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Progress Report

SOME TENSILE PROPERTIES OF TOOTH STRUCTURE AND SEVERAL RESTORATIVE MATERIALS

by

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U. S. DEPARTMENT OF COMMERCE
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SOME TENSILE PROPERTIES OF TOOTH STRUCTURE AND SEVERAL RESTORATIVE MATERIALS[1]

Abstract

Values for strength and modulus of elasticity were obtained in tension for various dental materials. The specimens were stored in distilled water at 37°C. for seven days. The specimens were loaded at 0.02 inches per minute head speed. Dentin and enamel specimens were prepared by cutting with rotating diamond tools using continuous water spray. They were formed into rectangular cross-section rods with a narrowed middle isthmus portion and notched ends. The middle portion was wound with a strip of heavy tinfoil until it fitted snugly into the stainless steel mold. The notched ends were imbedded in direct filling resin. After the resin had hardened, the flash was trimmed and the foil strip unwound. The materials had the following average tensile strength in pounds per square inch: enamel, 1,500; dentin, 7,500; silicate cements, 700; zinc phosphate cements, 550; direct filling resins, 4,200 and an experimental direct filling material containing about 70 percent treated fused silica, about 5,000, depending on surface treatment of the silica powder. The materials had the following average moduli of elasticity when measured in tension (times 10^6 PSI):

[1] This investigation was supported in part by research grant D-589, Synthesis of a Silica-Resin Direct Filling Material, to the American Dental Association from the National Institute for Dental Research.

dentin, 2.8; a silicate cement, 3.1; direct filling resins, 0.27 and an experimental direct anterior filling material containing about 70 percent vinyl silane treated fused silica powder, 1.6.

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1. INTRODUCTION

Tensile strength and stiffness are two important properties determining the usefulness of structural materials where mechanical forces act upon them and where rupture or excessive deformation cause loss of function. This is certainly the case with dental structures and materials. A growing number of research workers believe that dental materials break often in tension [1]. To our knowledge, however, the tensile strengths of dentin, enamel and the silicate and zinc phosphate cements have not previously been reported.

The present studies were made to fill this gap and to pave the way for studies of adhesion to hard tooth tissues. This is desirable since the load an adhesion joint will support is limited by the strength of the materials as well as by the interface bonding.

Also, measurement of the tensile strength and stiffness (modulus of elasticity) of the experimental silica-resin material is part of a broader project of evaluating this anterior direct filling material.

2. EXPERIMENTAL PROCEDURE

2.1 Molds for Preparing Specimens

Split molds were made for preparing the specimens [2]. Most of the specimens were made in two stainless steel molds such as that shown in Figure 1. The middle narrow portion of the molds was 0.100 ± 0.002 inch wide by 0.125 ± 0.002 inch deep and 0.3 and 0.5 inch long to allow the attachment of Tuckerman optical strain gauges (0.25 inch gauge length) directly to the specimens. The round end portions of the molds were 0.200 inch in diameter. The angles connecting the large ends and the narrow middle portions were rounded. Thus, a top view of direct filling material specimens made in these molds showed a dumbbell shape, while the outlines of the side and end views were rectangular.

2.2 Preparing Specimens of the Commercial Materials

The commercial materials that were tested are listed in Table 1; the other materials are described below.

The certified silicate cements were each mixed at a standard consistency according to American Dental Association Specification No. 9 [3]. The other materials were mixed according to the directions of the manufacturers.

Each of the filling materials and cements was mixed within one to one and one-half minutes and was placed directly into the mold. A glass cover was pressed down tightly over the mold with finger pressure and held with a clamp. This assembly was placed,

within two or three minutes, into an atmosphere of 100 percent relative humidity and 98.6 ± 1.8°F. for 15 to 30 minutes for the material to harden. The specimen was then removed from the mold and placed in distilled water at 98.6 ± 1.8°F. for seven days before it was tested.

To measure the modulus of elasticity of silicate cement, larger specimens were needed. Due to the brittleness and low tensile strength of silicate cement, the small specimens in the steel grips broke during attempts to attach the optical strain gauges. The larger specimens were the same general shape as the smaller ones but were 3/16 inch square in cross section and long enough to take a one-inch gauge length (Figure 1, F). The molds and grips were both made of the same poly (methyl methacrylate) plate (Figure 1, E and G).

2.3 Preparation of Experimental Silica-Resin Material and Specimens

The synthetic resins A152-105 and A201-85C were essentially the addition products of Bisphenol A [4] (bis [4-hydroxyphenyl] dimethylmethane) and glycidyl methacrylate (2,3-epoxypropyl methacrylate) [5] containing reactive diluents to reduce the viscosity. The reactive diluent in resin A152-105 was tetraethyleneglycol dimethacrylate (22 percent) and in A201-85C it was methyl methacrylate (15 percent). These sirupy liquids each contained the accelerator N,N-dimethyl-p-toluidene so that polymerization would occur at room temperature (in the presence of benzoyl peroxide).

To make the specimens, these sirups were mixed (within one and one-half minutes) with treated fused silica powder [6] containing benzoyl peroxide, and placed in the stainless steel molds as described above. The polymerization of the crosslinking resin sirup bound together the treated fused silica particles in a thermoset organic matrix. The time of setting (hardening time) of this material was between three and eight minutes when measured according to the American Dental Association Specification No. 9 for Dental Silicate Cement.

Special preliminary surface treatment of the silica powder, such as treatment with vinyl trichlorosilane, had previously been found to impart a water-repellent nature to the particles and increase their affinity for organic liquids. Specimens were made using powders with various surface treatments to see if this would affect the tensile strength of the highly filled resins. These specimens were also immersed in distilled water at 98.6 ± 1.8°F. for one week before testing.

2.4 Method of Preparing Tooth Specimens

Enamel, dentin and aluminum specimens were cut freehand with a 3/8 inch rotary diamond disk using a commercial water-turbine handpiece [7] with a free-running speed of about 55,000 revolutions per minute. A stream of water kept the specimens cool during the cutting, and they were kept moist at all times. The extracted teeth had not been exposed to chemical preservatives but had been stored in water under refrigeration until used.

