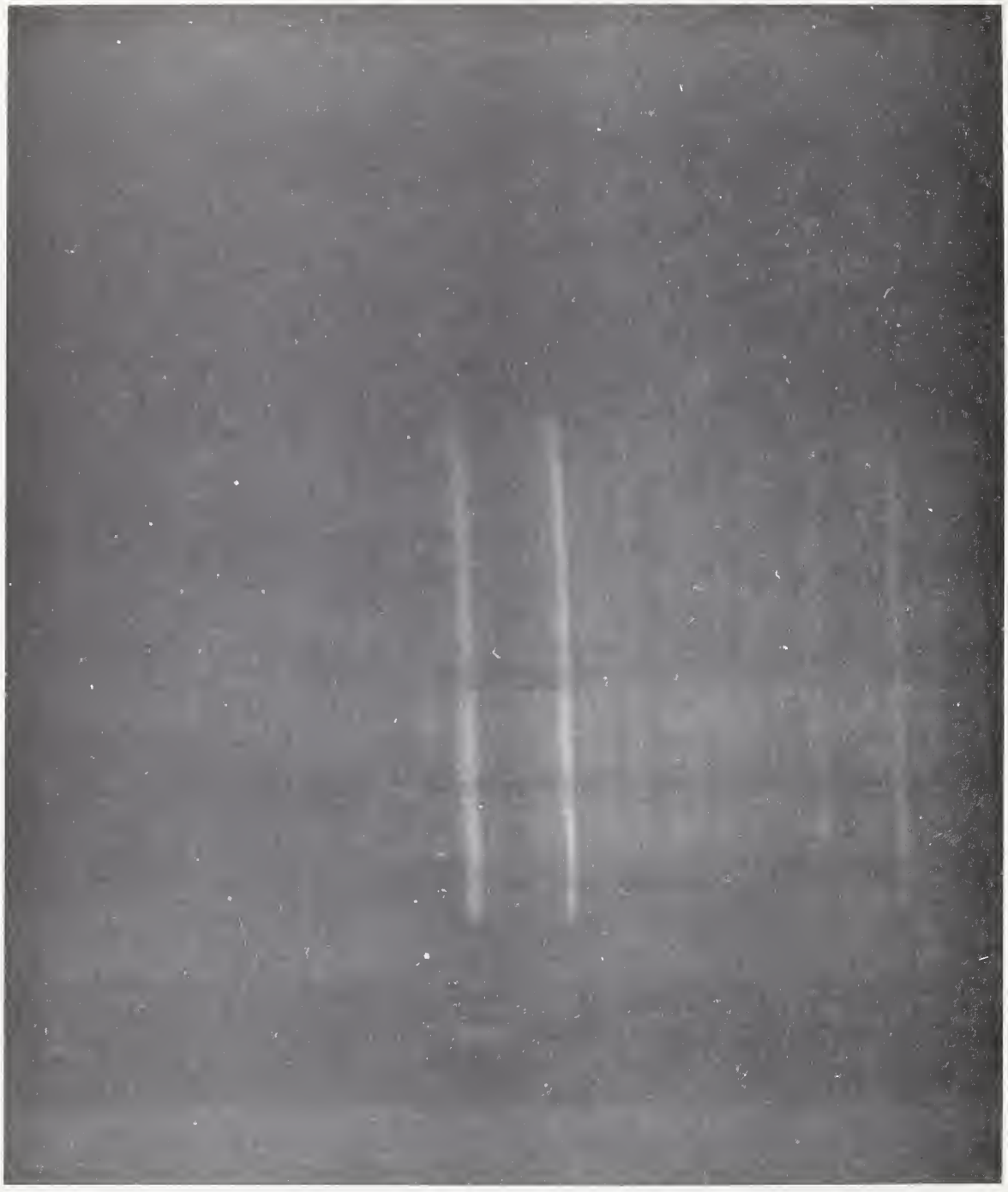

Figure 6. Diffraction spectrum as seen on ground glass plate. The most intense lines are from left to right: yellow 5770 Å, green 5461 Å, blue 4358 Å, and violet 4047 Å.

