Dynamic Strength Testing of Adhesives and Piezoceramics.



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U. S. Navy Underwater Sound Laboratory
Fort Trumbull, New London, Connecticut

# DYNAMIC STRENGTH TESTING OF ADHESIVES AND PIEZOCERAMICS*

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## INTRODUCTION

The power which an electroacoustical transducer can deliver is often limited by the mechanical strength of the vibrating structure. With transducers of the electrostrictive ceramic type tensile failure may occur either in the ceramic itself or in the adhesive bonds which are usually a necessary part of the transducer structure. Dependence on these vulnerable links is sometimes circumvented by application of a bias force to the structure so that excursions into tension are reduced or eliminated, but such schemes are not feasible for all designs.

The present investigation was aimed at determining the inherent strength exhibited by barium titanate metal structures which are executing extensional vibrations. Little prior data existed on the strength of such sandwich structures, and the applicability of data obtained by static tests was questionable. The present tests employed alternating stress, but they did not include fatigue investigations. The stress was raised to the destruction level in a period of a few minutes.

Figure 1 shows a steel bar resonator used for the testing of ceramic disks. It operates at the lowest longitudinal resonance, which is 2.5 KC. This frequency was selected for all of the tests since it is in the middle of the range in which metal ceramic composite resonators are most extensively used. The ceramic disk is cemented in place at the center of the bar, where the stress is maximum. An ac voltage is applied to the disk to set the bar into vibration, and the voltage is slowly raised until failure occurs.

A capacitive vibration pickup is set up at the end of the bar. A small [illegible] separates the stationary capacitor plate from the end of the bar and the gap spacing is measured by a dial gauge. Condenser microphone [illegible] which will be described later, is used to measure the variation [illegible]

[illegible] of the ceramic. The

*[illegible] presented at the 59th Meeting of the Acoustical Society of [illegible]

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bar resonator is at the center of the Figure, and above it is a tubular resonator which is used for testing ceramic rings as they would be applied in a longitudinal vibrator. In the foreground are some ceramic samples, some before and some after testing.

## STRESS MEASUREMENT METHODS

The main problem in this work was to obtain an accurate measurement of the stress at which failure occurred. The most appealing approach for rapid and convenient testing was to measure the displacement at the end of the resonator, using the capacitive pickup previously illustrated, and derive the stress from this data. For a simple half-wave resonator the equation at the top of Figure 3 gives the relation between the stress at the center ($T_0$) and the displacement at the end ($\xi_\ell$). Modifications required of this formula for the composite metal ceramic structure are considered in the Appendix, and they are found to have negligible effect in this experiment.

The circuit of the capacitive pickup is also shown in Figure 3. A shunt condenser ($C_s$) is used in parallel with the vibrating capacitor to minimize the effects of stray capacity and reduce the sensitivity to a convenient value. We adapted this electrostatic transducer to absolute measurements, calculating its sensitivity from the measured gap length, polarizing voltage, and capacities. Repeatability of the displacement measurement was about ± 10%, the principal variation being in the measurement of the gap length. This error could be reduced by setting the gap electrically, and we will adopt this method when we obtain more sophisticated electronics employing an a.c. rather than a d.c. carrier.

For a more direct measurement of stress, a resistance wire strain gauge was mounted on the steel resonator adjacent to the ceramic. The circuit employed is shown at the right of Figure 3. For the bar resonator the results were within 7% of those obtained from the capacitive pickup, but for the tube resonator the strain gauge consistently gave values 1.6 times as great as those from the capacitive pickup. It was concluded that the tube did not act as a simple longitudinal resonator, even though its length to diameter ratio was 8 and its tangential displacement, as measured by a phonograph pickup along its length, did not differ appreciably from a sine wave. The strain gauge was not convenient for routine measurements, but the correction factor which it provided was applied to all data obtained on the tube resonators with the capacitive pickup.

While the two measurement methods described above were the main ones used, a number of other methods were experimented with to check the validity of the results. The displacement of the end of the resonator was checked optically, initially by direct viewing through a microscope and later by optical interference methods using an interferometer. The microscope measurement of displacement confirmed the capacitive pickup results both for the tube and the bar resonators, though the estimated accuracy obtainable with the microscope was not very high. The interferometer was used with the bar resonator as shown in Figure 4. An accelerometer was added to the end of the bar to monitor the vibration amplitude, and the mirror for the interferometer was temporarily affixed to the top of the accelerometer. When the capacitive pickup was again used, the mirror was removed and a correction for its mass was applied to the accelerometer calibration. The interferometer results agreed with the capacitive pickup results within the latter's accuracy expectations.

The stress at the center of the resonator was checked with a piezoceramic force gauge. This consisted of a thin barium titanate plate which was incorporated in the resonator sandwich between the driving ceramic and the steel. The ceramic plate was 1/8" thick and was annular shaped for the tube resonator and circular for the bar resonator. The electromechanical circuit and sensitivity equation for the force gauge is shown in Figure 5A. The compliance of the ceramic plate may be computed from the material properties with adequate accuracy, and the other parameters of the sensitivity equation can be measured. The electromechanical ratio $N_1$ was determined statically by subjecting the resonator to longitudinal pressure in a hydraulic press and measuring the voltage developed across a large storage condenser with an electrometer when the pressure was released. The calibration of the resistance strain gauge was also checked in this setup. An independent estimate of $N_1$ was derived from dynamic measurements of the $d_{31}$ piezoelectric constant of the plate; since the relation $d_{33}/d_{31} = 2.69$ holds fairly well for the ceramic employed irrespective of its degree of polarization, $d_{33}$ could be estimated, and from this value and the dimensions of the plate $N_1$ could be calculated.

When an average was taken of the static $N_1$ and the dynamic $N_1$ to obtain the force gauge constant, the resulting measurements of stress agreed with the resistance strain gauge results within 10% for both the tube and bar resonators. These various checks gave confidence to the belief that both stress and displacement were being measured accurately and that the failure of the stress and displacement data to jibe in the case of the tube resonator was due to some unknown feature of the mode of vibration.