The enamel, dentin or aluminum was first cut into a linear specimen which was rectangular in cross-section and as long as the tooth tissue or mold would allow. The ends were notched on each edge for retention in the self-curing resin that was to be cast around them (Figs. 2, 3 and 4). This was necessary to prevent them from slipping and pulling out of the plastic ends when under load. Even though the notches were staggered to maintain maximum cross-section, many specimens failed at these notches near the ends (when load was applied) and had to be rejected. At this point, and/or later, the specimen was narrowed in the middle portion using a 5/32 inch diameter cylindrical diamond instrument. Its axis of rotation was at right angles to the long axis of the specimen so that the minute scratches left by the diamonds were aligned with lines or direction of tensile loading. This gave the least possible stress concentrations due to notch-like effects across the specimens. This also gave 5/64 inch as minimum radius of curvature where the narrow portion joined the larger end portions, eliminating notches in these areas.

Next, a strip of 0.002 inch tin foil was cut slightly less wide than the narrow middle portion of specimen. This was wound tightly around the middle portion of the specimen until it fitted snuggly into the stainless steel mold or was flush with the top of the mold (Fig. 5). If necessary, foil was folded accordian-style and equal thickness added to the top and bottom (or to each side)

so that the specimen was aligned with, and held directly in the center of the mold.

Then, a thin mix of a direct filling resin was packed, with a slight pumping action, around each notched end of the specimen. A glass plate was pressed down over the top of the mold and was held with a clamp. This assembly was put into a chamber having 100 percent relative humidity and a temperature of 98.6 ± 1.8°F for 15 to 30 minutes for the resin to polymerize. The mold was then removed and opened, the flash removed from the resin casting with a low speed fissure bur and the foil strip was unwound from the specimen.

2.5 Measurement of Cross-sectional Area of Specimens

The narrow isthmus portion of each specimen was measured with a micrometer caliper accurate to 0.0004 inch and/or a measuring microscope accurate to 0.0001 inch. The tooth and aluminum specimens were about 0.03 by 0.05 inch across the narrow middle portions, giving cross-sectional areas on the order of 0.0015 square inch. The other larger specimens were approximately the size of the molds used to form them; they were measured with the micrometer caliper just before being tested.

Since the reduction of cross-sectional area on the tooth specimen was smaller than could be measured on the measuring microscope, it did not matter whether it was measured before or after the specimen was broken. Therefore, the measurements across the broken ends

of the tooth-specimens, made with the measuring microscope, were used to calculate area because they could be made with greater precision.

The cross-sectional dimensions of the narrow isthmus portions of the aluminum specimens were measured with the microscope before they were tested in tension.

2.6 Testing Conditions

The specimens were kept moist at all times before and during testing. The tests were conducted at 73.4 ± 3.6°F. Specimens were allowed at least one hour in distilled water at this temperature before measurements were made.

Stainless steel grips were made which fitted the ends of the specimens; the grips were clamped to the testing machine (Fig. 6).

An Instron testing machine [8] was used for applying and measuring the load on the specimens. A tensile load cell was used having a range from one to 50 kilograms (2.2 to 110 pounds), for full scale deflection on the recording chart.

To measure strain in the specimens, for the evaluation of Young's modulus of elasticity (stiffness), the knife edges of an optical strain gauge [9] were placed directly on the specimen (Fig. 7). A one-inch gauge length was used on the large silicate cement specimens, and a quarter-inch gauge length was used on all the others.

A low cross-head speed of 0.002 inch per minute was selected to allow time for simultaneous measurements of strain in some

materials. This constant cross-head speed (stretching rate) produced a somewhat lower loading rate in terms of pounds per square inch per minute on the more pliant materials than on the rigid ones.

3. RESULTS AND DISCUSSION

3.1 Tensile Strengths

Tensile strengths were determined for human enamel and dentin, bovine enamel and dentin, five brands of silicate cement, two brands of zinc phosphate cement, four brands of direct filling resin, an aluminum alloy and 11 groups of experimental materials containing from 64 to 72 percent treated fused silica powder with a crosslinked polymer (Tables II, III and IV).

The enamel was seen to be much lower in tensile strength than the dentin. The bovine enamel did not have the lamellae, cracks or surface crazing often seen in the human enamel; this may have accounted for the higher tensile strength of bovine enamel.

As a group, the silicate cements had about one-half the tensile strength of enamel and about one-tenth that of dentin.

The tensile strengths of the zinc phosphate cements were of the same order as silicate cements.

The ultimate tensile strengths of the commercial direct filling resins and the experimental silica-resin material were about midway between enamel and dentin (Tables II, III, and IV).

The average tensile strength of the six aluminum alloy 2024-T4 (24S-T4) specimens tested was within four percent of the average

value reported in literature [10] which substantiates the method and values for tooth structures.

With the silica-resin material, the surface treatment of the silica powder was very important. All specimens were measured after the 7-day immersion of the materials in water. The material using powder with the best surface treatment had three times the tensile strength of material otherwise the same except that the powder had no surface treatment. This difference would probably have been much less if the materials had not been immersed in water for a week.

An average tensile strength of 4,800 PSI was obtained when 70 to 72 percent of the vinyltrichlorosilane-treated silica was used with resin A201-85C (not shown in tables). For the data shown in Table IV, however, only 67 percent was used because no more of the untreated powder could be incorporated into the mix; for comparison, the same percentage was used with all of the powders.

The best properties appeared to be obtained when as much as possible of the powder (with the best surface treatment) was mixed with a given amount of resin sirup; this was usually about 70 percent treated silica and 30 percent resin by weight.

3.2 Moduli of Elasticity

The modulus of elasticity (stiffness) is the ratio of stress to corresponding strain below the proportional limit. This is an indication of the amount of deformation that will occur in the

dental material when a masticatory load is applied to it. This is important in the distribution of stresses within a restored tooth during mastication.

