# NATIONAL BUREAU OF STANDARDS REPORT

10 068

Progress Report

on

DETERMINATION OF SHEAR MODULUS OF DENTAL MATERIALS BY MEANS OF THE TORSION PENDULUM



U.S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

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# DETERMINATION OF SHEAR MODULUS OF DENTAL MATERIALS BY MEANS OF THE TORSION PENDULUM

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### DETERMINATION OF SHEAR MODULUS OF DENTAL MATERIALS BY MEANS OF THE TORSION PENDULUM

By

J. A. Barton, Jr., G. Dickson, P. L. Oglesby, and J. W. Kumpula

# 1. Introduction

The determination of physical and mechanical properties of dental materials is often complicated due to the small size specimen necessary to compare dimensionally with a dental restoration. A method for determining the shear modulus of such small specimens has been developed employing a torsion pendulum apparatus.

Previous efforts have demonstrated the torsion pendulum as being a sensitive instrument for determining the shear properties particularly on plastic and rubbery material.<sup>1</sup> Myerson investigated the structure of cross-linked methacrylate systems by studying the changes in the mechanical energy absorption and stiffness using this method.<sup>2</sup> Shear modulus and mechanical loss tangent of four denture base polymers have been determined using a torsional pendulum apparatus.<sup>3</sup>

If a torsion pendulum is allowed to oscillate freely after an initial force is applied, it will oscillate at a frequency dependent upon the characteristics of the torsion bar and the moment of inertia of the system. The amplitude of the oscillations will decay or damp with time as energy is dissipated. From the frequency of the system the shear modulus of the torsion bar material can be calculated by means of the relationship<sup>1</sup>

$$G = 2 I \ell (4 \Pi^2 - \delta^2) f^2$$
$$\Pi r^4$$

where G = shear modulus I = moment of inertia & = length of specimen & = logarithmic decrement (to base e) f = frequency of the system r = radius of the specimen.

Where  $4 \Pi^2 >> \delta^2$ , the equation reduces to

$$G = 8\Pi I \ell f^2/r^4$$

### 2. Methods and Materials

The torsion pendulum apparatus shown in Figure I consists of a base which supports the pendulum; an upper collet which supports the upper end of the specimen which may be raised or lowered by means of a screw attached to the base; a lower collet which rigidly attaches the lower end of the specimen to the pendulum arm; two brass weights attached to the arm which may be positioned to increase or decrease the moment of inertia; and a slit aperture flag of brass which is suspended from one end of the pendulum arm. An electromagnet attached to the base of the instrument was used to activate the rotational vibrations to the specimen.

The recording system employs a light source directed on a photo multiplier tube through the slit aperture of the signal flag mentioned above. The head of the photo multiplier tube has a cover with an opening in its center in the shape of an equilateral triangle and was positioned to produce a decreasing signal as light travels from the base line toward the apex of the opening.<sup>4</sup> A Honeywell 906C Visicorder provided the tracing of the signal from the photo tube on light sensitive paper for frequency determinations. The end of the tube with its cover, the light source, and the slit aperture signal flag were covered to minimize the effects of light other than that of the light source, on the tracing.

The total moment of inertia was calculated taking into account each component (signal flag, steel arm, core, weights and collet) and was verified experimentally by observing the changes in frequency when the weights were moved and all other conditions were held constant. Lengths of the specimens between the collets were measured using a cathetometer. Radii of the specimens were measured with a micrometer. Frequency determinations were made by using the tracings from the recorder chart with time intervals provided from the

Visicorder. A standard one second interval signal from the National Bureau of Standards connected to the Visicorder was also used for time determinations.

Briefly, the operation of the instrument was as follows: the specimen in the form of a cylinder, whose diameter had been measured to the nearest 0.01 mm, was clamped between the two collets as shown in Figure II. The length of the specimen between the collets was measured with the cathetometer. The electromagnet, positioned in the plane of rotation of the pendulum arm was activated and released, producing typical oscillation curves as shown in Figure III.

The effect of temperature on shear properties of dental amalgam was determined over the temperature range of 23-51°C by placing the entire assembly in a controlled temperature chamber. Frequencies were measured at 4-5 intermediate temperatures between 23-51°C.

Since the maximum length to radius ratio that can be obtained with a specimen comparable to the size of a dental restoration is small, specimen end effects have a relatively large influence on the frequency of the system. This was initially observed using rods of steel brass, and aluminum, at different specimen lengths with the factors G, I, and r remaining constant. When using the equation  $G = 8 \Pi I \ell f^2 / r^4$ 

and assuming l to be the measured length of the specimen between the grips, the calculated value for G decreased as the specimen length between the collets was decreased. This error was corrected by determining an effective value for l equal to L, the measured length, plus a correction C. Frequency determinations were made at 4 to 5 lengths ranging from 6 to 12 mm on each specimen and G and C were determined using the least squares method by fitting the data to the equation

$$L = 15.37(\frac{r^4}{If^2}) G + C$$

where L = measured length of specimen between grips (in.)
15.37 = a constant (32.2 × 12)
r = radius of specimen (in.)
I = moment of inertia (lb/in<sup>2</sup>)
f = frequency of the system (H\_)
G = shear modulus (lb/in<sup>2</sup>)
C = specimen length correction (in.)