Another method which was tried was to calculate the stress from the voltage applied to the resonator, using its measured equivalent circuit parameters. The electromechanical circuit and the equation for stress are given in Figure 5B. This method was not considered desirable for general use because of its indirectness and because any non-linear characteristics of the equivalent circuit parameters would lead to incorrect results at high stress levels. The possibility of non-linear damping or of non-linear joint compliance certainly existed, but for checking the other methods it was sufficient to run this experiment at low stress levels, not greatly above the levels at which the equivalent circuit parameters were determined. The stress determined by this method was in fair agreement (20% or better) with that determined by the capacitive pickup method for both the tube and the rod resonator. Thus, this method failed to detect the large error which existed in the capacitive pickup determination of stress in the tube resonator, as revealed by the strain gauge measurement. This was to be expected since both the capacitive pickup method and the equivalent circuit method are based on the assumption that the mode of vibration conforms to the simple theoretical mode.

## RESULTS OF STRENGTH TESTS

In the test program to date we have driven these resonators to failure about 80 times. The cleaning procedure prior to bonding in this initial work was rather rudimentary: The ceramic pieces were scrubbed in acetone for about two minutes and the metal surfaces were wiped with acetone. The pressure applied during bonding was just that due to gravity, with the resonators standing vertically.

The failures were classified into three main categories: Ceramic breaks, bond breaks, and mixed bond and ceramic breaks. Figure 6 shows the data on the ceramic breaks, encompassing three different types of samples. The rings were 1/2" wide by 1/2" thick by 3-3/8" diameter and were fabricated at the Underwater Sound Laboratory. The disks were 1-1/8" in diameter, and the 1/2" disks were fabricated at USL, while the 1/4" disks were of commercial manufacture. The rings were cemented to common steel, while the disks were cemented to stainless steel. The data in the fourth column of the Table, labeled Epoxy I Bare, was obtained on rings which were electroded for radial field excitation; this permitted their bonding surfaces to be free of silver. For all other samples bonding was done to the silvered surfaces of the ceramic.

The cement listed in the last column is a proprietary cyanoacrylate monomer which is very fast setting and produces thin bonds. Bond thicknesses in the 2 to 6 mil range were obtained with the epoxy cements, except that the data in column 3, labeled thick joint, is for a cement layer 1/16" thick. This large thickness was obtained by using a few small spacer blocks of cured epoxy in the joint. Since ceramic polarized in silicone oil had had a shop reputation of being difficult to cement, we included samples of this type (columns 2 and 6). During cleaning these samples were soaked in acetone for a longer period than the others.

Since all of the data in Figure 6 are for pure ceramic breaks, the variables of the cementing procedures are not really relevant, except the curing temperature which may cause prestressing of the ceramic. The epoxy cements were cured in the temperature range 65-75° C. The cyanoacrylate cement, on the other hand, is a room temperature curing adhesive, and the fact that a number of ceramic breaks were obtained with this cement suggests that the curing temperatures used were not a dominant factor in limiting ceramic strength. The data reveals no pronounced differences in strength between rings and disks between 1/2" and 1/4" disks, or between USL and commercial ceramic. It also indicates that polarization in silicone oil or use of unusually thick joints does not necessarily preclude achievement of bond strength as great as the average ceramic strength.

Figure 7 presents data on bond breakage, where the ceramic emerged unscathed. The cyanoacrylate cement appeared to suffer cohesive failure, that is, a thin layer remained on each surface after cleavage. With the epoxies, on the other hand, failure of adhesion at the ceramic surface occurred. It is presumed that this type of failure could be lessened by improved cleaning procedures and future work will proceed in that direction. In a few cases poor adhesion of the silver electrodes contributed to these breaks; in fact, some electrode failures were experienced at stresses well below 1000 p.s.i., but these samples were discarded as obviously defective. Silver adhesion was not commonly the weak link in the joint; Figure 6 indicates the ability of the electrodes to hold when the ceramic could not survive.

The data of Figure 8 is for breaks where a substantial part of the ceramic broke away and remained with the metal member. Part of the break was along the glue line; so improved cleaning might reduce this type of casualty.

An epoxy cement which cured at 150° C was tried briefly, since its reported strength is greater than that of the epoxies which cure at lower temperatures. To avoid cooling the ceramic through its Curie point (where a large strain occurs) lead titanate zirconate rather than barium titanate was used, and the samples were of the ring type. The thermal stresses proved excessive, with the ceramic fracturing before the alternating stress could be raised to a measurable level. Next invar buffer rings 1/2" thick were welded to the steel tubes, and the PZT ceramic was cemented to the invar surfaces. This effort at alleviating the thermal stresses proved insufficient, and the ceramic again broke at low stress.

In another experiment, an attempt was made to eliminate thermal stresses, while using the Epoxy I cement, by employing a room temperature cure of eight days duration. Two ring samples were used and the failures occurred at 2400 and 3300 p.s.i. However, the failures were of the bond break type (Figure 7); so this experiment was inconclusive.

## CONCLUSIONS

Summarizing, we note that the average ceramic strength in these tests was about 2400 p.s.i. This is only about one-fifth that of an early published value for barium titanate, which was derived from static bending tests.* Since power is proportional to the square of the allowable stress, this difference has great significance in transducer design. The low strength of the ceramic emphasizes the importance of including mechanical bias force in transducer designs whenever feasible. Improvements in ceramic fabrication techniques are needed which will yield a stronger material. At present improved ceramic strength is more vitally needed than improved adhesive strength.

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Physicist

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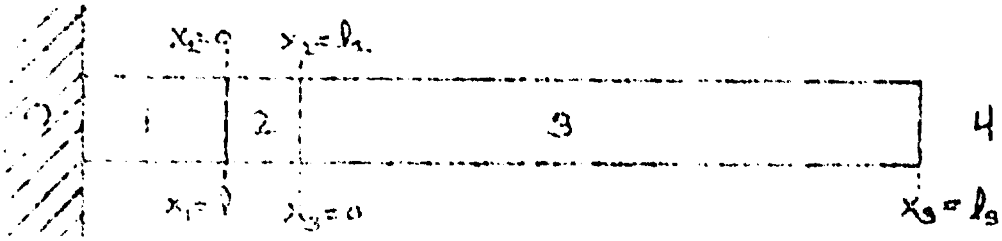
Physicist

*This strength value is given in the Clevite Corporation publication "Piezo-[illegible]". The information on the method of test was obtained [illegible] of Clevite Corporation.