Stress-strain curves were made and Young's modulus of elasticity was determined in tension for human dentin, a silicate cement, two commercial direct filling resins one of which contained about 25 percent glass fibers averaging about one-half millimeter in length), a silica-resin material and a standard aluminum alloy as a reference material.

Typical stress-strain curves in tension for human dentin and these restorative materials are shown in Figure 8. The values for Young's modulus of elasticity in tension were determined in the straight-line portion of the curves and these values are shown in Table V.

The modulus of elasticity in tension for human enamel could not be determined by the method described due to difficulty of getting long enough enamel tensile specimens for the attachment of Tuckerman optical strain gauges.

The modulus for human enamel in compression is reported in the literature to be in the range of 1.4 to 6.9 x 10^6 depending on the location and orientation of the specimens relative to the anatomy of the teeth [14].

In order to test the overall accuracy of the method used, an aluminum alloy 2024-T4 (24S-T4) was tested in the same manner as were the dentin specimens, and the average modulus values for

the aluminum alloy was within three percent of its value reported in the literature [10].

These values of the modulus of elasticity in tension for dentin and these dental materials are in good accord with values that have been measured in compression [14, 15].

Silicate cement had a modulus of elasticity equal to dentin.

The modulus of elasticity of the silica-resin material was measured to see if the incorporation of 70 percent treated silica powder into an organic resin would give a material with significantly higher stiffness than that of an unreinforced plastic. It did, as shown in Table V. The modulus of the highly reinforced resin approached that of dentin. Young's modulus of elasticity was about six times higher than in the commercial direct filling resins (Fig. 8). These latter resins had moduli only one-tenth that of dentin.

4. SUMMARY AND CONCLUSION

1. A method of preparing specimens of enamel and dentin for testing tensile properties was developed.
2. The tensile strengths of tooth structures and some filling materials were determined. The tensile strength of enamel was lower than dentin but higher than a silicate cement.
3. The modulus of elasticity in tension of a silicate cement and human dentin was approximately the same. They had higher moduli of elasticity in tension than two commercial direct

filling resins.

4. An experimental filling material containing 70 percent treated fused silica powder and 30 percent organic polymer had a modulus of elasticity approaching the values of dentin and had tensile strength values between those of dentin and enamel.

5. The tensile strength of a highly filled resin was dependent upon the surface treatment of the filler.

5. REFERENCES

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15. Craig, R. G. and Peyton, F. A. Elastic and mechanical properties of human dentin. J. D. Res. 37:710 August 1958.

TABLE I
COMMERCIAL MATERIALS TESTED

Brand Name	Manufacturer	Lot Number or Date Received
Achatite	Vivadent Corporation	Jan. 5, 1959
Ames Plastic Porcelain	The V-B Ames Company	July 1, 1957
DeTrey's Synthetic Porcelain	The L. D. Caulk Company	July 1, 1957
Fleck's Extraordinary	Mizzy, Incorporated	Powder: C-9 Liquid: E-84
Kadon	The L. D. Caulk Co.	Powder: 16733-S Nov. 1958 Liquid: November 1958
Mer-Don 7	American Consolidated Manufacturing Company, Incorporated	Powder: February 2, 1960 Liquid: December 22, 1959
Improved Replica Colorfast Plastic Filling Material	Cosmos Dental Products, Inc.	December 22, 1959
Sevriton	The Amalgamated Dental Co., Ltd.	Powder: SE F2 Nov. 1958 Liquid: November 1958 Catalyst: MS3 Dec. 1958
Smith's Certified Enamel Improved	Lee S. Smith and Son Mfg. Co.	July 2, 1959
S. S. White New Filling Porcelain	The S. S. White Dental Mfg. Co.	June 9, 1959
S. S. White Zinc Cement Improved	The S. S. White Dental Mfg. Co.	Powder: 56799268 Liquid: 15759302

TABLE II

TENSILE STRENGTHS

Material	Average Tensile Strength in PSI*	Number of Speci-mens	Standard Deviation** in PSI	Coefficient of Variation,%***
Human Enamel	1,500	9	370	25
Human Dentin	7,500	9	1,500	20
Bovine Enamel	3,000	8	800	28
Bovine Dentin	8,500	8	1,700	20
Silicate Cements				
A	440	6	110	25
B	510	5	130	25
C	630	5	130	20
D	930	5	310	33
E	1,010	5	290	29
Zinc Phosphate Cements				
F	410	8	100	25
G	690	8	140	20
Direct Filling Resins				
H	3,800	5	100	3
I	4,000	5	530	13
J	4,100	5	150	4
K	5,100	5	620	12
Aluminum Alloy				
Aluminum Alloy 2024-T4 (24S-T4)	65,500	6	2,200	3
Accepted Values[10]	68,000	-	-----	--

* PSI = Pounds per square inch

** Standard Deviation = $\sqrt{\frac{\Sigma(\bar{x} - x_1)^2}{n-1}}$

*** Coefficient of variation = $\frac{\text{standard deviation}}{\text{average}}$

TABLE III

TENSILE STRENGTHS

Material	Average Tensile Strength in PSI	Number of Speci-mens	Standard Deviation in PSI	Coefficient of Variation,%
Methyl methacrylate sirup [11] plus 66% fine* silica treated with TMEVS**	3,700	6	620	17
Methyl methacrylate sirup [11] plus 72% coarse*** silica treated with TMEVS	3,900	6	330	9
Synthetic resin A152-105 plus 64% fine* silica powder treated with TMEVS	4,800	4	390	8
Synthetic resin A152-105 plus 64% fine* silica powder treated with vinyl trichlorosilane	5,400	5	480	9

* "Fine" powder passed a No. 325 Sieve [12].
** TMEVS = tris (2-methoxyethoxy) vinylsilane [13].
*** "Course" powder passed a No. 100 Sieve, but was retained on a No. 325 Sieve [12].

