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ULTRASONIC METHODS FOR DETERMINATION OF MECHANICAL PROPERTIES OF DENTAL MATERIALS

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1. INTRODUCTION

The most commonly reported mechanical property of dental materials is strength, usually compressive or tensile. However, strength is not a measure of the reaction of a material within its functional range. To obtain some characterization of the mechanical reaction of a material to the forces encountered in use, Young's modulus is often determined. Generally, Young's modulus is calculated from a stress-strain curve obtained by loading a specimen in a testing machine and measuring the change in length with increase in load. While the modulus determined in this way is usually considered a measure of the elastic characteristics of the material, the data often represent a combination of properties: elastic, retarded elastic and viscous with varying magnitudes of time and stress dependence. This is illustrated in the stress-strain curves for dental amalgam shown in Figure 1.[1]

It is evident that even with low stresses, the behavior of dental amalgam is not purely elastic. A plot of the strain of dental amalgam vs. time when under constant stress (Figure 2) indicates that at least three types of response to mechanical force are present, an

instantaneous elastic response, a retarded elastic response and a viscous response.[2]

To determine the magnitude of the instantaneous elastic response, it is necessary to isolate this particular property of the material.

Time dependent responses such as viscous creep and retarded elasticity can be eliminated or reduced to a negligible level if the stress can be applied and removed rapidly enough. One method of accomplishing this is by introducing sonic or ultrasonic waves into the material. Essentially this causes small volume elements of the material to be cyclically strained as a series of stress waves is propagated through the specimen. The velocity of the stress wave depends upon the mass or density of the particles or small volume elements of the material which are displaced and upon the elastic characteristics of the material which provide the forces tending to restore the particles to their equilibrium positions.

In addition to minimizing time dependent responses, ultrasonic methods have other advantages. They are rapid and non-destructive, and they permit repeated measurements on the same specimen as it is aged or subjected to temperature change or other treatments. Ultrasonic methods can be applied to small and brittle specimens on which other methods of stress application and strain measurement may be difficult. Also, ultrasonic methods are particularly suitable for

measuring elastic coefficients in different directions in anisotropic materials.

For measuring the mechanical properties of materials, low amplitude or low energy ultrasonic waves are employed and the effects of the medium on the wave are observed. High amplitude waves which would produce a permanent change in the specimen are avoided.

There are two types of stress waves which are particularly useful for determination of mechanical properties. These (Figure 3) are the compressional or longitudinal wave in which particle motion in the medium is parallel to the direction of propagation of the wave, and the shear or transverse wave in which the particles vibrate at right angles to the direction of propagation of the wave.

2. VELOCITY AND MODULUS DETERMINATIONS

In plane waves in an elastic homogeneous medium[3], the velocities of the transverse and longitudinal waves can be shown to be:

$$V_T = \sqrt{\frac{G}{\rho}} \qquad \text{and}$$

$$V_L = \sqrt{\frac{E(1-\gamma)}{\rho(1+\gamma)(1-2\gamma)}}$$

where

ρ = density

G = shear modulus

E = Young's modulus

γ = Poisson's ratio

When the wave length is large compared to specimen dimensions[4], the equations for propagation in an infinite elastic medium do not apply. For example, for wave propagation parallel to the sides of a thin plate

$$V_L = \sqrt{\frac{E}{\rho(1-\gamma^2)}} \quad .$$

For propagation in a rod with radius small compared to wavelength[4], the longitudinal wave velocity is

$$V_L = \sqrt{\frac{E}{\rho}} \quad .$$

Where frequencies high enough so that the wavelength is small compared to specimen dimensions are used[3], shear modulus, Young's modulus, the bulk modulus and Poisson's ratio can be determined from the velocities of the longitudinal and transverse waves, and the density of the material as follows:

Shear modulus $\quad G = \rho V_T^2$

Young's modulus $\quad E = \rho V_T^2 \left(\frac{3V_L^2 - 4V_T^2}{V_L^2 - V_T^2} \right)$

Bulk modulus $\quad K = \rho \left(\frac{3V_L^2 - 4V_T^2}{3} \right)$

Poisson's ratio $\quad \gamma = \frac{V_L^2 - 2V_T^2}{2(V_L^2 - V_T^2)}$

From these equations, it is evident that determination of elastic constants is essentially a problem of determination of the velocity of sound in the material. Various methods can be employed for determining the velocity of stress waves. One of the most widely used is the pulse-echo technique, which has been described in numerous papers and books[3,4,5,6]. In this method (Figure 4), a quartz crystal, or other transducer driven by a pulsed oscillator, is used to introduce the wave at one of two parallel faces of the specimen. The same transducer, or a similar one at the opposite face of the specimen, is used to receive the mechanical vibration and produce an electrical signal which, after amplification, can be observed on an oscilloscope. Unless the attenuation of the specimen material is very high, the stress wave will echo back and forth through the specimen a number of times, and signals representing each double transit will be observed.

If a piezoelectric quartz transducer is used, the crystal may be cut with axes oriented so as to produce either longitudinal vibrations (x cut) or transverse vibrations (Y or AC cut) in the specimen. The crystal can be driven at its natural frequency or at an odd harmonic by a pulsed oscillator tuned to the required frequency. The time interval from one pulse to the next is made long enough to allow the echoes to die out between pulses. The transducers can be coupled to the specimen with a thin film of oil or a viscous resin.

A waveform generator (Figure 4) can be used to control the pulse repetition rate and supply a trigger signal to initiate both the pulse and the oscilloscope sweep. The rectified echo signals, after passage

through a broadband amplifier, may be displayed on an oscilloscope (Figure 5) with a calibrated time scale and the time between echoes determined. The frequency, pulse length and pulse repetition rate may vary over a wide range.