Figure 7. Spectrum as seen with filter in place to remove light other than the 5461 Å green line.

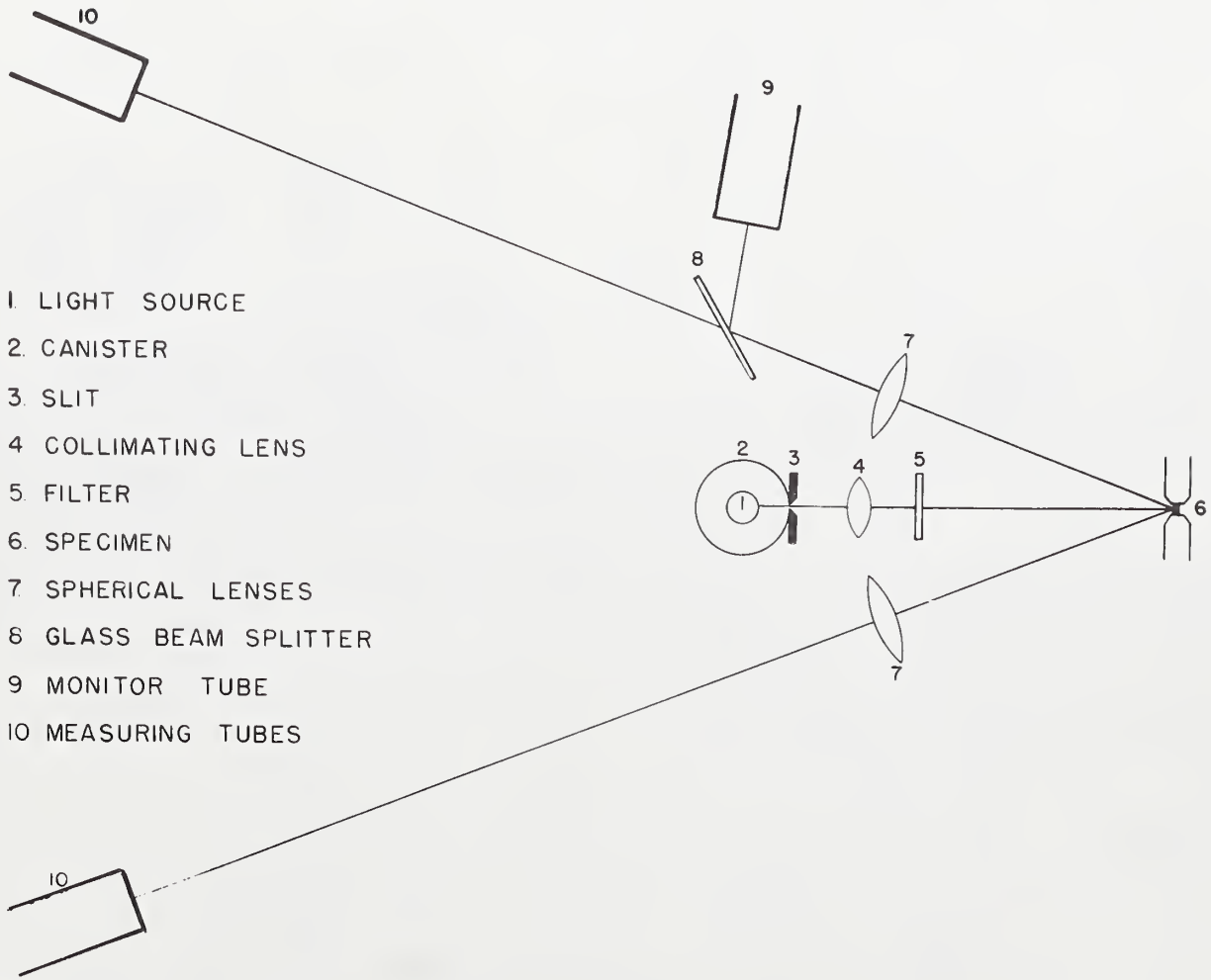


Figure 8. Schematic arrangement of measuring device.