TABLE IV

TENSILE STRENGTHS

Synthetic Resin A201-85C plus 67% silica passing a No. 400 sieve[10] and treated as follows:	Average Tensile Strength in PSI	Number of Speci-mens	Standard Deviation in PSI	Coefficient of Variation,%
No treatment (control powder heat-cleaned only)	1,500	7	300	20
Control powder treated with gamma-amino propyl triethoxy-silane then with 0.9% glycidyl methacrylate	2,700	6	610	22
Control powder treated with 0.5% tris (2-methoxyethoxy)-vinylsilane (TMEVS) using morpholine as a catalyst	3,000	6	900	30
Control powder treated with 1.5% TMEVS using sodium hydroxide as a catalyst	3,000	6	280	9
Control powder treated with 1.0% TMEVS using sodium hydroxide	3,800	6	630	16
Control powder treated with 0.5% TMEVS using sodium hydroxide	3,800	6	450	12
Control powder treated with 0.9% vinyl trichlorosilane applied in an inert solvent	4,400	6	990	23
Control powder treated with 0.9% vinyl trichlorosilane and 2% synthetic resin sirup	4,600	6	900	20

Double lines separate groups with significantly different strengths ($P < 0.05$).

TABLE V

MODULI OF ELASTICITY

Material	Modulus of Elasticity x 10^6 PSI	Number of Specimens	Standard Deviation x 10^6 PSI	Coefficient of Variation, %
Human Dentin	2.8	7	0.79	28
A Silicate Cement	3.1	5	0.89	29
A Direct Filling Resin	0.26	6	0.01	5
A Direct Filling Resin Containing 25% Glass Fibers	0.28	5	0.03	11
An Experimental Silica-Resin Material with 70% Treated Silica Powder	1.6	10	0.20	12
An Aluminum Alloy 2024-T4 (24S-T4)	10.3	6	1.2	11
Accepted Values [10]	10.6	-	---	--

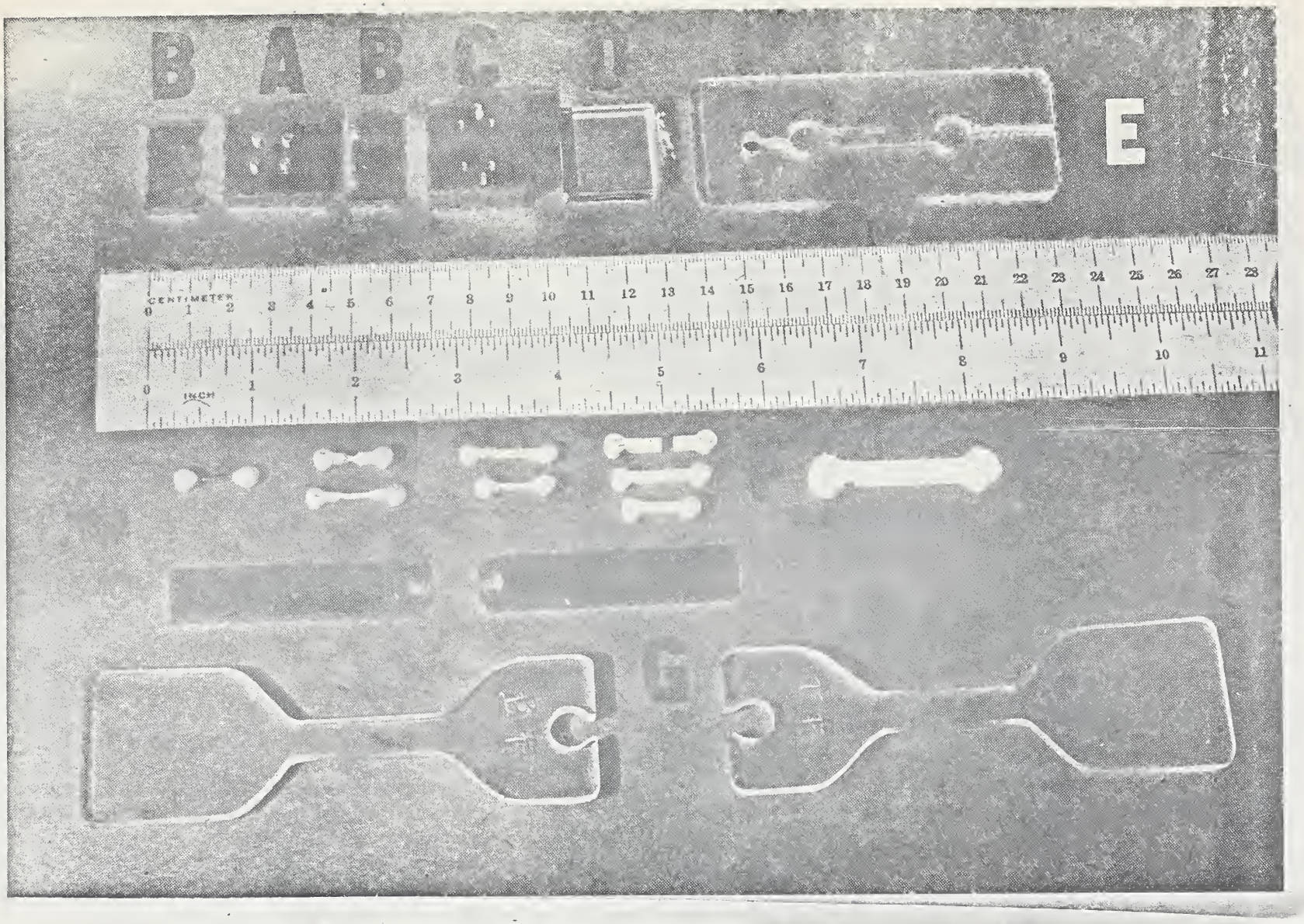

Figure 1. Molds used to prepare specimens:

A = bottom of stainless steel mold;
B = sides of mold;
C = bottom and sides of mold assembled;
D = glass top of mold;
E = sides of poly (methyl methacrylate) mold;
F = specimens prepared in molds;
G = grips used to apply loads to specimens in testing.

