THE UNIVERSITY OF MANITOBA

A SAMPLING SYSTEM FOR LOW FREQUENCY DIELECTRIC SPECTROSCOPY

by

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ABSTRACT

A novel technique is described for fast permittivity measurement over the frequency range 0.5 Hz to 16 kHz.

A sampling system has been developed, to perform measurement of the current-time response to a voltage step of a capacitor containing a dielectric material.

The data is recorded at sixteen logarithmically spaced times with the base of 2 from 10 µs to 327.68 ms. Analog to digital conversion technique has been employed, in order to facilitate time to frequency domain transformation by means of digital computer. A computer program based on an appropriate Fourier transform has been developed for processing of the experimental data. The results of the relative permittivity and loss factor can be calculated at sixteen descrete frequencies spaced at octave intervals.

Particular emphasis is given to the problems concerning sampling circuitry.

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CHAPTER 1

INTRODUCTION

The technology for the measurement of the dielectric parameters has been highly developed in recent years. It is customary to make such measurements at discrete frequencies. This requires a trained operator to execute some crucial adjustments, depending on the technique employed at each frequency. The time taken in manipulations, and conversion of the results into usable form, make such technique very laborious. Especially, at the low frequency range of the spectrum such measurements become extremely time consuming due to large period of the exciting wave. The rate of sweeping of the oscillator covering the required frequency range must be slow in comparison with the wave cycle, which also increases the measurement time.

When the results of a variation of dielectric parameters have to be evaluated quickly and often, it becomes necessary to look for a faster method of measurement.

The step-response method, or transient-method, does not suffer from these problems. A single voltage step function is used for excitation of the dielectric material, and its time response is measured. The Fourier transform converts results from the time to frequency domain. It should be emphasised, that the data covering the whole range of frequencies is obtained within duration of a single voltage step, therefore, the measurements at very low frequencies have enormous advantage of speed.

In this project, a system employing the principle of the transient method has been developed. A sampling system has been designed and constructed for measurement of the time--response of a capacitor containing a dielectric material to a single voltage step. Sixteen samples from the response curve are taken at logarithmically spaced time intervals with the base of 2.

The first sample is recorded at 10 µs after applying the voltage step exciting the dielectric. Due to limited conversion speed and accuracy of analog to digital converters, typical sampling circuitry employing a separate voltage step for recording each data had to be built. The last sampling time is equal $/2^{15} \times 10/$ µs.

Using appropriate Fourier transforms, the results of the relative permittivity and loss factor can be calculated at sixteen discrete frequencies spaced at octave intervals. The measurement covers the frequencies from 0.5 Hz to 16 kHz. The time taken to record data by sampling system is approximately 30 sec.

CHAPTER 2

SOME PROPERTIES OF DIELECTRICS

2.1. Basic parameters and background material

The a-c characteristics of materials can be defined in terms of circuit theory concepts /1/. The interaction of electromagnetic field with dielectric material can be described using condenser and coil to separate the influence of electric and magnetic fields.

2.1.1. Permittivity

A capacitor, connected to a sinusoidal voltage source $V = V_o e^{j\omega t}$ of the angular frequency $\omega = 2\pi f$, stores, when vacuum is its dielectric, a charge $Q = C_o V$ and draws a charging current

$$I_c = \frac{dQ}{dt} = j\omega C_o V \qquad /2.1/$$

This current is leading the voltage by a temporal phase angle 90^o /Fig.2.1/. C_o is the vacuum or geometrical capacitance of the condenser.

When filled with some substance, the condenser increases its capacitance to

$$C = C_o \frac{\varepsilon'}{\varepsilon_o} = C_o \varepsilon'_r \qquad /2.2/$$

where ε' and ε_o designate the real permittivities or dielectric constants of the dielectric and of the vacuum, respectively, and their ratio ε'_r the relative dielectric constant of the material / $\varepsilon_o = 1/36\pi \times 10^{-9}$ farad/m/.

Simultaneously, there may appear, in addition to the charging current component I_c, a loss current component

$$I_l = \sigma_c V \qquad /2.3/$$

in phase with the voltage, where σ_c represents the conductivity associated with conduction current in the dielectric.