# APPENDIX

The equation relating stress and displacement given in Figure 3 is for a resonator of homogeneous material. Actually the resonator contains in addition to the metal a layer of ceramic and two thin layers of cement. These layers are so thin compared with the total length of the resonator that the simple equation of Figure 3 continues to apply quite well to this composite resonator. This conclusion requires verification, however; so the more complicated equations which do not neglect the effect of the different layers are presented and illustrated below.

Since the resonator is symmetrical about its center it is sufficient to consider only the half of the resonator to the right of the nodal plane, as shown below.




Assuming plane wave propagation in the bar, the basic equations required are:

Displacement $\xi(x) = 2\xi_+ \cos(kx-\delta)$ $\quad k = \omega/c$

Stress $T(x) = -\omega\rho c\, 2\xi_+ \sin(kx-\delta)$

Impedance $Z(x) = \dfrac{TA}{j\omega\xi} = j\rho c A \tan(kx-\delta)$

where $\xi_+$ is the complex amplitude of the traveling wave going to the right, and $\delta$ is related to the traveling wave amplitudes by the equation: $e^{j2\delta} = \xi_-/\xi_+$. The three variables given by the equations are continuous across the boundaries of the sections of the bar. In applying these equations each section of the bar will have its own coordinate system, with $x = 0$ at the left hand end of the section.

Since $\xi_1 = 0$ at the node we have from the displacement equation: $\delta_1 = \pi/2$. Since $T = 0$ at the free end we have from the stress equation: $k_3 l_3 = \delta_3$. From the impedance equation we obtain equations giving $\delta_2$ and $\delta_3$:

$$[illegible] \quad \frac{\rho_1 c_1}{\rho_2 c_2} \tan(k_1 l_1 - \pi/2)$$

$$[illegible] \quad \frac{\rho_2 c_2}{\rho_3 c_3} \tan(k_2 l_2 - \delta_2)$$

The stress and displacement equations may be used to derive the following relations for the stress at the various boundaries in terms of the displacement at the free end, $\xi_{34}$ :

$$T_{01} = \frac{\omega \rho_1 c_1 \cos \gamma_2 \cos \gamma_3}{\cos(k_1 l_1 - \pi/2) \cos(k_2 l_2 - \gamma_2)} \xi_{34}$$

$$T_{12} = \frac{\omega \rho_2 c_2 \sin \gamma_2 \cos \gamma_3}{\cos(k_2 l_2 - \gamma_2)} \xi_{34}$$

$$T_{23} = \omega \rho_3 c_3 \sin \gamma_3 \xi_{34}$$

The material properties required are:

| | | |
|---|---|---|
| Barium Titanate : (Calcium additive) | C = 4500 m/s | $\rho c$ = 2.25x10[7] $\frac{kg}{m^2\text{-sec.}}$ |
| Stainless Steel : (Type 304) | C = 4930 m/s | $\rho c$ = 3.91x10[7] $\frac{kg}{m^2\text{-sec.}}$ |

We will consider first a vanishingly thin bond and will determine the effect of a 1/2" thick ceramic piece on the stress-displacement relation for the bar resonator. The resonant frequency for very thin bonds (and 1/2" samples) was found to be 3494 c.p.s. Applying the equations above to this case we have:

$$l_2 = 0 \qquad l_1 = 1/2'' \qquad k_1 l_1 = .0310$$

$$\tan \gamma_2 = \frac{\rho_1 c_1}{\rho_2 c_2} \tan 1.540 = 32.5 \frac{\rho_1 c_1}{\rho_2 c_2}$$

$$\tan \gamma_3 = \frac{\rho_1 c_1}{\rho_3 c_3} \tan \gamma_1 = 32.5 \frac{\rho_1 c_1}{\rho_3 c_3} = 18.70 \qquad \gamma_3 = 1.517$$

$$T_{01} = \frac{\cos \gamma_3}{\cos(k_1 l_1 - \pi/2)} \omega \rho_1 c_1 \xi_{34} = 1.740 \omega \rho_1 c_1 \xi_{34} = 1.000 \omega \rho_3 c_3 \xi_{34}$$

$$T_{12} = T_{23} = \sin \gamma_3 \omega \rho_3 c_3 \xi_{34} = .999 \omega \rho_3 c_3 \xi_{34}$$

It is seen that the stress-displacement relations obtained by this more exact method do not differ appreciably from the simple relation of Figure 3.

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Next we consider the case where the adhesive layer was intentionally made thick. The ceramic disk in this experiment was 1/4" thick and the adhesive layer of Epoxy I was 1/16" thick. The resonant frequency was 3280 c.p.s., while for the same type of resonator with a very thin bond it was 3563 c.p.s. The density of a block of cured Epoxy I was measured and found to be 1710 $kg/m^3$.

The first step is to find the sound velocity of Epoxy I. The thin-bond data provides a value for $\delta_3$:

$$\tan\delta_3 = -\frac{\rho_1 c_1}{\rho_3 c_3}\tan(k_1 l_1 - \pi/2) = .576 \tan 1.555$$

$$\delta_3 = 1.543 = k_3 l_3 \qquad @\ 3563\ \text{c.p.s.}$$

$$@\ 3280\ \text{c.p.s.} \qquad \delta_3 = k_3 l_3 = \frac{3280}{3563}\times 1.543 = 1.421$$