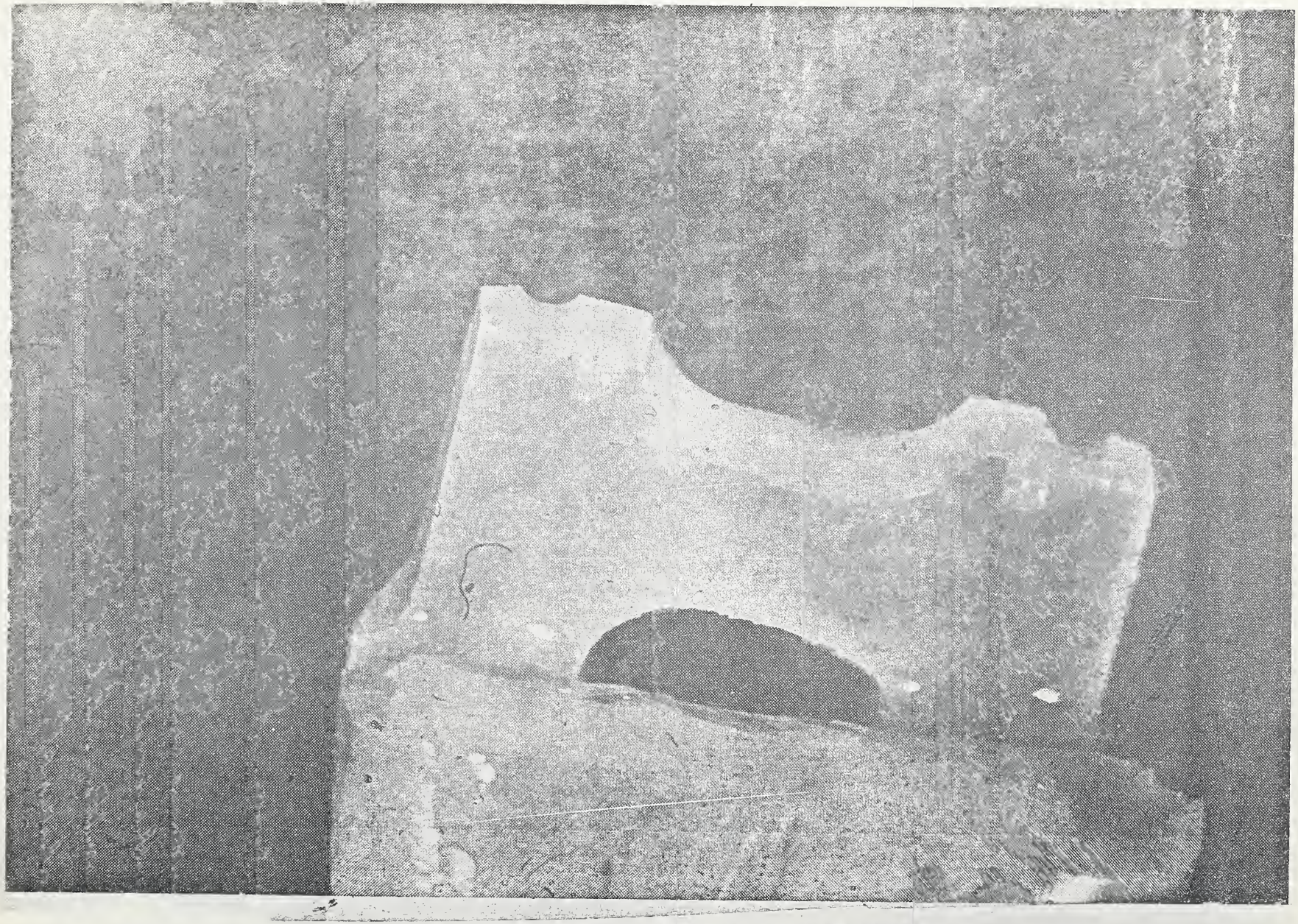

Figure 2. Preparation of human enamel specimen.