Accuracy of the velocity determination will depend on the quality and quantity of echoes which can be observed, which, in turn, depend upon the attenuation of the signal by the specimen material, the presence of spurious signals which may result from reflection from sides of the specimen with the probability of mode conversion from longitudinal to transverse waves or vice versa and to unsatisfactory specimen preparation. Specimen parallelism to 2 parts in 10^4 or better is recommended, although less accurate parallelism may produce usable results for some purposes. Under favorable conditions, velocity determinations with a sensitivity of 0.1% and an accuracy of 1% can be obtained[5].

In another pulse method, the sing-around technique (Figure 6), the signal received when the stress wave has passed through the specimen is used to trigger the next pulse from the oscillator, and the pulse repetition frequency is measured. Since time delays other than that required for the stress wave to pass through the specimen are ordinarily comparatively small, the period of the system is essentially the transit time. For accurate determinations, these other delays must be taken into account. With this simple system, velocity changes of less than one part in 10^5 can be detected[5].

Refinements of the sing-around method have been made to provide

sensitivity to velocity changes of a few parts in 10^7 [7]. These refinements consist of using the signal after a specific number of echoes rather than after the first transit to refire the transmitter and also using a specific cycle within the pulse. This method is particularly useful when the interest is in measurements of small changes in velocity rather than in an absolute value.

Interference methods can be used for measuring velocities. In optical interferometers, such as the type used in measuring the setting expansion of amalgam and in ultrasonic interferometers in a liquid medium, the number of wave lengths between two reflecting surfaces is changed by a continuous variation of path length. Such a system is not feasible for ultrasonic measurement of the properties of solids since path length cannot be readily varied. A similar effect can be obtained, however, by a continuous variation of frequency and consequent change in wave length. For example, sets of two gated, phase coherent pulses from a continuous wave oscillator may be spaced in time so that the second pulse which has passed once through the specimen arrives at the receiving transducer at the same time as the first pulse which has echoed and passed three times through the specimen[3]. If the oscillator frequency is then adjusted, the phase relationship of the pulses will vary and when the signals are added a series of nulls will be produced. The velocity can be determined from the frequency difference necessary to pass from one null to the next. If the phase change on reflection from the ends of the specimen can be neglected

$$V = 2\ell(f_2 - f_1) \quad .$$

where ℓ is the length of the specimen and f_1 and f_2 represent the frequencies at successive null positions. If the specimen material causes relatively high attenuation of the ultrasonic wave, a continuous wave interferometric method can be used[3]. Here, the signal from the receiving transducer is compared with the signal being fed into the transmitting transducer and again phase change is observed as frequency is varied.

Resonance methods can also be used for velocity determinations. Such methods generally are used with frequencies in the lower ultrasonic range. At the natural frequency of a half-wave length specimen bar, the longitudinal velocity is

$$V_L = 2\ell f$$

and Young's modulus is

$$E = \rho(2\ell f)^2 .$$

One experimental arrangement[8] which has been used for such resonance determination involves the use of a three-component system, shown in Figure 7, consisting of a quartz crystal driver with the specimen of closely matched resonance frequency cemented to one end and a second quartz bar, also closely matched in frequency, cemented to the other end of the driver to act as a piezoelectric gauge.

Another method of velocity measurement analogous to optical index of refraction measurement is based on the determination of the angle of refraction of a stress wave as it passes from a liquid medium into a solid specimen or on the determination of the focal length of an ultrasonic lens made from the specimen material[3].

Velocity measurements can also be made by reflection methods[3]. If ultrasonic pulses are reflected from the surface of a solid immersed in a liquid medium, the reflection coefficient can be defined as follows

$$\alpha_r = \frac{P_r}{P_i} = \frac{\rho_2 V_2 - \rho_1 V_1}{\rho_2 V_2 + \rho_1 V_1}$$

where

P_r and P_i represent the pressure amplitudes of the reflected and incident pulses, respectively;

V_1 and V_2 represent longitudinal wave velocities in the incident (liquid) and reflecting (solid) media;

ρ_1 and ρ_2 represent densities of the liquid and solid media.

Instead of making absolute measurements of the reflection coefficient, the amplitude of the pulse reflected from the test specimen may be compared with the amplitude of the pulse reflected from a specimen for which the reflection coefficient is known. In this case

$$\alpha_{r_2} = \alpha_{r_1}\left(\frac{A_2}{A_1}\right)$$

where A_1 and A_2 represent the amplitudes of the pulses reflected from the known and unknown materials, respectively. From the reflection coefficient and the density of the test material, the velocity of the longitudinal wave and the accoustic impedance, ρV, can be calculated. This method can be used with materials which have a high attenuation. It has the advantage also that only one flat surface is required on the specimen. The accuracy is reported to be of the order of 5 percent[3].

3. ATTENUATION OF ULTRASONIC WAVES

The attenuation of ultrasonic stress waves in materials and loss interactions are discussed in detail by Truell, Elbaum and Chick in a recent book, Ultrasonic Methods in Solid State Physics[5]. In brief, energy losses may be divided into two categories, those that are dependent on the physical characteristics of the specimen material and those that are characteristic of the method of measurement. In the latter group are such as coupling losses, losses due to non-parallelism of specimen faces, phase effects in the transducers and diffraction and mode conversion effects in the specimen. The losses that are of interest, those that are characteristic of the material, are of two main types, scattering effects and absorption effects. Scattering losses are caused by lattice defects or other discontinuities in the medium. The relative size of wavelength and defect and the defect density will determine whether or not the scattering can be measured. Adsorption losses include dislocation damping losses, thermo-elastic losses, conduction electron damping losses, phonon-phonon interactions, ferromagnetic resonance effects, paramagnetic resonance effect and nuclear spin energy interactions.