The total current traversing the condenser

$$I = I_c + I_l = /j\omega C + \sigma_c/ .\ V \qquad /2.4/$$

is inclined by a power factor angle $\theta < 90^o$ against the applied voltage V, that is, by a loss angle δ against +j-axis /Fig.2.2/.

The frequency response of this current, which can be expressed by the ratio of loss current to charging current, that is, loss tangent $\tan\delta$ as

$$\tan\delta = \frac{I_c}{I_l} = \frac{1}{RC\omega} \qquad /2.5/$$

may not at all agree with that actually observed because the conductance term need not stem from migration of charge carriers, but can represent any other energy-consuming process. It has become customary to refer to the existence of a loss current in addition to a charging current, by the introduction of the complex permittivity

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \qquad /2.6/$$

The total current I, may thus be rewritten as

$$I = /j\omega\varepsilon' + \omega\varepsilon''/ \frac{C_o}{\varepsilon_o} V = j\omega C_o \varepsilon_r^* V \quad /2.7/$$

where

$$\varepsilon_r^* = \frac{\varepsilon}{\varepsilon_o} = \varepsilon_r' - j\,\varepsilon_r'' \qquad /2.8/$$

is the complex relative permittivity of the material, and ε'' and ε_r'' are the loss factor and relative loss factor, respectively. The loss tangent becomes

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} = \frac{\varepsilon''_r}{\varepsilon'_r} \qquad /2.9/$$

Since a parallel - plate condenser of the area A and the plate separation d, has the vacuum capacitance

$$C_o = \frac{A}{d}\varepsilon_o \qquad /2.10/$$

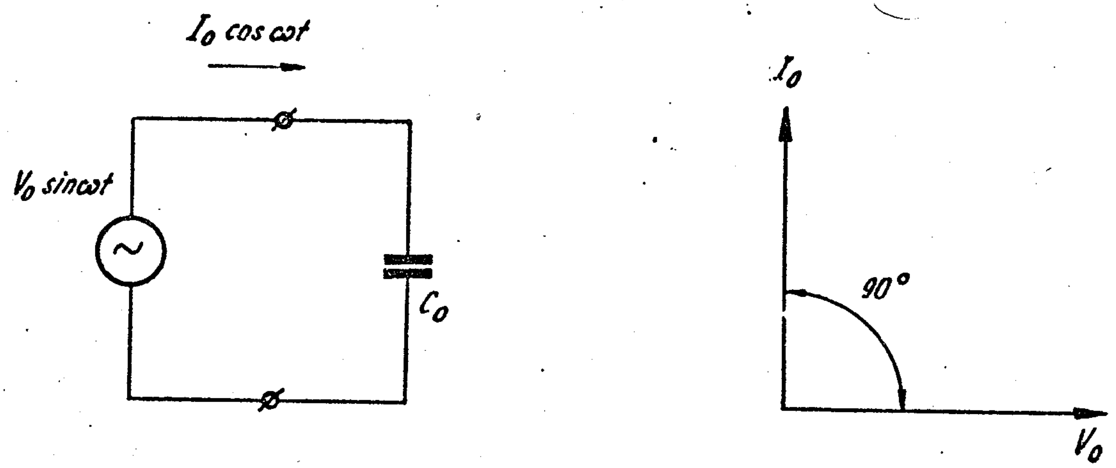


Fig. 2.1 Current-voltage relation in ideal capacitor

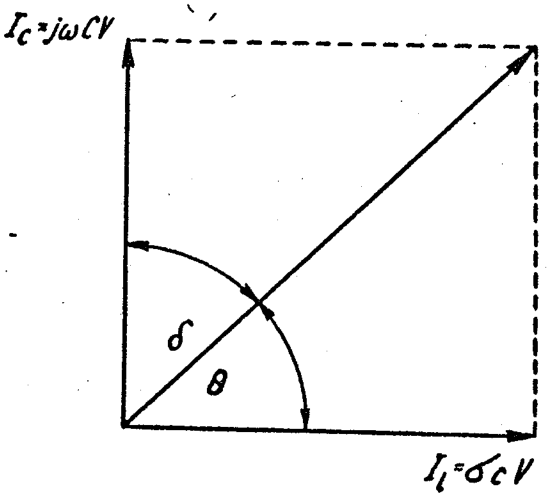


Fig. 2.2 Capacitor containing dielectric with d-c loss

the current density J traversing a condenser under the applied field strenght $E = V/d$, becomes

$$J = /j\omega\varepsilon' + \omega\varepsilon''/E = \frac{dE}{dt} \qquad /2.11/$$