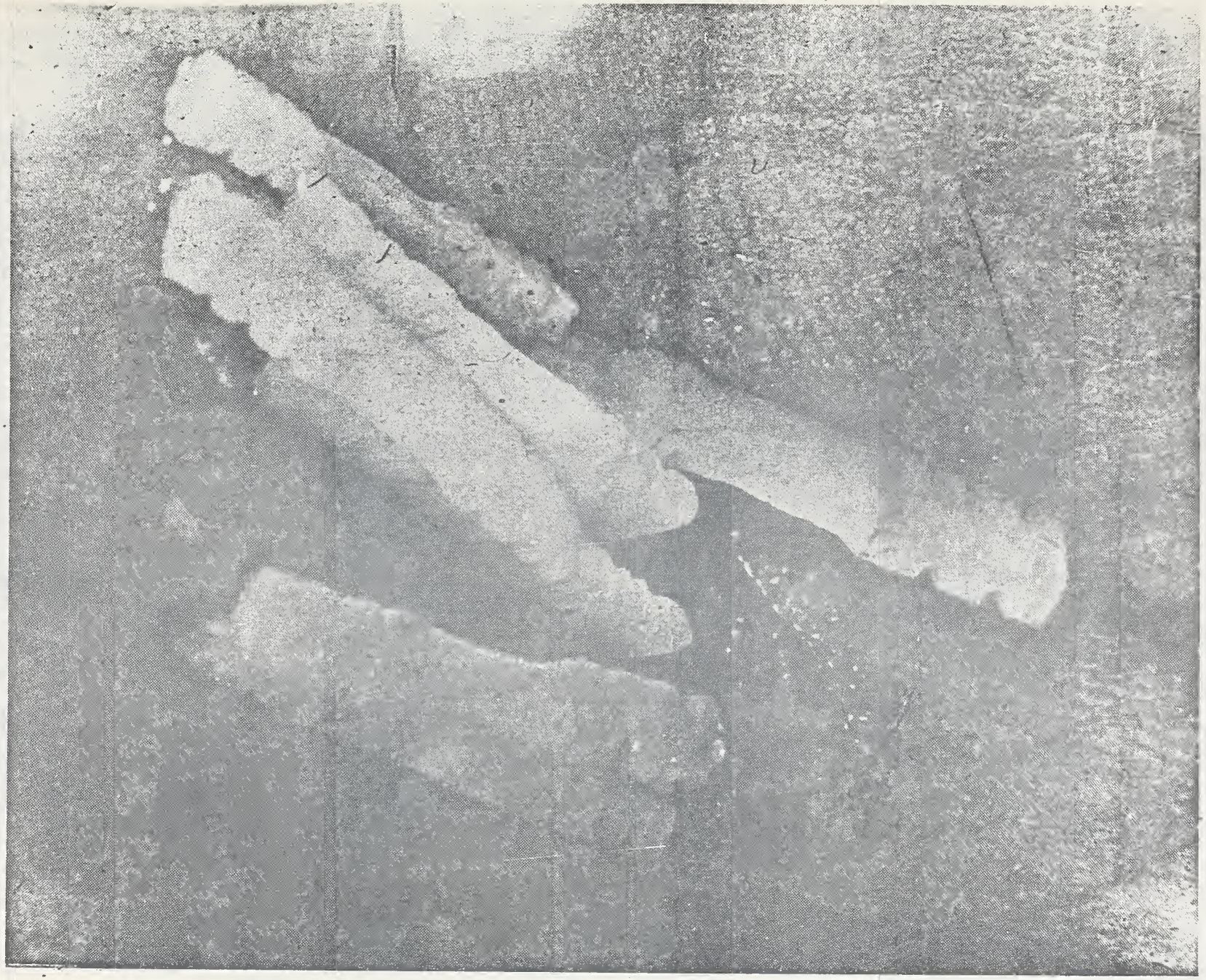

Figure 3. Dentin specimens.

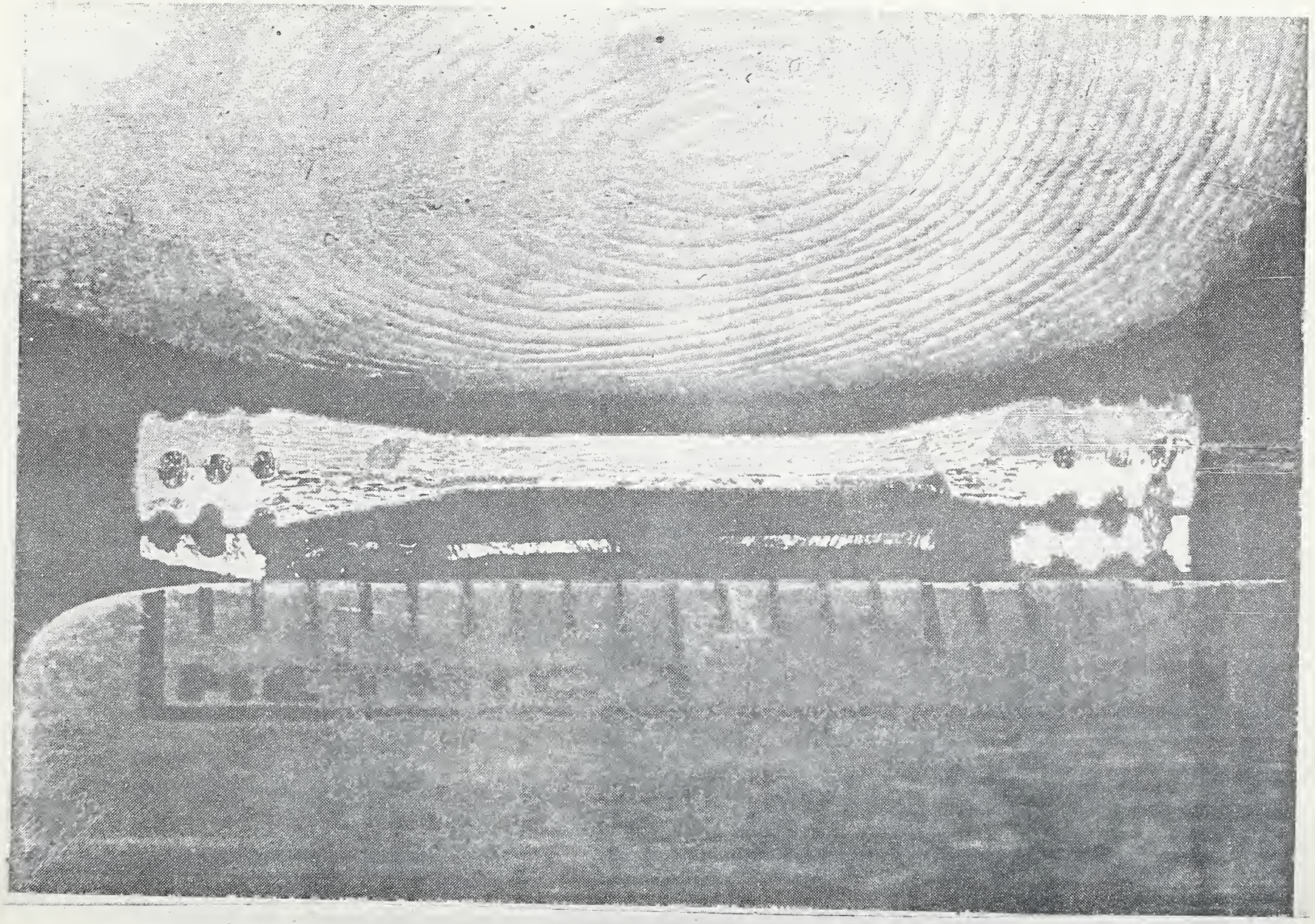

Figure 4. Aluminum (control) specimen; the smallest units on the scale are millimeters.

Figure 5. Specimen in mold before potting ends in plastic. The stainless steel split mold holds the narrowed isthmus portion of specimen (wrapped in tin foil strip). The notched ends of specimen are visible before they are embedded in plastic.

