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Dynamic Measurement of Bulk Modulus of Dielectric Materials Using a Microwave Phase Shift Technique

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PREFACE

The work described in this report was performed by the Propulsion Division of the Jet Propulsion L.boratory. The investigation of the subject was suggested by Mr. W. L. Dowler.

Mr. B. J. Barker performed this work while he was a summer employee of the Jet Propulsion Laboratory. He is presently at Stanford University.

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ABSTRACT

A microwave Doppler shift technique has been developed for
measuring the dynamic bulk modulus of dielectric materials such as
solid propellants. The system has a demonstrated time resolution on
the order of milliseconds and a theoretical spatial resolution of a
few microns. Accuracy of the technique is dependent on an accurate
knowledge of the wavelength of the microwave in the sample being tested.
Such measurement techniques are discussed. Preliminary tests with
two solid propellants, one non-aluminized and one containing 16%
aluminum, yielded reasonable, reproducible results. It was concluded
that, with refinements, the technique holds promise as a practical
means for obtaining accurate dynamic bulk modulus data over a variety
of transient conditions.

I. INTRODUCTION

The bulk modulus of solid propellants and other polymeric materials is presently measured with devices such as a dilatometer[1], which require relatively large material displacements (large pressure differentials) and a number of tests in order to obtain an accurate measurement. A new microwave technique for experimentally determining the bulk modulus of dielectric materials (solid propellants in this instance) has been explored, with highly encouraging results. Included in this report are a description of the technique, the theoretical basis for interpretation of the data, an estimation of the potential accuracy of the measurements, and a discussion of the preliminary results.

II. THEORETICAL RELATIONSHIP OF BULK MODULUS TO LINEAR STRAINS

The bulk modulus of classical elastic materials is a measure of hydrostatic compressibility defined as

$$K = \frac{E}{3(1-2\nu)} = V_0 \frac{\Delta P}{\Delta V} \tag{1}$$

where K = bulk modulus

E = Young's Modulus

ν = Poisson's Ratio

V_0 = Initial volume

and ΔV = Incremental change in volume for an incremental change in hydrostatic pressure, ΔP.

For visco-elastic materials this has meaning only in the instantaneous sense where

$$K = V_0 \frac{dP}{dV} \tag{2}$$

For an arbitrary cube with sides of some small length ℓ_0, the bulk modulus can be related to linear strain in the following manner:

$$V_0 = \ell_0{}^3 \tag{3}$$

$$V = V_0 + dV = (\ell_0 + d\ell)^3$$
$$= \ell_0{}^3 + 3\ell_0{}^2 + 3\ell_0(d\ell)^2 + (d\ell)^3$$

Neglecting second and third order terms we have

$$dV = 3\ell_0{}^2 d\ell \tag{4}$$

Substituting (3) and (4) into (2) yields

$$K = \ell_0{}^3 \frac{dP}{3 \ell_0{}^2 d\ell}$$

$$= \frac{\ell_0}{3} \frac{dP}{d\ell} \tag{5}$$

which can be written

$$K = \frac{\ell_0}{3} \frac{dP/dt}{d\ell/dt} \quad \text{where } t = \text{time} \tag{6}$$

This relationship holds true for rectangular and cylindrical elements subject to the assumption that strains $d\ell/\ell_0$ are equal in each direction. This assumption would appear justified for homogeneous propellants.

III. EXPERIMENTAL APPARATUS AND TECHNIQUE

A microwave technique for measuring the rate of regression of a solid propellant was described in detail in Ref. 2. Much of the same test apparatus was used in these experiments. A block diagram of the basic test apparatus is shown in Fig. 1. A klystron, operating at 10^{10} Hz and stabilized to one part in 10^{5} Hz, propagates a signal into a standard X-band waveguide. A 1% directional coupler picks up a reference signal, and the remainder of the signal travels to a cylindrical test sample, which acts as an unconfined a dielectric waveguide (Fig. 2). A portion of the propagated wave is reflected from the propellant upper surface, and another 1% coupler senses the reflected signal. The phase difference, ϕ, between the incident (reference) and reflected (test) waves is a function of their respective path lengths. By monitoring and recording this phase difference with a microwave network analyzer, a continuous measurement of any variation in length of the test material is obtained. Consequently, $d\ell/dt$ is directly proportional to the measured $d\phi/dt$.