For the thick-bond case we have:

$$\tan\delta_2 = \frac{\rho_1 c_1}{\rho_2 c_2}\tan 1.556 = 68\,\frac{\rho_1 c_1}{\rho_2 c_2}$$

$$\tan\delta_3 = 6.63 = -\frac{\rho_2 c_2}{\rho_3 c_3}\tan(k_2 l_2 - \delta_2)$$

Combining the two equations above we obtain an equation for determining $C_2$:

$$\frac{\tan\frac{\omega}{c_2} l_2 - 68\frac{\rho_1 c_1}{\rho_2 c_2}}{1 + 68\frac{\rho_1 c_1}{\rho_2 c_2}\tan\frac{\omega}{c_2} l_2} = -6.63\,\frac{\rho_3 c_3}{\rho_2 c_2}$$

whence:

$$\tan\frac{\omega}{c_2} l_2 = \frac{-6.63\frac{\rho_3 c_3}{\rho_2 c_2} + 68\frac{\rho_1 c_1}{\rho_2 c_2}}{1 + 450\frac{\rho_1 c_1 \rho_3 c_3}{(\rho_2 c_2)^2}} = \frac{-\frac{[illegible]\times 10^5}{c_2} + \frac{8.95\times 10^5}{c_2}}{1 + \frac{1.356\times 10^{11}}{c_2^2}}$$

or

$$[illegible] \quad .346\times 10^{-6} c_2 \quad \frac{1.323\times 10^5}{c_2}$$

The result is $C_2 = 2440$ m/s.

Before calculating the stresses we recheck the values of the required parameters and obtain:

$$K_1 l_1 = .0148 \qquad K_2 l_2 = .0134 \qquad \rho_2 c_2 = .417\times10^7$$
$$\tan\delta_2 = 365 \qquad \delta_2 = 1.568 \qquad \tan\delta_3 = 6.75 \qquad \delta_3 = 1.424$$

Then the stress equations are:

$$T_{01} = \omega\rho_1 c_1 \xi_{34} \frac{\cos\delta_3}{\sin K_1 l_1 \left[\cos K_2 l_2 + \sin K_2 l_2 \tan\delta_2\right]}$$

$$= 1.679\,\omega\rho_1 c_1 \xi_{34} = .966\,\omega\rho_3 c_3 \xi_{34}$$

$$T_{12} = \omega\rho_2 c_2 \xi_{34} \frac{\cos\delta_3}{\cos K_2 l_2 \cot\delta_2 + \sin K_2 l_2}$$

$$= 9.06\,\omega\rho_2 c_2 \xi_{34} = .966\,\omega\rho_3 c_3 \xi_{34}$$

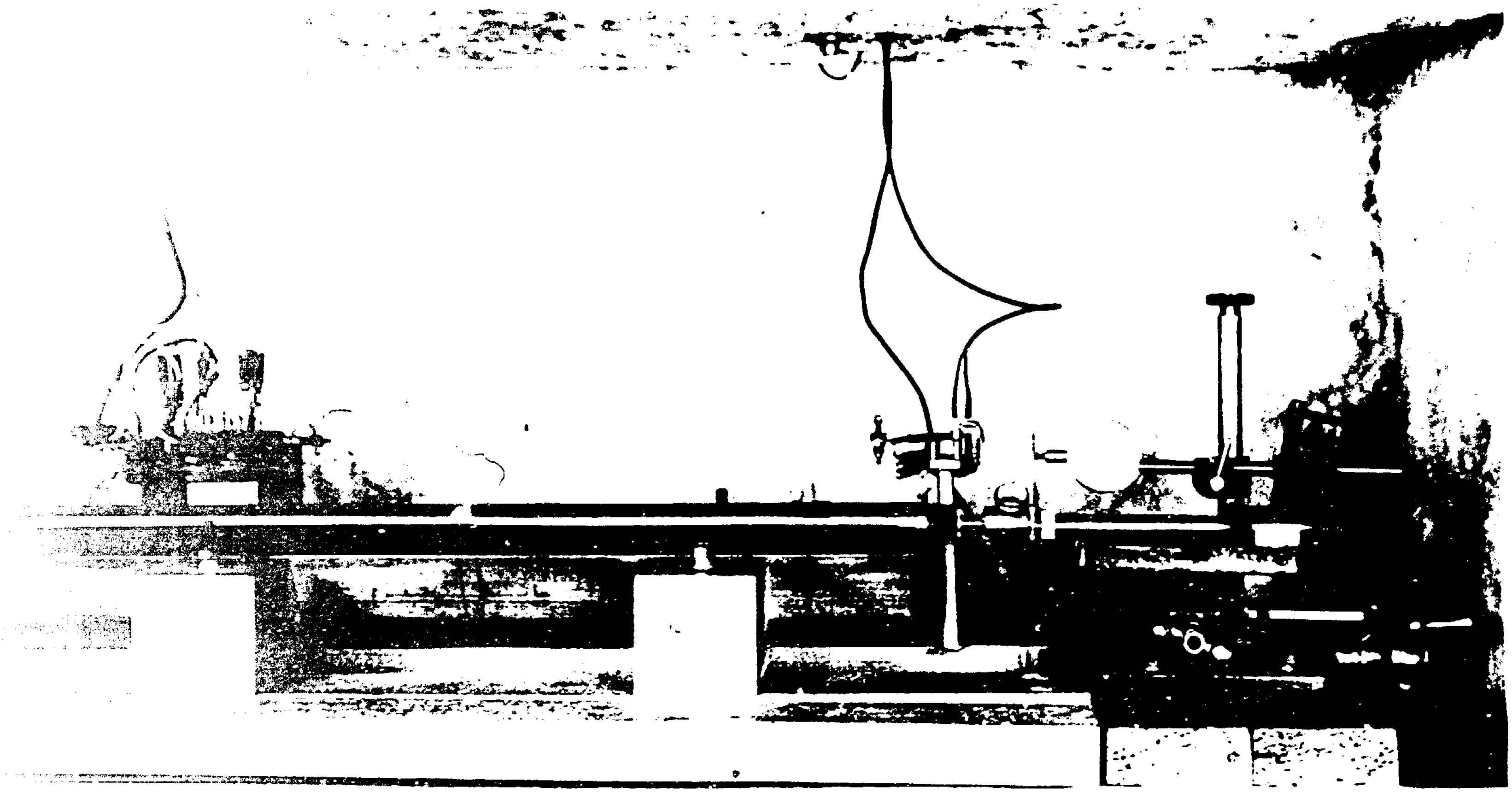
$$T_{23} = .89\,\omega\rho_3 c_3 \xi_{34}$$

While the 1/16" thick bond caused a very noticeable change in resonant frequency, it caused the stress to differ but 4% from that given by the simple relation of Figure 3.