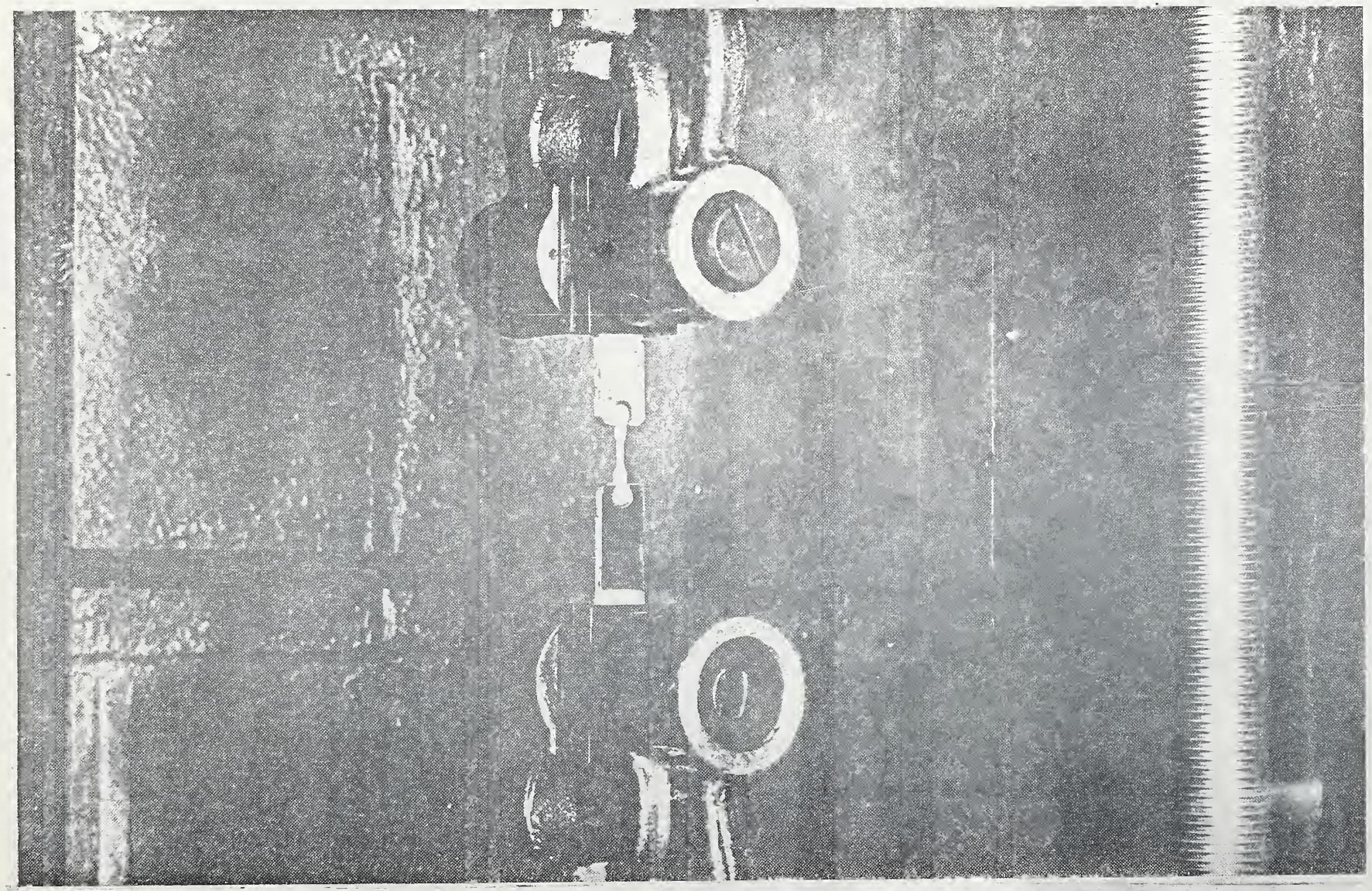

Figure 6. Specimen held by grips for applying tensile load.