A dielectric transition piece (Fig. 2) is used to attempt to minimize the discontinuity as the microwave propagates from the air-filled waveguide, where the wavelength $\lambda = 3cm$, into the 2.5-cm diameter unshielded-propellant samples, where λ equalled approximately 1.3 and 0.3 cm for two JPL composite test propellants (JPL 540A and 540K), containing no metal and 16% Al respectively. In spite of this the test signal into the network analyzer contains a spurious reflection from the lower surface of the propellant. In order to eliminate

this secondary signal a slide screw tuner (Fig. 1) and tuning propellant sample are used. A long tube of propellant like the sample is placed in contact with the upper surface, as shown in Fig. 3. Due to the lossy nature of the propellant, this tuning tube absorbs essentially all of the microwave energy entering the propellant sample. Since the incident signal is completely absorbed, the test signal should be composed solely of fixed reflections, which are then minimized with the slide screw tuner. Typically, the fixed reflections can be tuned to a level 75dB or more below the reference signal amplitude.

The pressure chamber consists of a nylon cylinder bolted to the microwave base plate using a lucite head plate, as shown in Fig. 2. The nylon and lucite construction avoids formation of a microwave resonator, which could introduce further spurious components into the test signal. The chamber is enclosed within an Instron temperature control box, to allow the test ambient temperature to be controlled. The complete test system is shown in Fig. 4.

Nitrogen gas is used as the working fluid in the chamber. In a majority of the experiments the chamber pressure transient was produced simply by rapidly opening or closing a Grove pressure regulator valve. More rapid depressurizations were obtained by opening a toggle valve on the vent line.

Chamber pressure is monitored by a standard Taber gauge. The P and ϕ test signals were amplified and recorded on an oscillograph (Fig. 5). Their time differentials were obtained from the test record using simple graphical means, allowing K to be calculated using Eqn. 6. Past experience (Reference 2) indicates that it should be possible to get direct readings of K by differentiating both the ϕ and P signals electronically. This was not attempted in these preliminary tests.

IV. POTENTIAL SYSTEM ACCURACY

The two outstanding features of the microwave system for measurement of propellant properties are its high time and spatial resolution, and its non-interference with the sample being tested.

The only physical contact between the sample and the test stand is an area of about 5 cm^2. Since the samples weigh on the order of 20 grams, the frictional forces acting as a restraint on the lower surface would appear negligible.

The system has demonstrated time resolution on the order of milliseconds in transient burning rate tests, and its spatial resolution can easily be calculated.

Since a $\Delta \ell$ of $\lambda/2$ produces $\Delta \phi = 360$ deg, $d\ell/d\phi = \lambda/720$ deg. If $\lambda = 1.3$ cm, then $d\ell/d\phi = 18$ μm/deg. The wave analyzer/phase meter combination in use at JPL will accurately measure differences of better than 0.2 deg, giving a theoretical resolution of less than 4 μm.

Typical strains can be calculated using the gross assumption of elasticity. If $K = 1.4 \times 10^5$ N/cm^2, $\ell_0 = 5$ cm, and $\Delta P = 210$ N/cm^2, then $\Delta \ell = 75$ μm. This sort of value would be reached as $t \rightarrow \infty$, but within a few seconds, changes in length of about 30-40 μm should be observed. The resolution vs. deflection situation is even more promising for aluminized propellants. A calculated dielectric constant $\epsilon = 95$ would produce $\lambda = 0.25$ cm and $d\ell/d\phi = 3.5$ μm/deg. Resolution in this case would be better than 1 μm, but surface imperfections are already on the order of several microns. It is worth noting that λ is much larger than the surface roughness, and the microwave system thus automatically takes an average value over the surface.

The primary difficulty in obtaining this sort of accuracy is knowledge of λ to several decimal places. If the dielectric constant is known, the wavelength can be calculated, but a more satisfactory procedure exists. A cylinder of propellant can be cast out of each sample batch having the same

section as the test samples. A ring type probe can be used to observe field strengths along the cylinder while the microwave is propagating through it. An average over perhaps 50 cycles in this manner should give the desired accuracy. This technique also should eliminate questions as to variations in electrical properties between batches.

V. PRELIMINARY RESULTS

Several test runs have been made using both JPL 540A and 540K. These tests have shown two things which are most encouraging. First, the sensitivity of $d\phi/dt$ to dP/dt is clearly visible and fairly easy to calculate. Second, the behavior of the propellant appears to be consistent for any given initial conditions.