## APPENDIX

Identification of Adhesives:

Cyanoacrylate Monomer ..... Eastman 910
Epoxy I .................. Armstrong A2 with Activator C
Epoxy II ................. Epon VI
Epoxy III ................ Epon 828
"Epoxy cured at 150° C" .. Araldite AN-100



Bar Resonator With Capacitive Displacement Transducer

USL Tech Memo No. 1150-34-60

U. S. Navy Underwater Sound Laboratory
NP 24 - 18944 - 5 - 60

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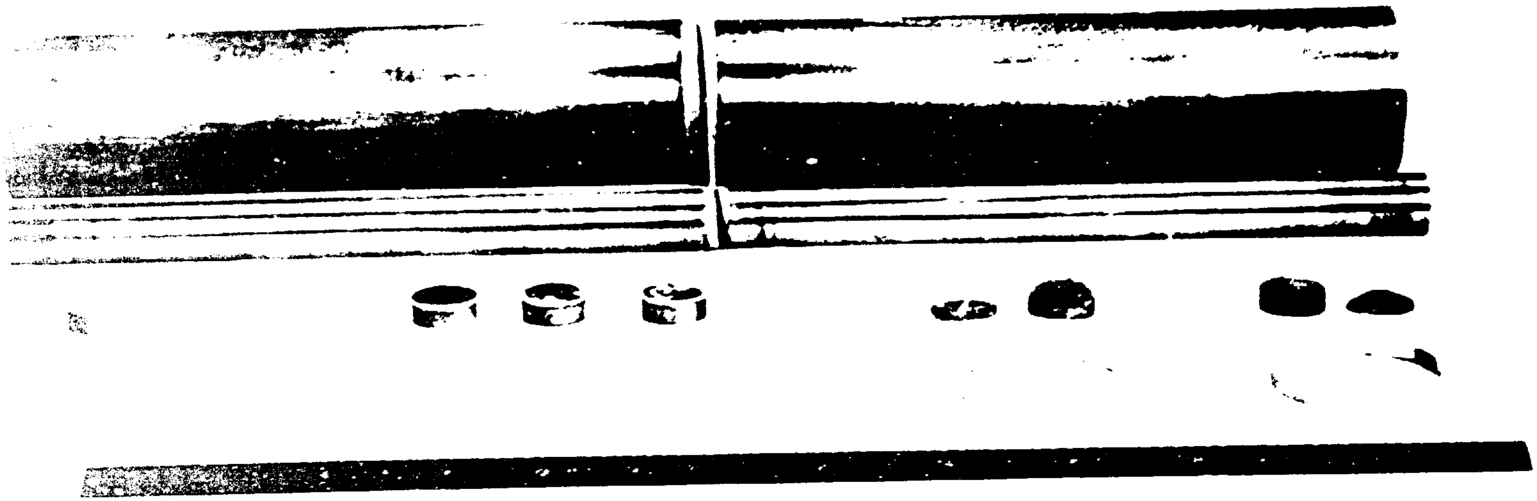
Figure 1



Tube and Bar Resonators After Ceramic Fracture and Ceramic Specimens

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U. S. Navy Underwater Sound Laboratory
NP24 - 18945 - 5 - 60

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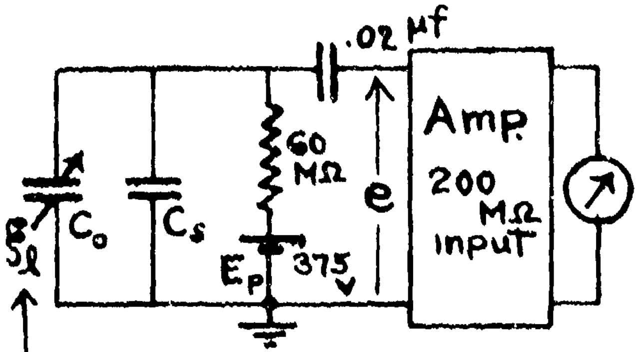
FIGURE 2

## STRESS-DISPLACEMENT RELATION

$$T_o = \rho c \omega_r \xi_\ell$$

$\omega_r$ = angular resonant freq.
$\rho c$ = spec. acous. impedance of metal

### CAPACITIVE DISPLACEMENT TRANSDUCER

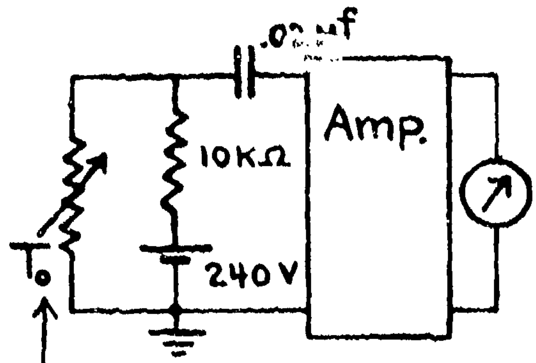



vibrating plate capacitor with gap $X_o$

displacement:

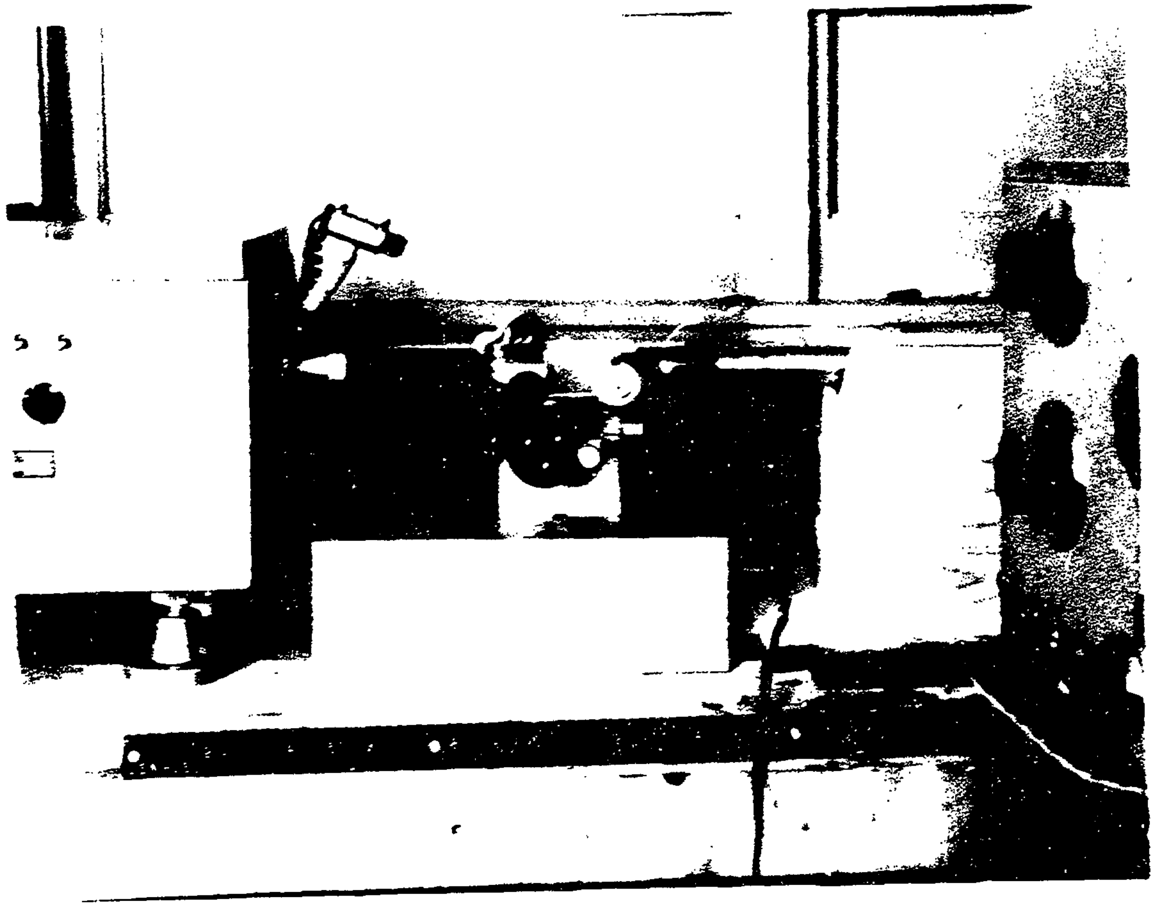
$$\xi_\ell = \frac{X_o}{E_p} \frac{C_o + C_s}{C_o} e$$

### STRAIN GUAGE CIRCUIT




strain guage SR-4 500 Ω

FIGURE 3



Calibration of the Resonator with an Optical Interferometer

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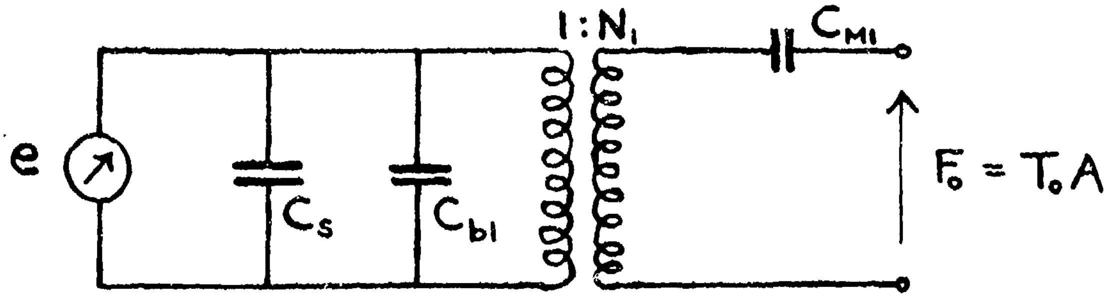
Figure 4