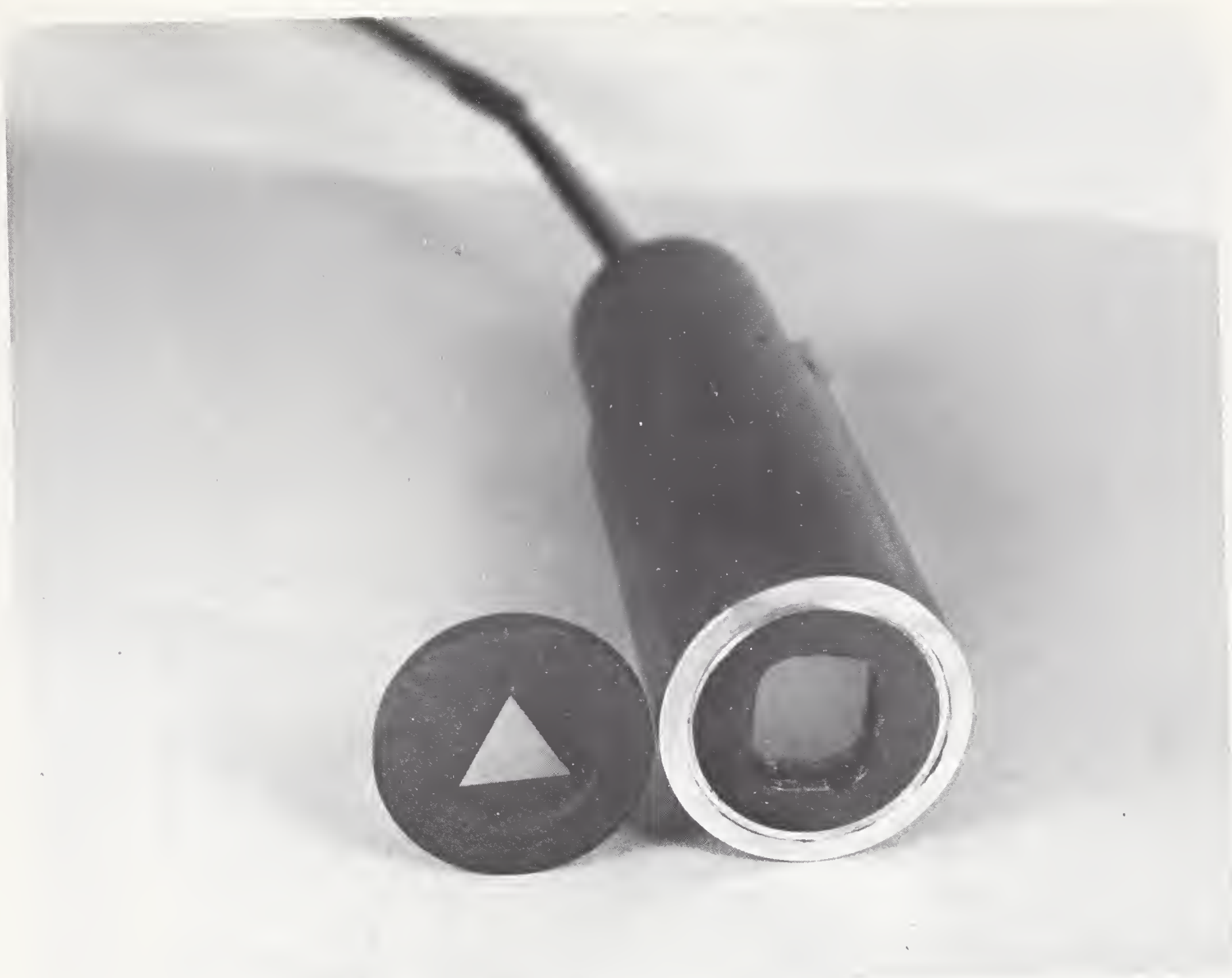

Figure 9. Photomultiplier tube with "V" slit removed.