In test run 18, using a 4.29-cm long sample of 540A, four pressurizations of the test chamber were separated by about 1/2 minute. The rate of pressurization varied from 20.0 to 28.3 N/cm^2-s. Throttling action in the nitrogen supply regulator provided a very nearly linear pressure rise. A mean value of $K=1.54 \times 10^5 \ N/cm^2$ was measured. The total spread was 3.3% (+1.4%, -1.9%) about the mean, which is less than would have been expected to be introduced by the data reduction technique alone. If we take $\nu = 0.500$, then this value of K would imply an initial Young's Modulus of about 460 N/cm^2, which is in the range suggested by Instron data. In this case, the tuning was such that the desired test signal amplitude was greater than the spurious reflections by a factor of 1.9×10^3 (75.5 dB). Since the phase angle of the spurious reflections is not known, only a worst case analysis can be performed. The maximum linear error this could introduce is about 1/2 micron, which would shift the value of K by about 2.5%. This possible error could best be eliminated statistically by repeated tests, or by using greater pressure differentials to reduce the percentage error.

The behavior of JPL 540K is equally consistent and less subject to tuning errors, due to the greater angular change for any linear change. Two pressurizations of a 4.04 cm long sample at $dP/dt = 19.0$ and 20.0 N/cm^2-s produced $K=3.05$ and $2.76 \times 10^5 N/cm^2$ assuming $\lambda = 0.25$ cm. The absolute values for

K are of no particular significance since λ is in fact not accurately known. These are fairly typical runs produced by the existing apparatus and do not represent the capabilities of the system with a number of fairly minor refinements.

Behavior of propellants during depressurization is of equal interest, and can be explored even more easily than pressurization behavior. Attempts were made to explore two aspects of transient behavior.

The simplest four-element viscoelastic model predicts that the instantaneous K will be proportional to dP/dt. The internal damping of the molecular structure will limit the rate of response of the sample, while the rate of change of pressure is arbitrary. This behavior can be observed with the existing facility. During Run 22 (540K, $\ell_0 = 4.04$ cm) dP/dt was increased from -42.3 to -92.0 N/cm^2-s (118% increase) and the indicated K increased from 1.97 to 2.30 X 10^5 N/cm^2 (16% increase). Again, the actual value of K is of no significance.

The viscoelastic model also predicts an instantaneous lag in the response of the propellant to a step change in pressure. No lag was readily observable because of insufficient abruptness of the pressure drop. A burst diaphragm is needed to obtain depressurizations rapid enough to conclusively explore this area.

VI. CONCLUSIONS

The most substantial questions as to the practicality and accuracy of this basic technique for measuring bulk modulus have been answered affirmatively. A number of refinements to the apparatus will improve both the speed and accuracy of the tests. These include: (1) a threaded test chamber that screws onto the microwave base plate, (2) a more mechanized method of holding and positioning the long tube of tuning propellant, and (3) electronic differentiation and division of the test data for direct read-out of the bulk modulus. Refinement

of the system should make it possible to accurately determine the wavelength for each propellant batch cast, and obtain dynamic bulk modulus data over a variety of transient conditions with an accuracy of within 1%.

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1. Mellette, R. V., The Solid Propellant Characterization Required for Grain Structural Analysis, Douglas Report SM-48942, Missile and Space Systems Division, Douglas Aircraft Company, Inc., Santa Monica, California, July 2, 1965.