$$T_0 = \frac{C_s + C_{b1} + N_1^2 C_{M1}}{N_1 C_{M1}} \; \frac{e}{A}$$

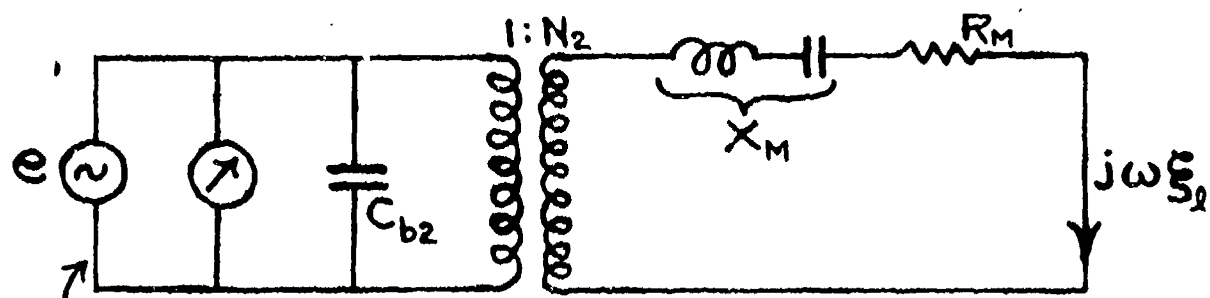
A = area of ceramic plate

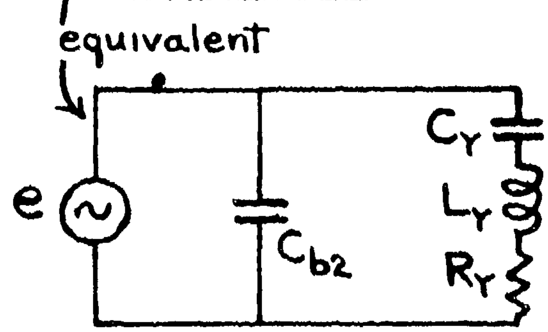
$l_1$ = thickness of ceramic plate

$$N_1 = \frac{d_{33} A}{s_{33}^{E} l_1} = \frac{(C_s + C_{b1})\,e}{C_{M1} F_0}$$

$$C_s + C_{b1} \gg N_1^2 C_{M1}$$

A. PIEZOELECTRIC FORCE GUAGE




$$T_0 = e_c \frac{N_2}{R_M} e \qquad X_M = 0$$

$$N_2 = \sqrt{\frac{\rho A l_2}{2 L_Y}}$$

$$R_M = N_2^2 R_Y = \frac{\omega_r \rho A l_2}{2 Q_M}$$

$l_2$ = length of resonator

B. ELECTROMECHANICAL CIRCUIT OF RESONATOR

FIGURE 5

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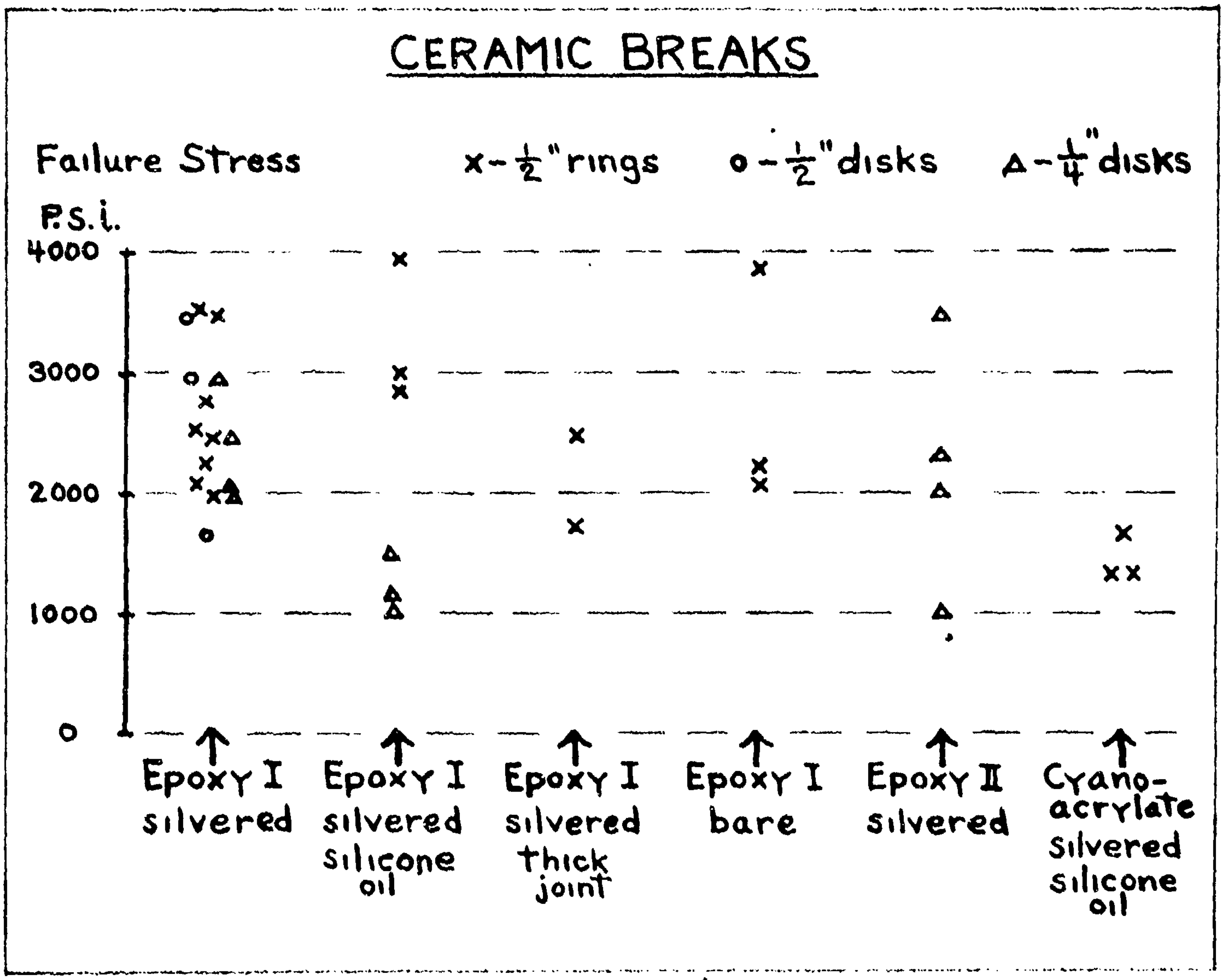