The product of angular frequency and loss factor is equivalent to a dielectric conductivity

$$\sigma = \omega\varepsilon'' \qquad /2.12/$$

This dielectric conductivity sums over all disspative effects and may represent an actual conductivity caused by migrating charge carriers and an energy loss associated with a frequency dependence (~~dispersion of ε' for example~~).

2.1.2. Permeability

If the dielectric material is transferred from the electric field of the capacitor into the magnetic field of a coil, the voltage V drives through the coil a magnetization current I_m according to Faraday's inductance law

$$\tilde{I}_m = \frac{V}{j\omega L_o \frac{\mu'}{\mu_o}} \qquad /2.13/$$

where L_o represents the vacuum or geometrical inductance of the coil. This magnetization current lags behind the applied voltage by 90^o. The permeabilities μ', and μ_o designate the magnetization of the material and of the vacuum, respectively, and their ratio μ'/μ_o the relative permeabilty of the material in which the magnetic field of the coil resides / $\mu_o = 4\pi \times 10^{-7}$ H/m/.

Because of the resistance R of the coil windings, an ohmic current component V/R exists. In addition, there may appear, in phase with V, a magnetic loss current I_1 caused by energy disspation during the magnetization cycle.

In complete analogy to the dielectric case a complex permeability is introduced

$$\mu^* = \mu' - j\mu'' \qquad /2.14/$$

The complex relative permeability is

$$\mu_r^* = \frac{\mu}{\mu_o} = \mu_r' - j\mu_r'' \qquad /2.15/$$

Thus the total magnetization current is

$$I = I_m + I_1 = \frac{V}{j\omega L_o\mu_r^*} \qquad /2.16/$$

2.1.3. Frequency behaviour

Values for dielectric properties of most materials are very much dependent upon the frequency of the alternating field. According to previous considerations, the macroscopic behaviour of a dielectric material is determined by the two complex parameters ε^* and μ^*.

For most materials other than ferromagnetic substances, μ^* has essentially the value of μ_o, and this holds true for biological materials and agricultural products.

Discussions of the anomalus dispersion /the decreasing of ε', with increasing frequency/ and the dielectric theories of Debye, Osanger, Cole, Kirkwood, Fröhlich, and others, are found in many references /1/, /2/, /12/. Basically, all of these formulations recognize a contribution to the dielectric constant of materials containing molecules with electric dipole moments, through the polarization, resulting from the orientation of the dipoles with the applied electric field. Two lesser contributions to the polarizability arise from electronic polarization, and atomic polarization. These two types of polarization are termed "deformation" polarization.

As frequency increases from low values, the polar molecules can follow the changes in direction of the electric field up to a point, and, as frequency continues to increase, the dipole motion can no longer keep up with the changing field. As a result, the dielectric constant drops with increasing frequency in this region, and energy is absorbed, as a result of the phase lag between the dipole rotation and the field. The relationship between the dielectric constant and loss factor is illustrated in Fig.2.3. Debye /1929/ developod the mathematical formulation which can be expressed as

$$\varepsilon_r^* = \varepsilon'_{r\infty} + /\varepsilon'_{rs} - \varepsilon'_{r\infty}/ / /1 + j\omega\tau/ \qquad /2.17/$$

where ε'_{rs} is the static or d-c value of the dielectric constant, $\varepsilon'_{r\infty}$ is the infinite frequency or optical value, and τ is the relaxation time. The loss factor, ε''_r, peaks at the relaxation frequency $\omega = 2\pi f = 1/\tau$. At this frequency, ε''_r has the value $/\varepsilon'_{rs} - \varepsilon'_{r\infty}//2$ and ε'_r has the value $/\varepsilon'_{rs} + \varepsilon'_{r\infty}//2$.

The dispersion of some pure liquids closely follows Debye equations.

It was noted by Cole and Cole, that plotting ε'_r and ε''_r in the complex plane in accordance with the Debye relation, results in a semicircle /Fig.2.4/.