2. Strand, Leon D., Schultz, August L., and Reedy, Gary K., Determination of Solid Propellant Transient Regression Rates Using a Microwave Doppler Shift Technique, Technical Report 32-1569, Jet Propulsion Laboratory, Pasadena, California, October 15, 1972.
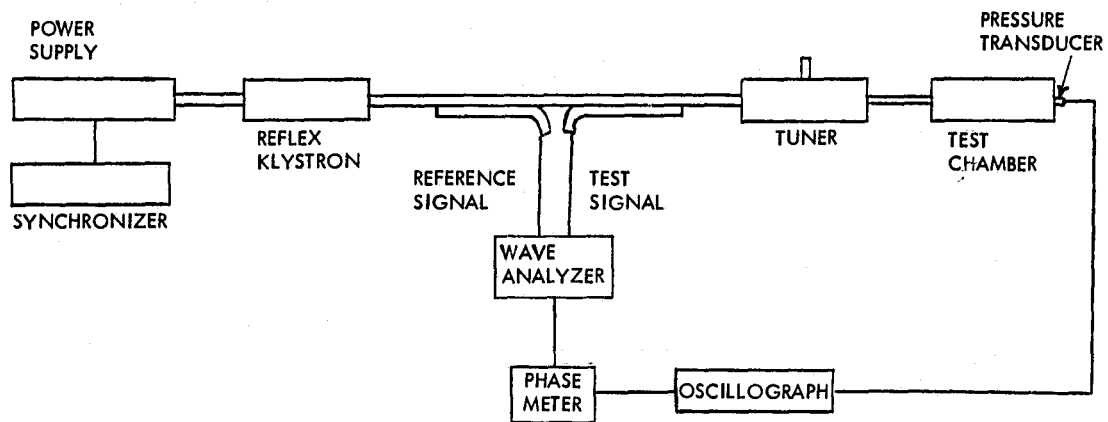
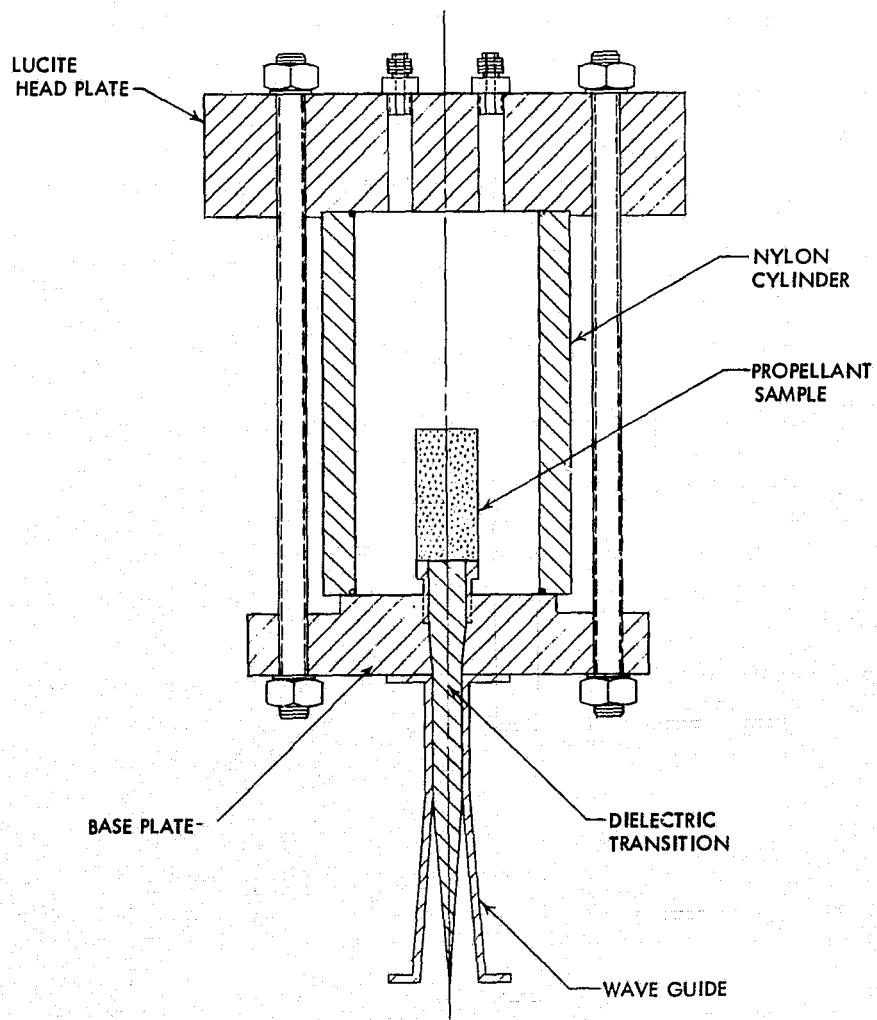Fig. 1. Block Diagram of Microwave Basic Test Apparatus