The attenuation of ultrasonic waves may be used to determine the internal friction of a material. Internal friction may be defined as the energy loss per cycle in an element of volume divided by 2π times the maximum energy stored in the element per cycle or as the tangent of the angle by which the strain lags behind the stress. When energy losses per cycle are not high[6]

$$Q^{-1} = \tan \alpha = \frac{\Delta E}{2\pi E} = \frac{\Delta A}{\pi A} = \frac{1}{\pi} \ln \frac{A_1}{A_2} = \frac{\delta}{\pi}$$

where E = energy

A = amplitude

δ = logarithmic decrement

The attenuation of ultrasonic waves in materials can be determined by a number of methods. With the pulse-echo technique, attenuation is measured by comparison of the amplitude of successive echoes. Losses which occur at each reflection also must be taken into consideration. Effects of coupling losses may be determined by making measurements with a transducer at only one end of the specimen and comparing with results obtained when an identical dummy transducer is coupled to the opposite end of the specimen[5]. By making measurements on specimens of different lengths, effects other than the increased path length can be kept constant and the attenuation calculated from measurements on the different specimens. Reflection of the wave from the sides of the specimen may result in mode conversion from transverse to longitudinal waves or vice versa, and result in energy loss and apparent attenuation. Such effects will depend on wavelength and specimen size.

When the attenuation is small, it can be determined from the decay curve of free vibrations in the specimen[6]. The resonance apparatus described for determination of velocity has as a primary use the determination of internal friction. It can be shown[8] that the logarithmic decrement of the specimen in such a system is proportional

to the ratio of the driver crystal voltage to the gage crystal voltage

$$\delta = K(\frac{Vd}{Vg})$$

where the constant K is a characteristic of the system. After K has been determined from the free decay of the system, the logarithmic decrement can be determined under different strain amplitudes or other specimen conditions by measurements of the two voltages.

The internal friction can also be determined from the amplitude frequency curve in the region of resonance from the relation

$$Q^{-1} = \frac{\Delta f}{f}$$

where Δf is the total spread from one side of resonance to the other where the amplitude has dropped to $\frac{1}{\sqrt{2}}$ times the resonance amplitude[6].

4. APPLICATIONS OF ULTRASONIC METHODS TO DENTAL MATERIALS

Ultrasonic methods have been used to measure the mechanical properties of a number of dental materials. Dickson and Oglesby[9] reported the elastic constants of dental amalgams in which the mercury content was varied by varying the condensation pressure. Specimens 8mm in diameter by 6 to 15mm in length were prepared as shown in Table 1. Measurements were made by a pulse-echo technic with a frequency of 5.5 MHz, a pulse length of 0.5 to 1.0 μ sec. and a pulse repetition rate of 6 KHz. Elastic constants obtained for an amalgam containing about 48% mercury are shown in Table 2. As the plot in Figure 8 shows, the relationship between Young's modulus and mercury content was essentially linear over the range investigated.

Using a resonance method with the three component system of specimen, driver crystal and gage crystal, Larson[10] investigated the Young's modulus and internal friction of amalgam. The frequency was in the 50 KHz range. Young's modulus values of 8.5×10^6 to 10.6×10^6 psi were found with mercury contents varying from 30 to 60%. A peak in both modulus and internal friction was reported in the 45 to 50% mercury ranges.

As would be expected, the Young's modulus values obtained for amalgam by the rapid strain rates inherent in ultrasonic methods are considerably higher than those reported from conventional stress-strain curve procedures. However, using a diffraction grating strain gage which permitted stress-strain curve data to be obtained in 3 to 4 seconds, Gardner, Dickson and Kumpula[11] obtained a modulus value of 8×10^6 psi which does not differ greatly from those obtained by ultrasonic methods.

In our laboratory, the pulse-echo method has been employed to measure the elastic characteristics of experimental composite quartz or glass-filled resins. The attenuation of the ultrasonic wave in these materials is high and often no suitable echoes are obtained so that the procedure is reduced to measurement of a single transit time. Effects of time delays other than transit time are eliminated by measurements on specimens of various lengths. Data on Young's modulus for an experimental restorative material are given in Table 3[12].

The elastic properties of apatites were investigated by Gilmore and Katz[13] using an ultrasonic interference technic. Powdered

specimens of hydroxyapatite, fluorapatite, chlorapatite and sodium chloride-hydroxyapatite composites were studied when subjected to pressures of up to 50 K bar. Transducers were mounted on the back faces of tungsten carbide pistons, as shown in Figure 9, and interference was obtained between reflections from the near and far specimen-piston interfaces. Values obtained for some of the apatites, as well as values for bovine dentin and enamel, are given in Table 4. Moduli of the apatites were found to increase linearly with pressure in the higher pressure ranges.

Lees[14] reported values for the specific accoustic impedance of bovine enamel and dentin obtained by the reflection coefficient method using specimens mounted in acrylic resin. A pulse of 60 nanoseconds length was reflected from the specimens in water. The signal amplitude was compared with that of a pulse reflected from stainless steel under similar conditions. Values obtained for accoustic impedance (product of density and sonic velocity) were in general agreement with values obtained by other ultrasonic methods.

The elastic coefficients of animal bone have been investigated by Lang[15] using an ultrasonic pulse method for measurement of velocities. Analysis of the crystallographic structure of components of bone and the piezoelectric and pyroelectric characteristics of bone suggested that bone should behave elastically as a hexagonal single crystal, and the elastic stiffness coefficients were determined on this basis. Young's and shear moduli found for axial and transverse bone directions, in dried bovine femur and phalanx and fresh

phalanx, are given in Table 5.

5. SUMMARY

A variety of ultrasonic methods are available for determining the elastic characteristics of materials. These methods are precise, rapid, non-destructive, applicable to small specimen, and they measure a specific property of the material. As a number of recent reports in the dental literature indicate, they offer particular advantages to the study of dental materials and mineralized tissues.