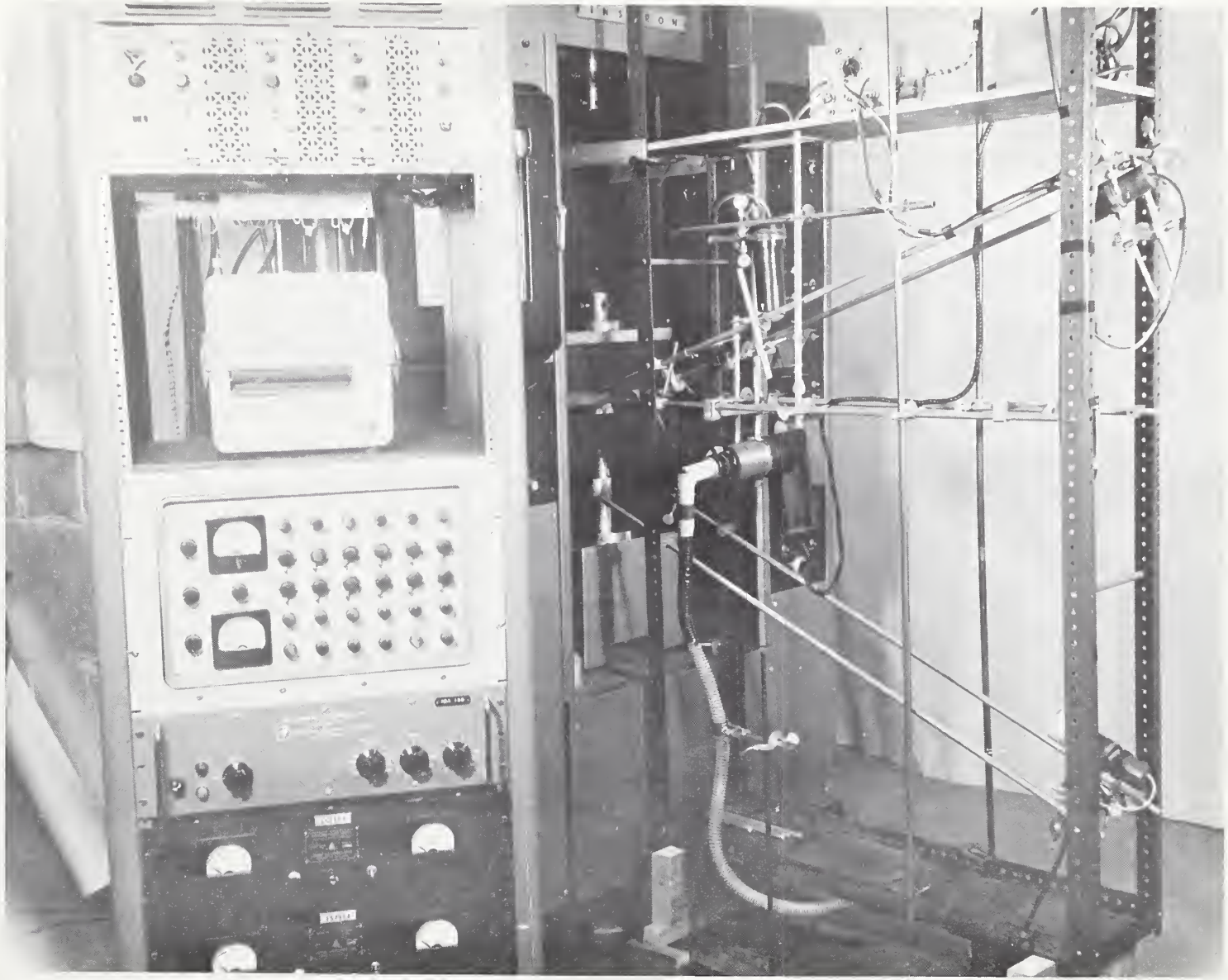

Figure 10. Equipment used for measuring strain by diffraction grating method.