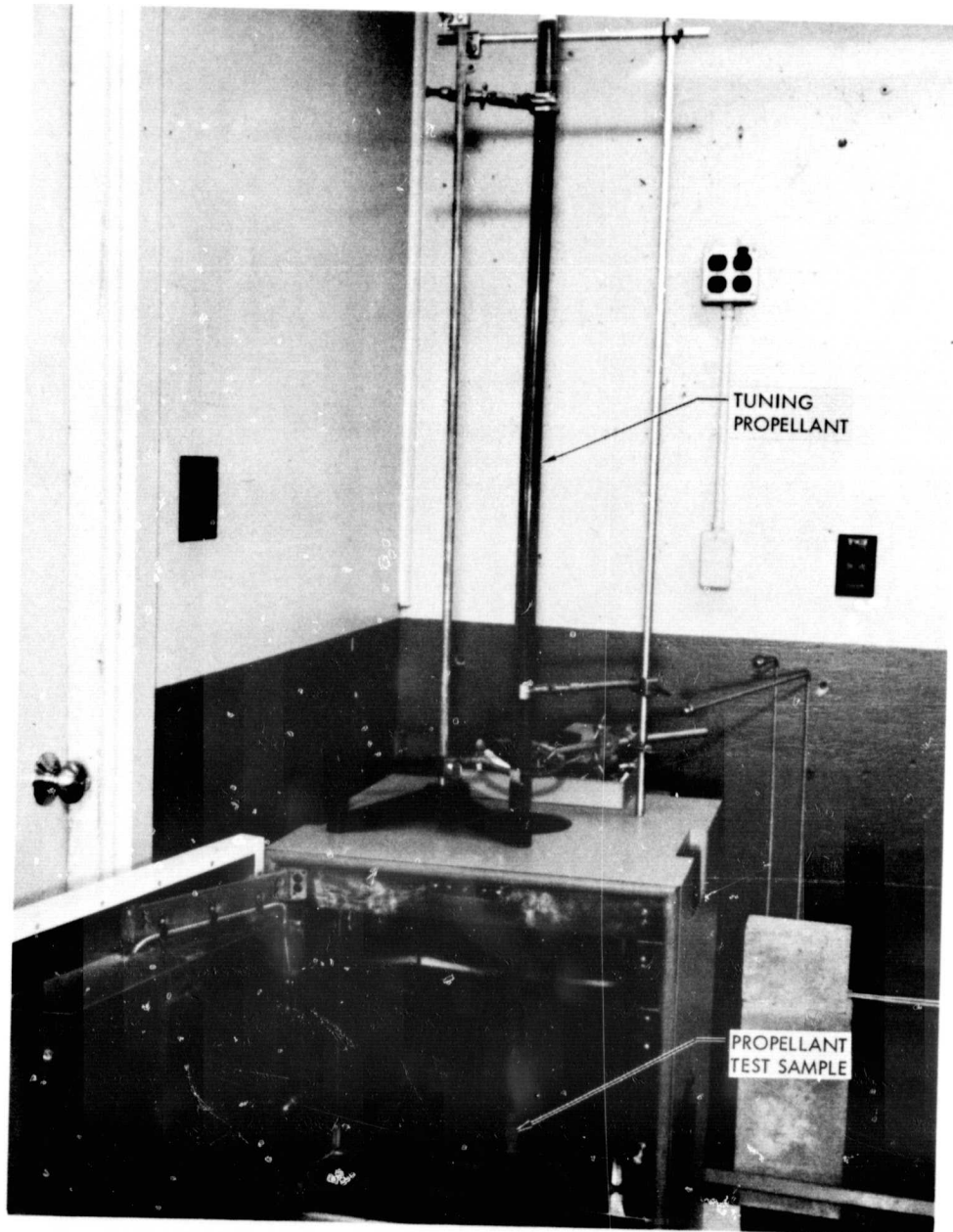Fig. 2. Pressure Chamber Cutaway Drawing

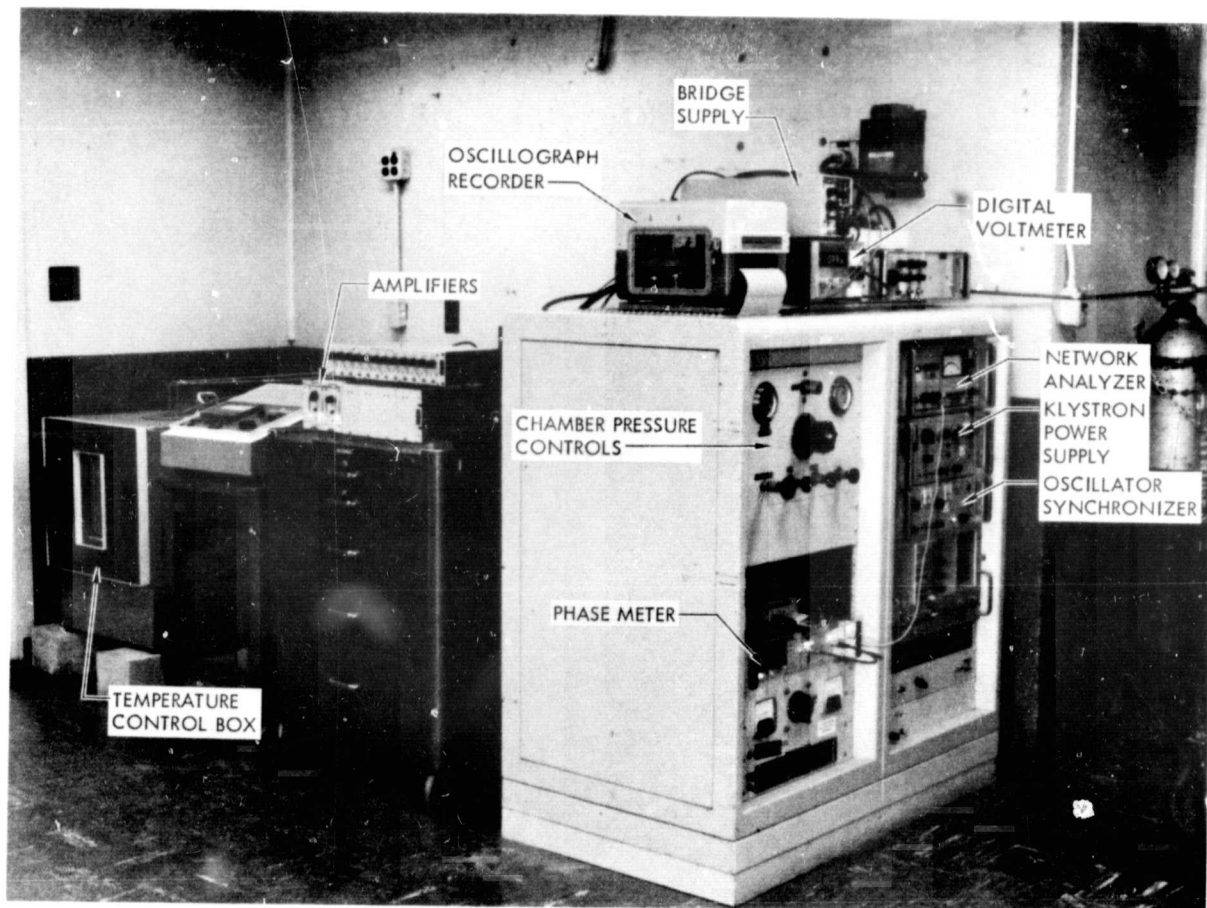