FIGURE 6

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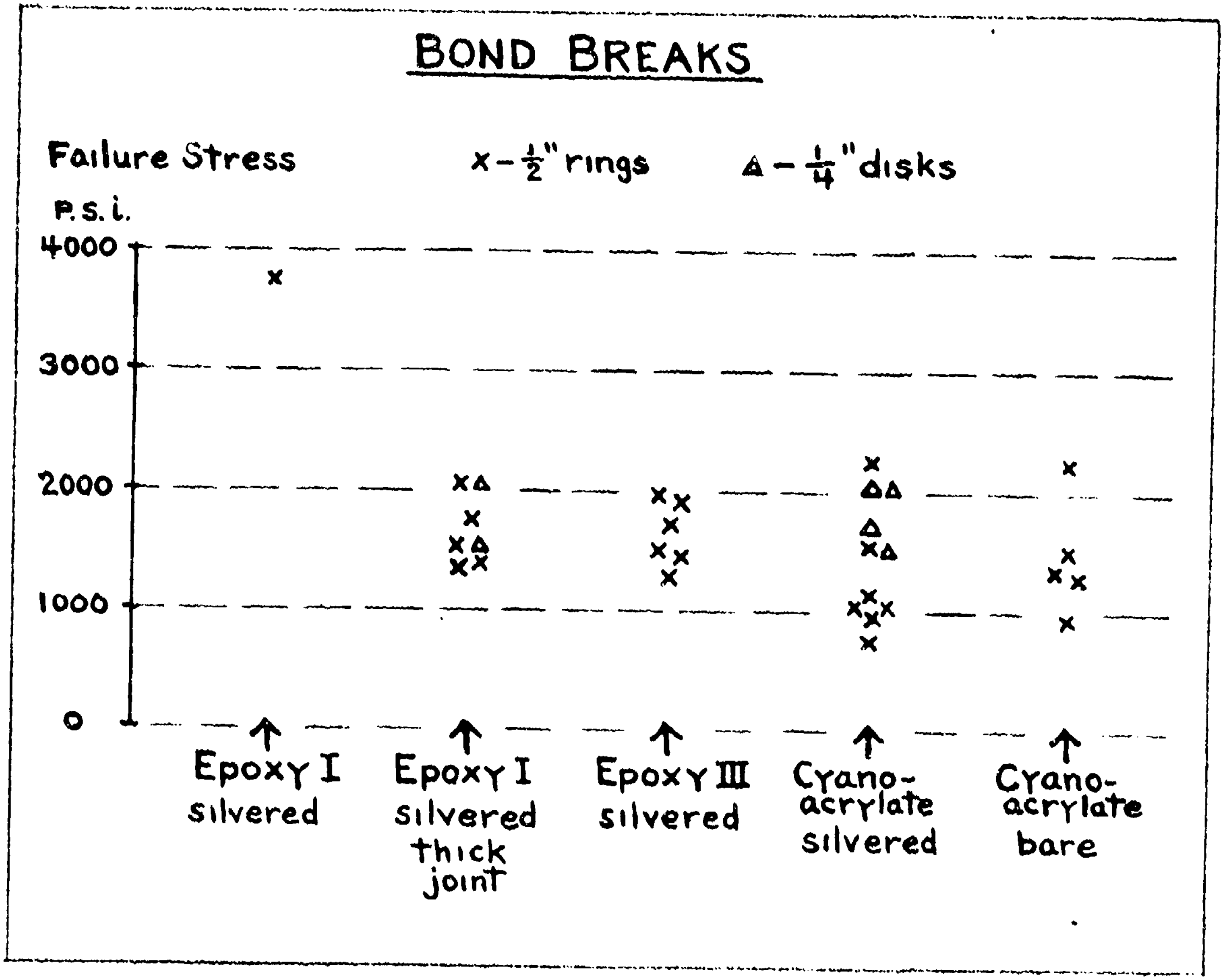
BOND BREAKS
Failure Stress
P.S.I.
x – ½" rings
Δ – ¼" disks
4000
3000
2000
1000
0
Epoxy I silvered
Epoxy I silvered thick joint
Epoxy III silvered
Cyano-acrylate silvered
Cyano-acrylate bare

FIGURE 7

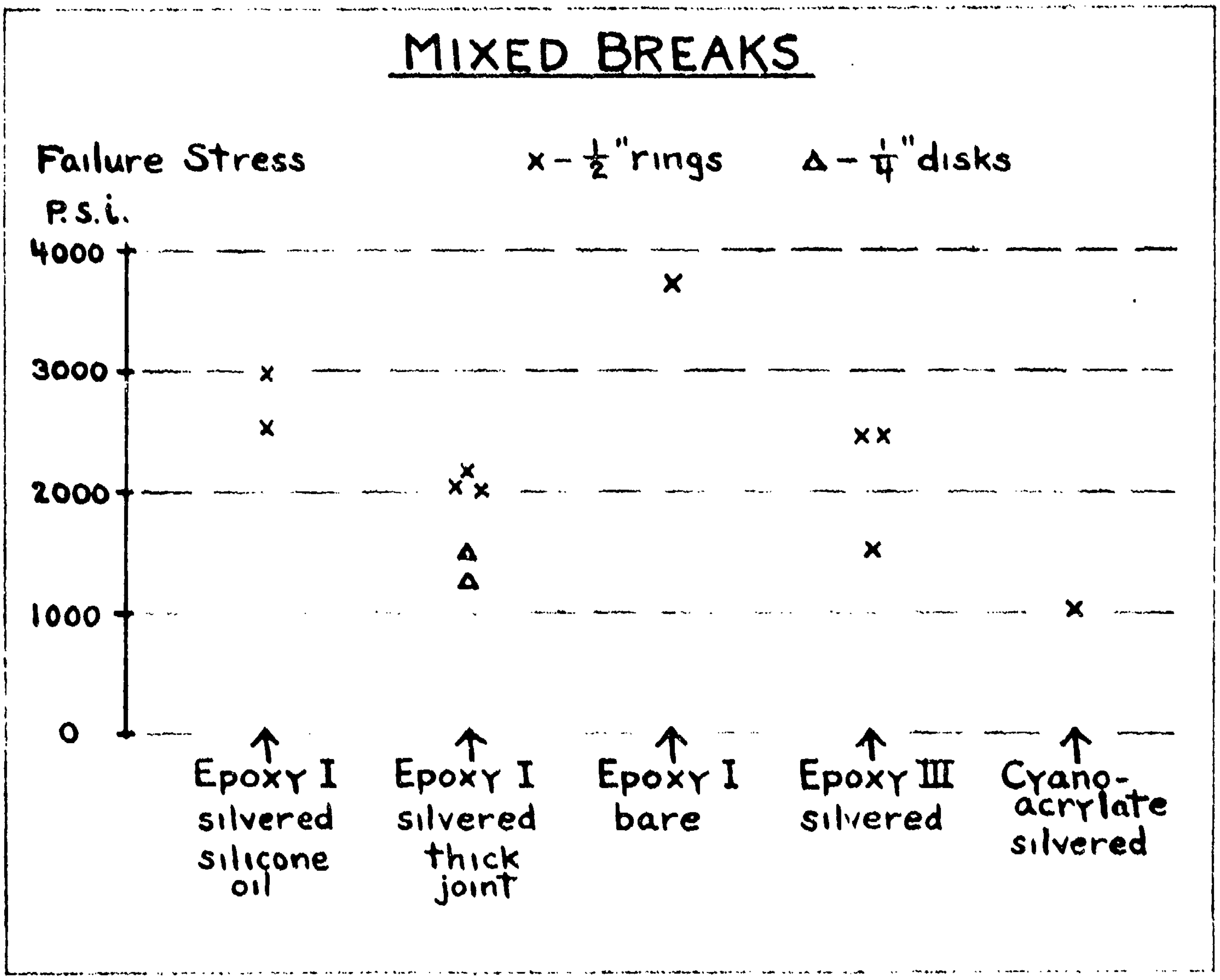


FIGURE 8

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