The effective length (L + C) was found to be larger than the measured length by approximately one to one and one half times the radius of the specimen.

Amalgam specimens were prepared from five different alloys certified to comply with American Dental Association for Specification no. 1/alloy for dental amalgam. The amalgam, with a 9 to 6 Hg-alloy ratio was mechanically triturated for thixty seconds and condensed into a split steel die. The cylindrical specimens were about 2.5 mm in diameter by 20 mm in length and had Hg contents of about 46%.

Cylinders of an experimental composite material of the same dimensions as the amalgam samples were also prepared in three different powder liquid ratios. These composites are based on a monomer formulation of a ternary eutectic system (isomeric pthalate esters of 2-hydroxyethyl methacrylates) and a resin filler consisting of vitreous silica and an x-ray opaque glass.<sup>5</sup>

### 3. Experimental Results

Shear modulus values with their respective standard deviations were obtained for each of the five amalgam alloys and three mixing consistencies of the experimental composite material. They are shown in Tables I and II with the values for dental amalgam in reasonably good agreement with those obtained by ultrasonic methods.<sup>6</sup> The shear modulus value of the experimental composite material using the torsion pendulum were about 10-30% lower than those obtained by ultrasonic methods.

Shear moduli of dental amalgam obtained over the temperature range of 23-51°C are shown in Figure IV. A decrease of shear modulus is observed as temperature is increased

#### 4. Discussion

By using the value for Poisson's ratio from reference 5 and experimental values for shear modulus G, Young's modulus E can be calculated from the equation

$$E = 2G(1 + \gamma)$$

where E = Young's modulus G = shear modulus $\gamma = Poisson's ratio$ 

These values are shown in Tables I and II.

The decrease in shear modulus of dental amalgam between 23 and 51°C is consistent with the results obtained for other mechanical properties. For example, Caul, Longton, Sweeney and Paffenbarger show an average decrease of approximately 30% in compressive strength of five amalgams between 23 and 50°C.<sup>7</sup>

One objection to the system is the fact that dynamic values may be affected to some extent by tensile or compressive loads on the specimen. However, it was observed that slight compression of the specimen by engaging the fine steel point attached to the lower collet into the lucite plate, produced no detectable effect upon the frequency during vibration.

Since the value of the radius is raised to the fourth power in the calculations, an error in measurement due to

irregularities on the surface of the specimen or nonuniformities in diameter, result in relatively large errors in the value for shear modulus. Other possible sources of error in the apparatus used in this investigation arise from the inability to read the frequency of the system on the recorder chart to better than one or two percent. Nonuniformities present in any inhomogeneous material also may contribute to a source of variation in results.

## 5. Conclusion

A method for determining the shear modulus characteristics of dental amalgam and an experimental composite material using the torsion pendulum has been demonstrated.

Shear modulus values of approximately 3 x 10<sup>6</sup> psi have been obtained which are in relative agreement with those obtained by other methods for dental amalgams. A decrease of shear modulus for dental amalgam was observed as temperature was raised to 51°C.

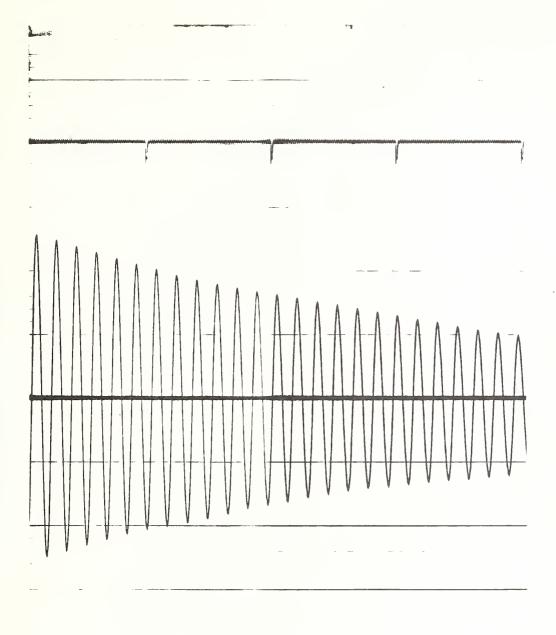
Shear modulus of an experimental composite material was determined by this method using three different mixing consistencies, with values of 0.7 to 0.8 x  $10^6$  psi being obtained compared to 0.9 to 1.0 x  $10^6$  psi by ultrasonic methods.