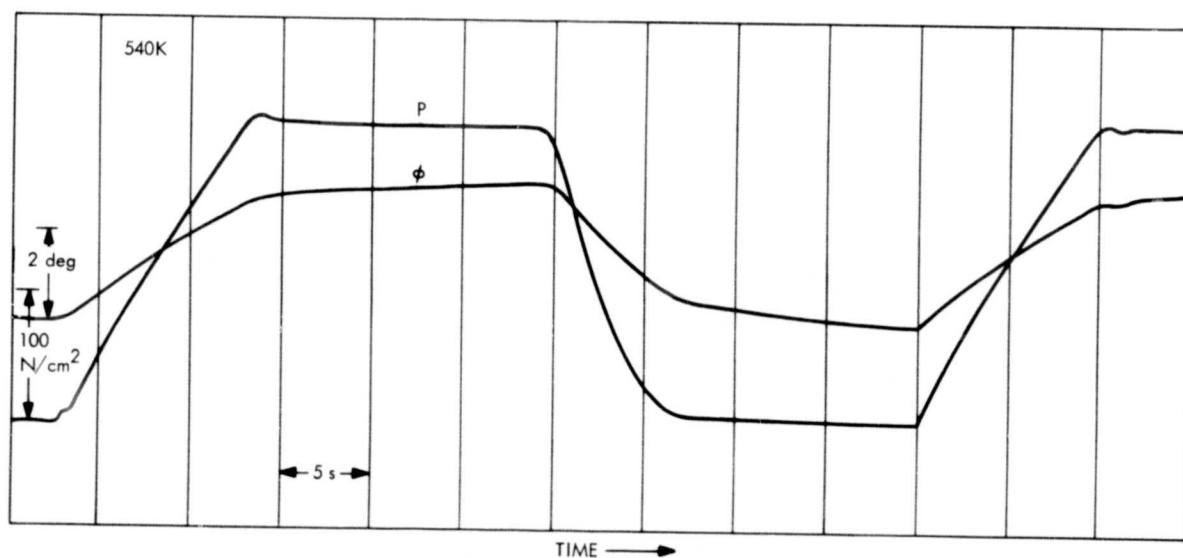Fig. 3. Setup for Microwave Tuning Operation

Fig. 4.   Over-all Test System

Fig. 5.   Segment of Oscillograph Test Record