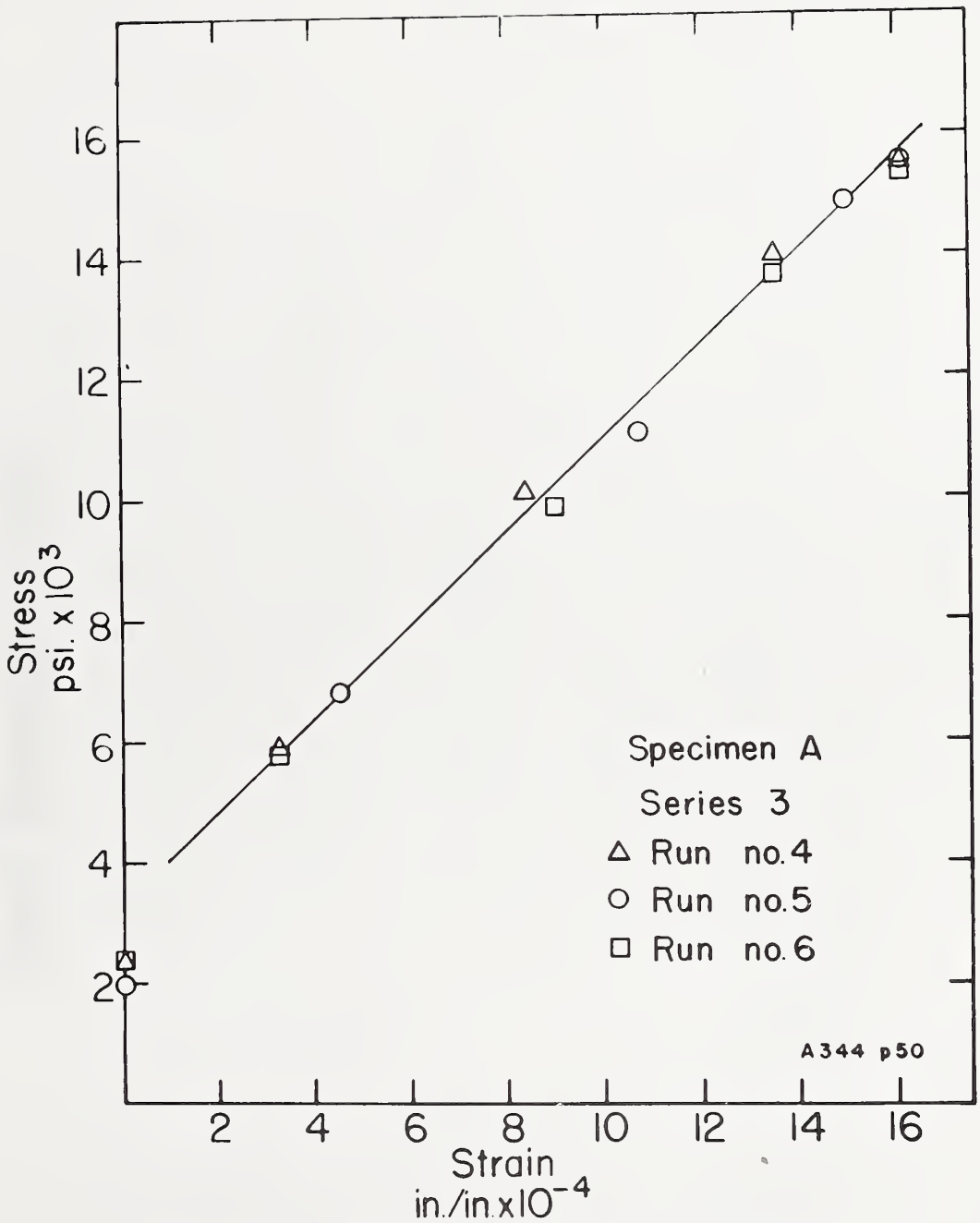


Figure 11. Stress-strain diagram for dental amalgam alloy.