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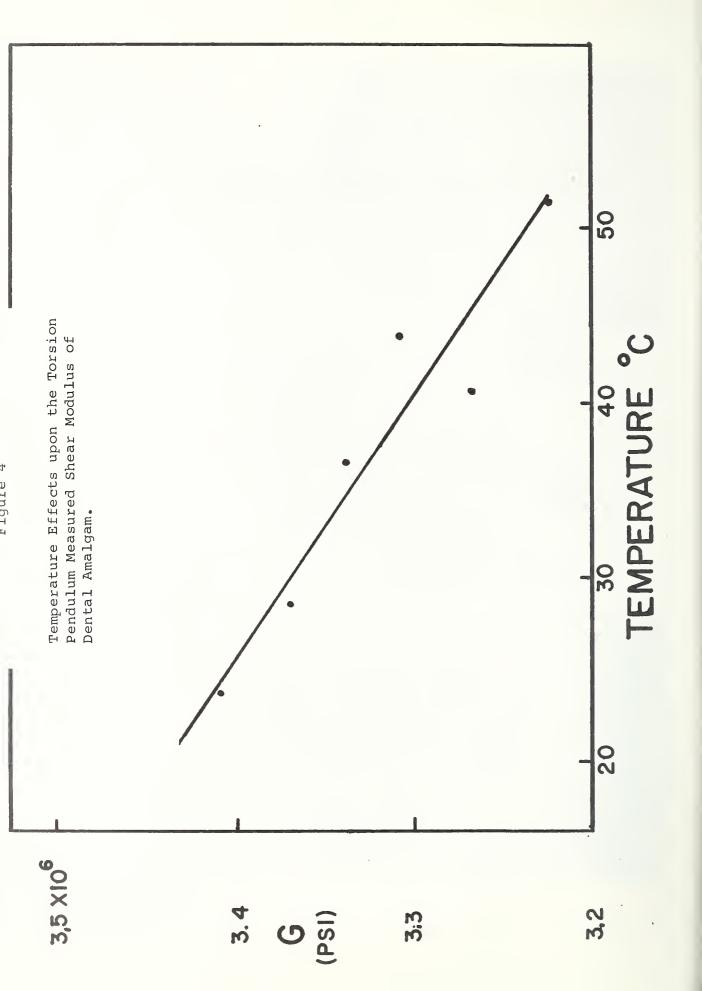






# Figure 3

Typical Oscillation Curve Recorded on Measuring Shear Modulus of Dental Amalgam by the Torsion Pendulum Method.



### Table l

Alloy		(	E								
	N∕m²	S.D.	PSI	S.D.	™m²	PSI					
A	22.8 x 10 <sup>9</sup>	0.76 x 10 <sup>9</sup>	$3.3 \times 10^6$	0.11 x $10^{6}$	60.7 x 10 <sup>9*</sup>	8.8 x 10 <sup>6</sup> *					
В	22.1	0.21	3.2	0.03	58.6 *	8.5 *					
с	20.7	1.52	3.0	0.22	55.2 *	8.0 *					
D	20.7	0.83	3.0	0.12	55.2 *	8.0 *					
E	20.0	2.14	2.9	0.31	53.1 *	7.7 *					
F <sup>4</sup>	20.7†		3.0†		55.2	8.0					
G <sup>6</sup>	23.4		3.4		62.7	9.1					

# Elastic Moduli of Dental Amalgam

- \* <u>Young's Modulus</u>--Calculated using values for G and Assuming P isson's ratio to be 0.334<sup>6</sup>.
- Shear Modulus--Calculated using values for E (diffraction grating<sup>4</sup>) and Assuming P isson's ratio to be 0.334<sup>6</sup>.

Table	Ι	Ι
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Elastic Moduli of Experimental Composit
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1.10		1.35		1.45	
N/m <sup>2</sup>	PSI	N/m <sup>2</sup>	PSI	N/m <sup>2</sup>	PSI
4.8 x 10 <sup>9</sup>	0.7 x 10 <sup>6</sup>	5.5 x 10 <sup>9</sup>	0.8 x 10 <sup>6</sup>	5.5 x 10°	0.8 x.10°
6.9	1.0	6.9	1.0	6.2	0.9
3.3 x 10 <sup>9</sup>	1.2 x 10 <sup>6</sup>	9.0 x 10 <sup>9</sup>	1.3 x 10 <sup>6</sup>	9.6 x 10 <sup>9</sup>	1.4 x 10 <sup>6</sup>
12.4	1.8	13.8	2.0	13.8	2.0
17.2	2.5	17.9	2.6	16.5	?.4
	4.8 x 10 <sup>9</sup> 6.9 8.3 x 10 <sup>9</sup> 12.4	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	$3.8 \times 10^9$ $0.7 \times 10^6 5.5 \times 10^9$ $0.8 \times 10^6$ $6.9$ $1.0$ $6.9$ $1.0$ $8.3 \times 10^9$ $1.2 \times 10^6$ $9.0 \times 10^9$ $1.3 \times 10^6$ $12.4$ $1.8$ $13.8$ $2.0^6$	$4.8 \times 10^9$ $0.7 \times 10^6$ $5.5 \times 10^9$ $0.8 \times 10^6$ $5.5 \times 10^9$ $6.9$ $1.0$ $6.9$ $1.0$ $6.2$ $8.3 \times 10^9$ $1.2 \times 10^6$ $9.0 \times 10^9$ $1.3 \times 10^6$ $9.6 \times 10^9$ $12.4$ $1.8$ $13.8$ $2.0^\circ$ $13.8$





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