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TABLE 1

AMALGAM SPECIMEN PREPARATION[9]

11 to 8 Hg/Alloy ratio

20 sec. trituration, Wig-L-Bug

3 to 6 mixes of two pellets

Condensed in steel die

8 mm diameter by 6 to 15 mm length

5000 to 25000 psi

35 to 49% Hg content

TABLE 2

ELASTIC CONSTANTS OF AMALGAM[9]

Property	Value	Range*
Hg Content	48.6%	0.7
Young's Modulus	9.09×10^6 psi	0.06×10^6
Shear Modulus	3.41×10^6 psi	0.03×10^6
Bulk Modulus	9.12×10^6 psi	0.22×10^6
Poisson's Ratio	0.334	0.005

* Range of three values

TABLE 3

ELASTIC MODULI OF EXPERIMENTAL
COMPOSITE RESIN RESTORATIVE MATERIAL
(In 10^6 psi)

Modulus	Method	Powder-Liquid Ratio (grams powder to 0.4 ml liquid)		
		1.10	1.35	1.45
E	Stress-strain curve	1.2	1.3	1.4
E	Ultrasonic	2.5	2.6	2.4
G	Torsion pendulum	0.7	0.8	0.8
G	Ultrasonic	1.0	1.0	0.9

TABLE 4

ELASTIC MODULI OF APATITES[13]
(In 10^6 psi)

	E	G	K
Hydroxyapatite	15.7	7.7	12.5
Fluorapatite	----	7.5	13.8
Chlorapatite	----	6.3	10.0
Dentin	3.0	1.2	2.6
Enamel	10.7	4.4	6.7

TABLE 5

ELASTIC MODULI OF BOVINE BONE[15]
(In 10^6 psi)

	Phalanx		Femur
	Fresh	Dried	Dried
E (axial)	3.19	4.42	3.77
E (transverse)	1.64	2.31	2.60
G (axial)	0.78	1.09	1.18
G (transverse)	0.65	0.94	1.07

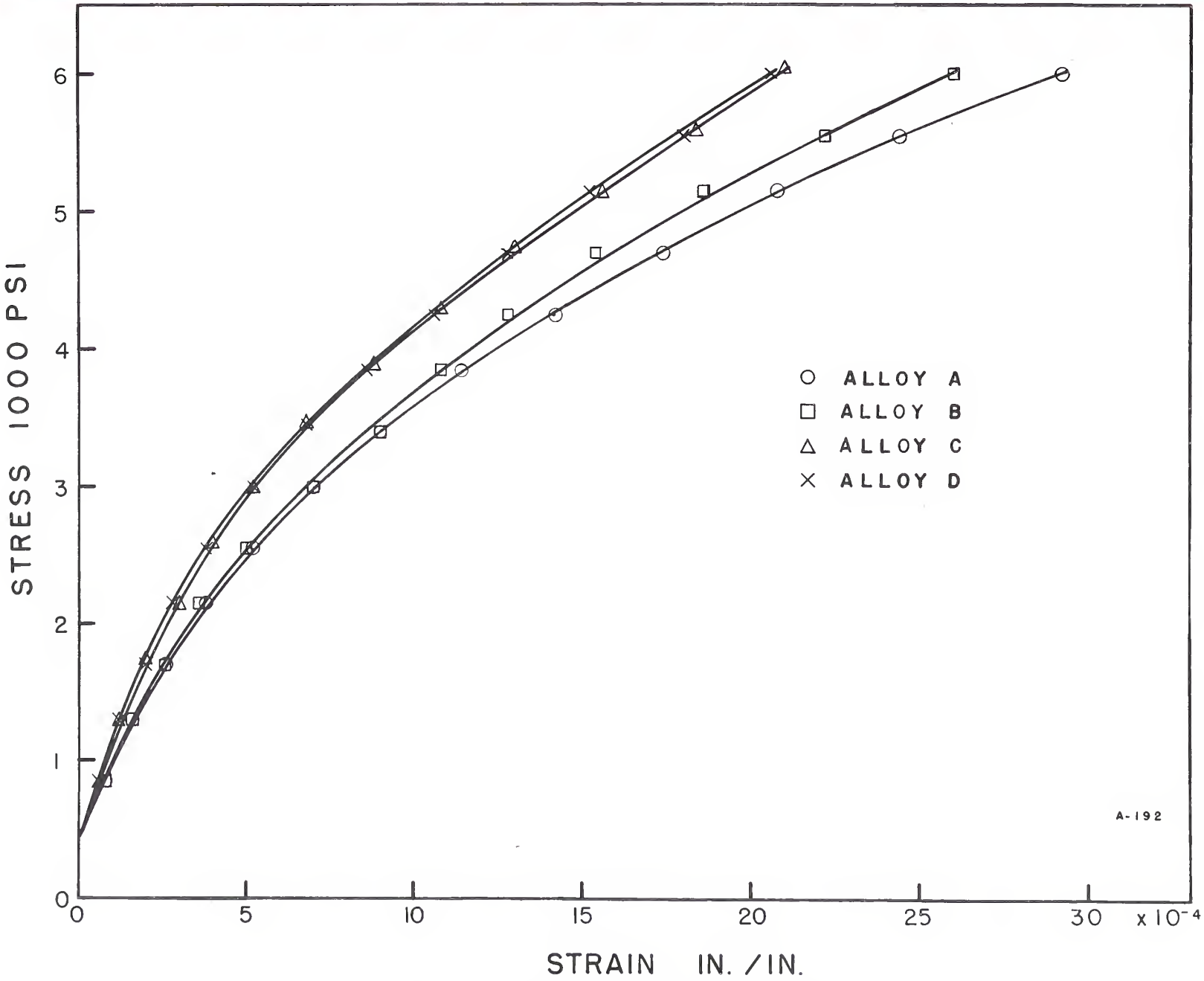


Fig. 1. Typical stress-strain curves of various amalgams in tension, using 0.003 in./min. head speed and 7 day old specimens (1)