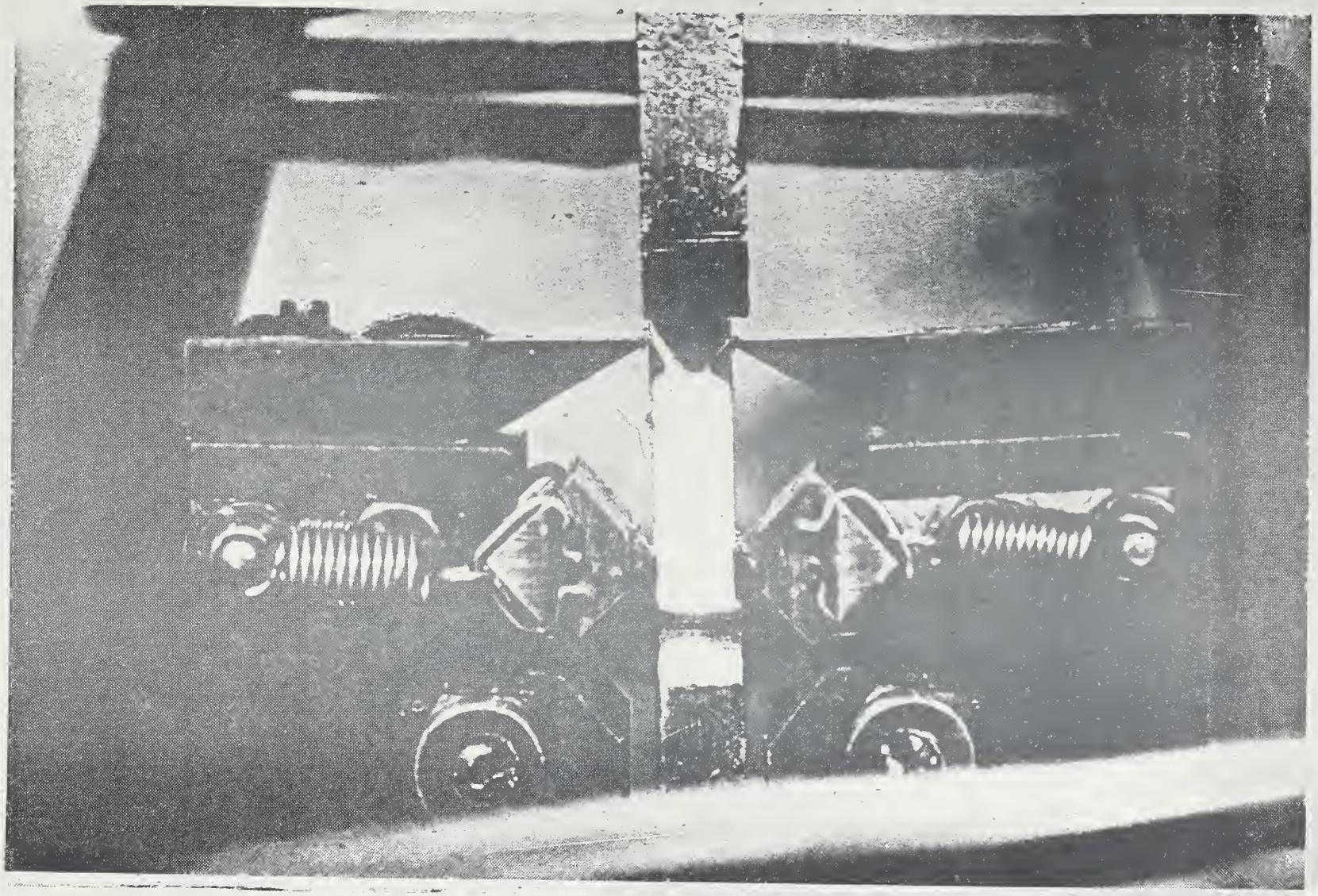

Figure 7. Measurement of elongation under tensile loading. The optical strain gauges [8] have their knife edges attached directly to specimen; the specimen (in the center) is partly hidden by the grips which apply the load.

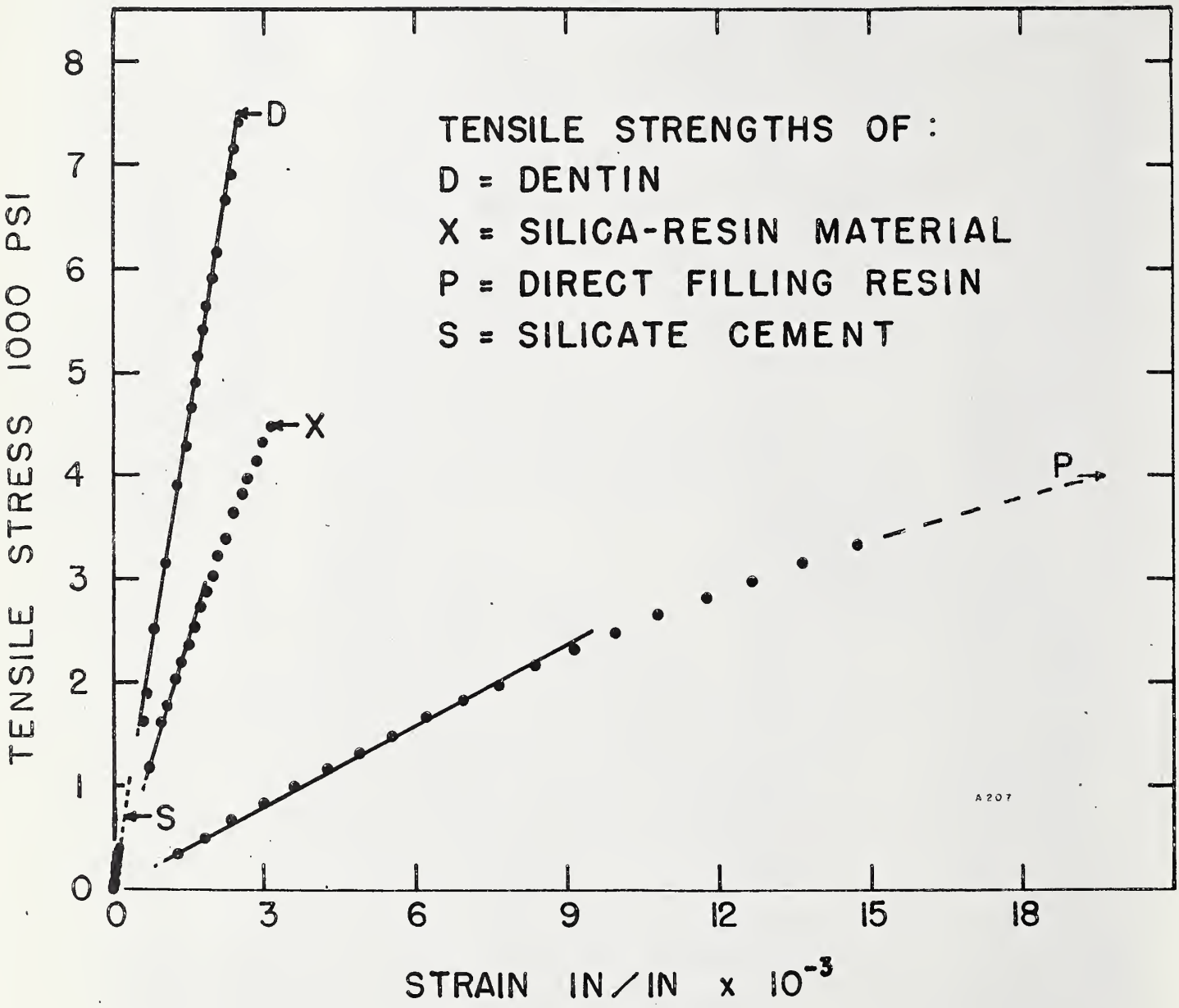

Figure 8. Typical stress/strain curves in tension of dentin and some filling materials. The arrows indicate the average tensile strengths. The steeper slopes show greater relative stiffness of the materials.