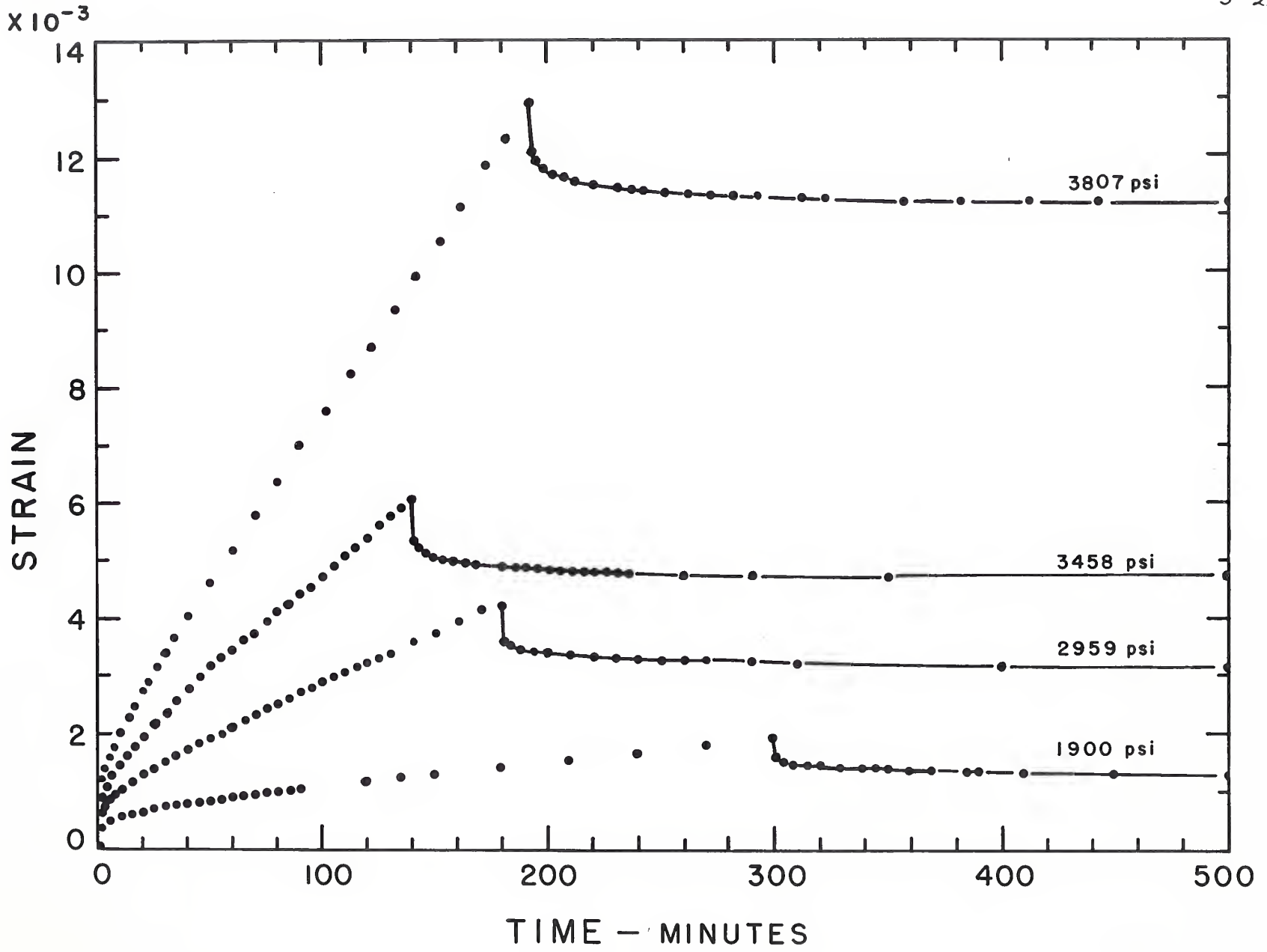
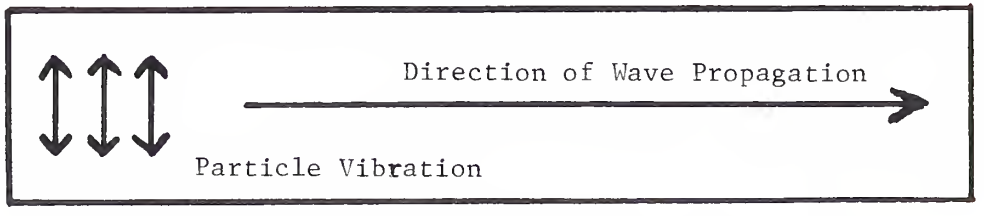
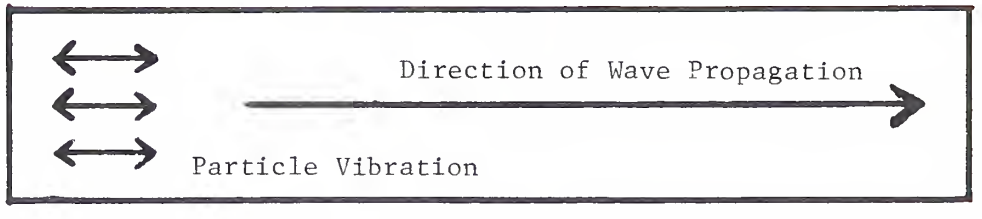


Fig. 2. Creep and recovery of amalgam loaded



Transverse Waves



Longitudinal Waves

Fig. 3.
Two types of ultrasonic stress waves used
for determination of mechanical properties of materials

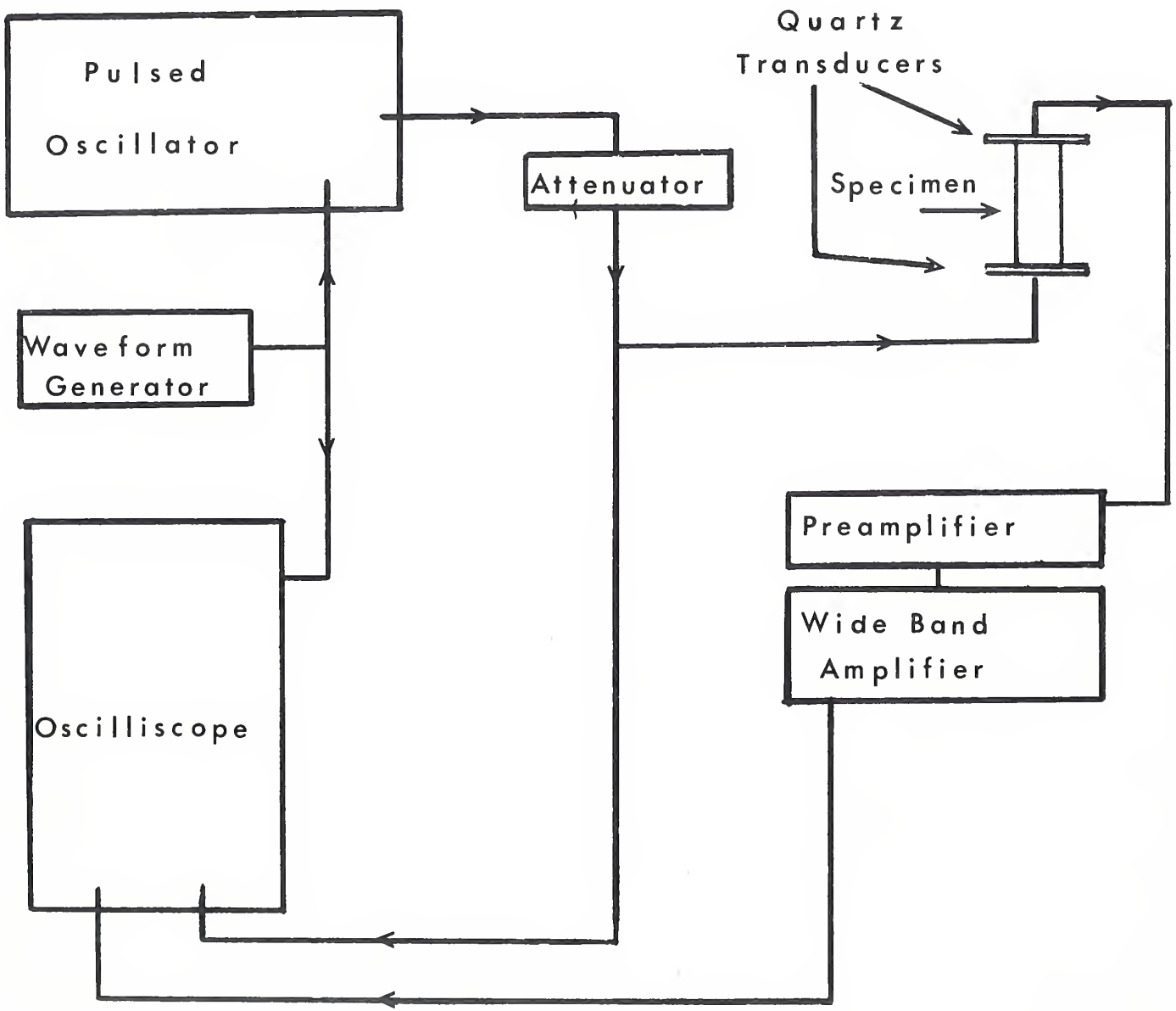


Fig. 4. Typical pulse echo system

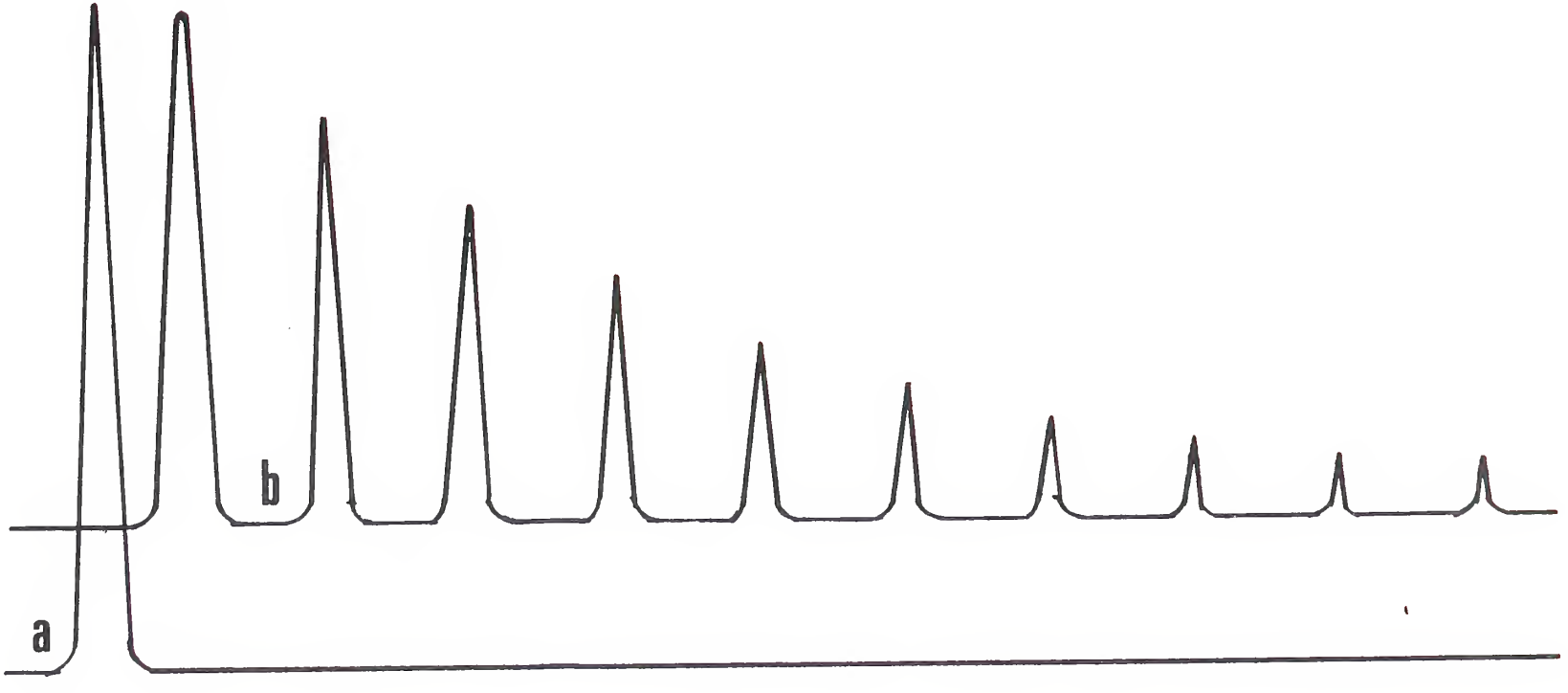


Fig. 5. Pulse echo pattern, a - input pulse, b - pulses after passage through specimen.

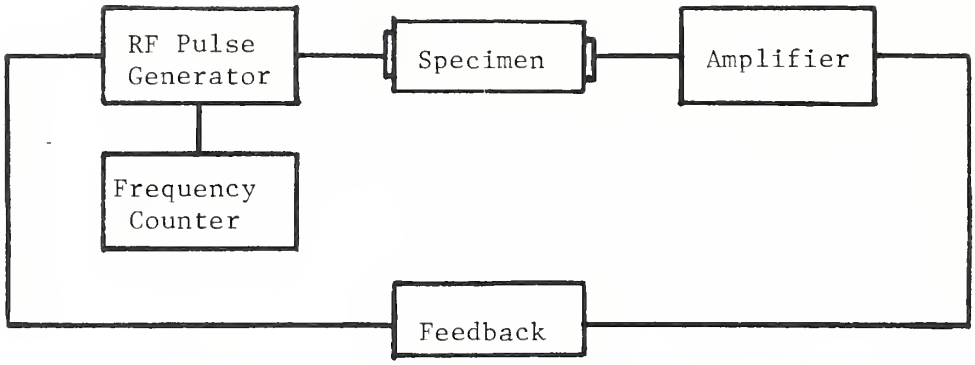


Fig. 6. Simplified diagram of sing-around system.

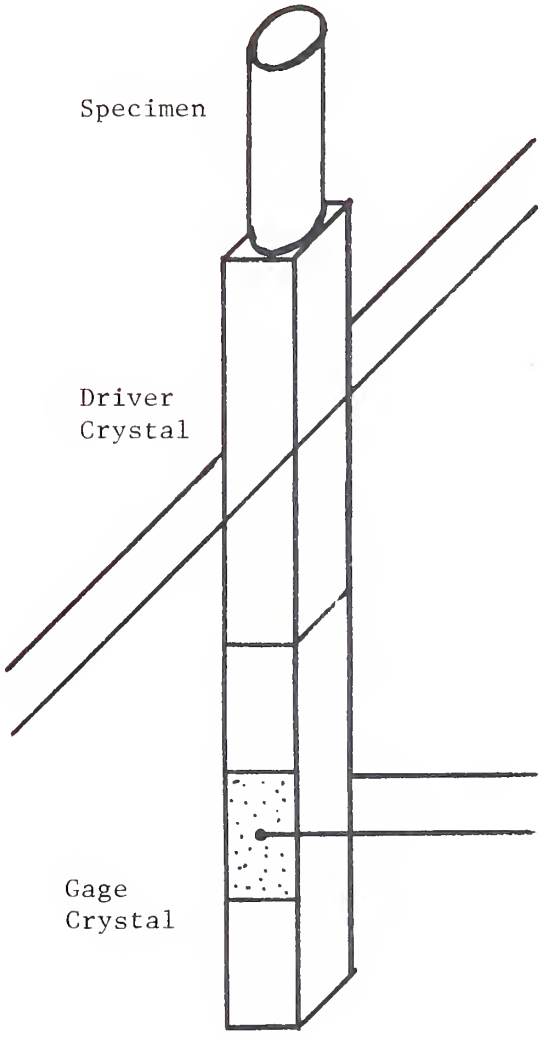


Fig. 7. Three component resonance system(8)

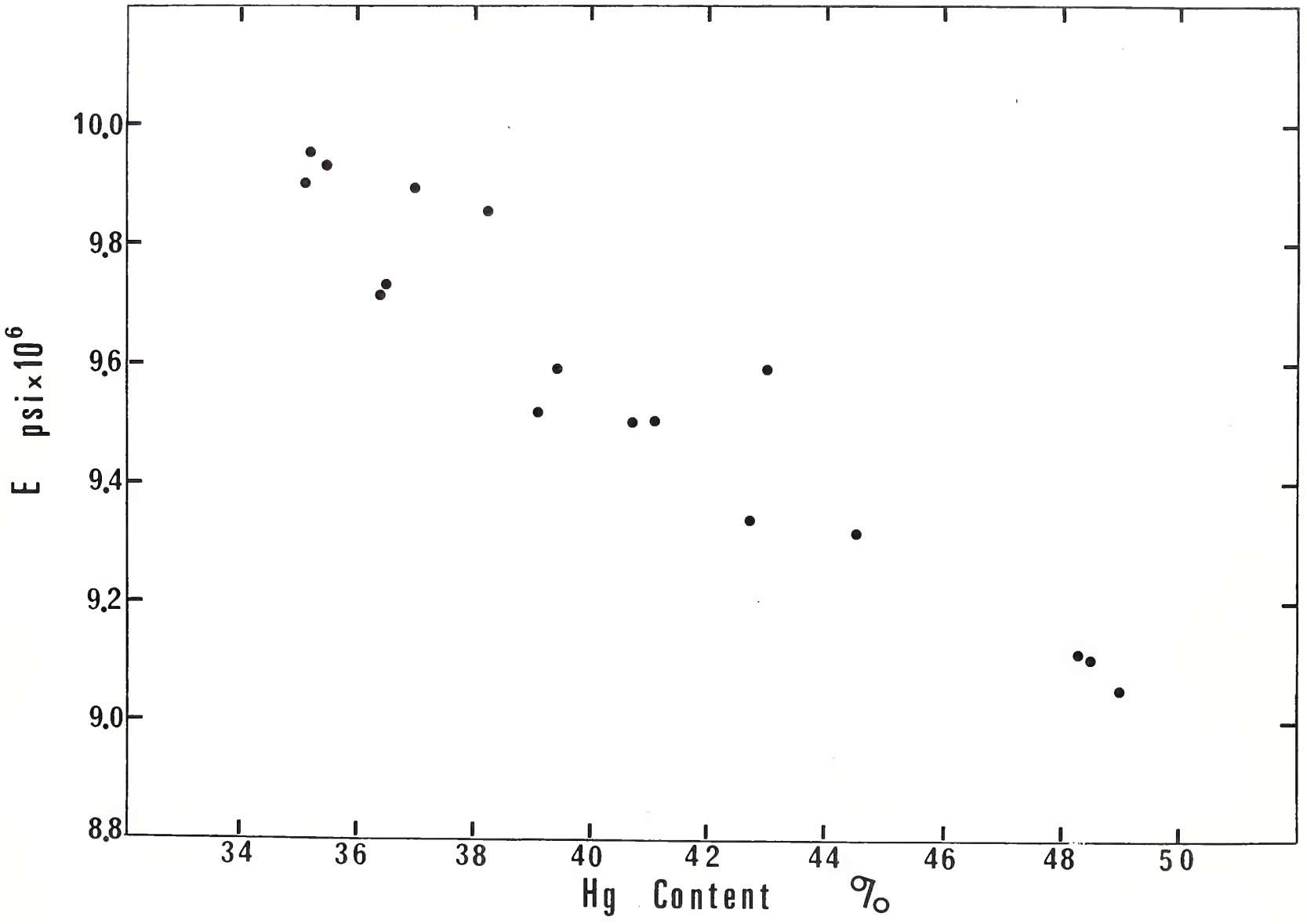


Fig. 8. Effect of mercury content on Young's modulus of dental amalgam[9]

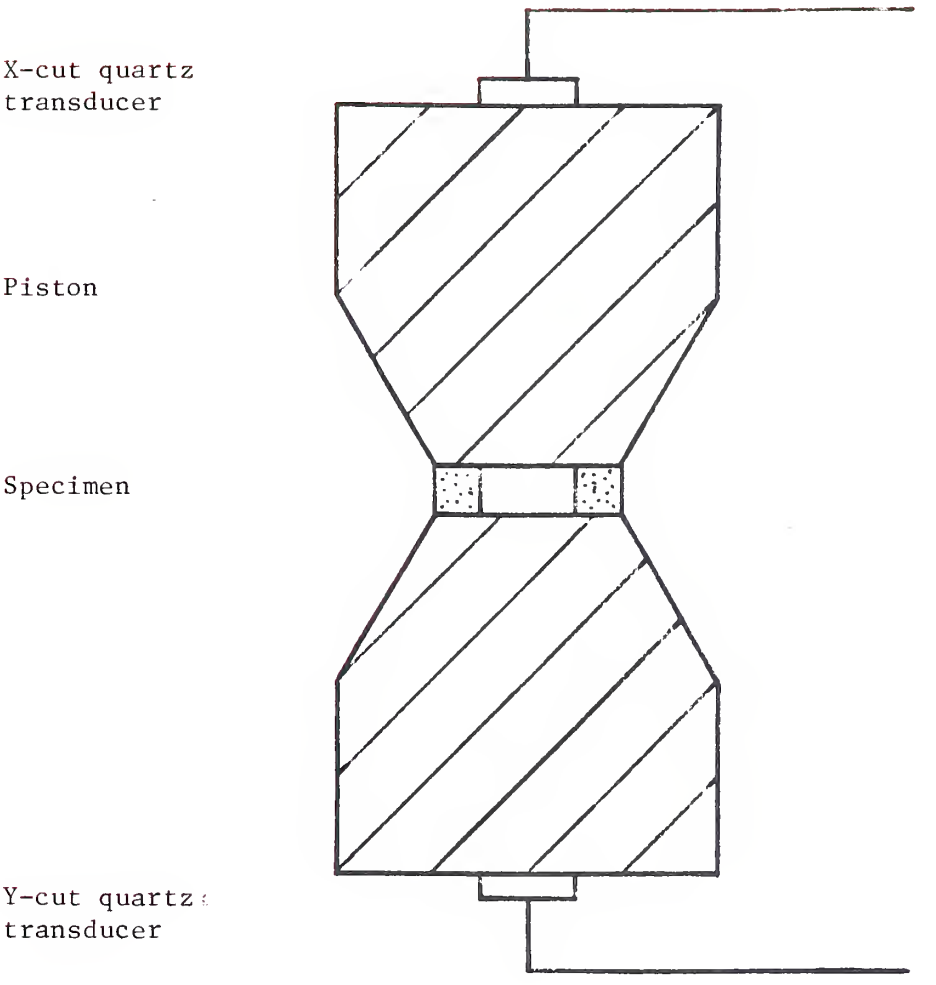


Fig. 9. Diagram of transducer, piston and specimen arrangement for determination of moduli under high pressure (13)