The modified equation presented by Cole and Cole /1941/, results in a Cole - Cole plot, which is an arc of a circle with the center below the $\varepsilon''_r = 0$ axis /Fig.2.5/.

$$\varepsilon_r^* = \varepsilon'_{r\infty} + \frac{\varepsilon'_{rs} - \varepsilon'_{r\infty}}{1 + /j\omega\tau/^{1-\alpha}} \qquad /2.18/$$

α is the empirical relaxation - time distribution parameter and takes values between 0 and 1.

Several other models have been developed to explain the behaviour of certain types of materials. One known as the Cole - Davidson representation, results in a skewed arc

/Fig.2.6/, and mathematically it is defined as

$$\varepsilon_r^* = \varepsilon'_{r\infty} + \frac{\varepsilon'_{rs} - \varepsilon'_{r\infty}}{/1 + j\omega\tau/^{\beta}} \qquad /2.19/$$

where β is restricted to values between 0 and 1.

The incidence of d-c conductivity appears as a distorting feature plotting ε'_r and ε''_r in the complex plane. This is shown in Fig.2.7. The constancy of / ε'' x f/ at low frequencies provides one of the best methods of evaluating conductance in poorly conducting media.

2.2 Biological materials as dielectrics

Generally, for biological materials and agriculture products $\mu^* = \mu_0$ and therefore the dielectric constant versus frequency dependence becomes of first interest in dielectric studies.

Another absorption process is likely to be present in agricultural products /10/, especially, at low frequencies /below 1kHz /2//. The general aspects of this absorption were evaluated by Wagner, and the dielectric effect is referred to, as Maxwell - Wagner absorption. It results from polarizations at interfacial boundaries and occurs in nonhomogeneous materials. The frequency character of the process is identical with that of the simplest /Debye/ dipolar absorption, and many biological specimens show it. The process is far more common in solid materials. In addition, energy dissipation can occur due to d-c conductivity of the medium and electrode polarization effects. Especially, this appears in systems which have an ionic conductance. Not infrequently, all these processes and dipolar absorption will occur simultaneusly, making dielectric studies particulary troublesome in this frequency range.

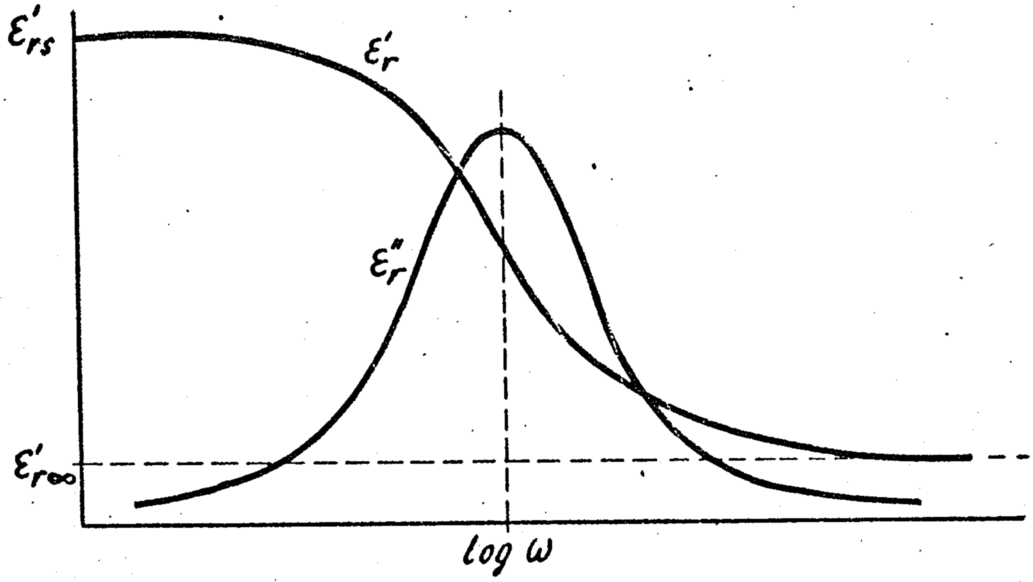


Fig. 2.3 Dispersion and absorption curves for a polar material following the Debye relaxation process

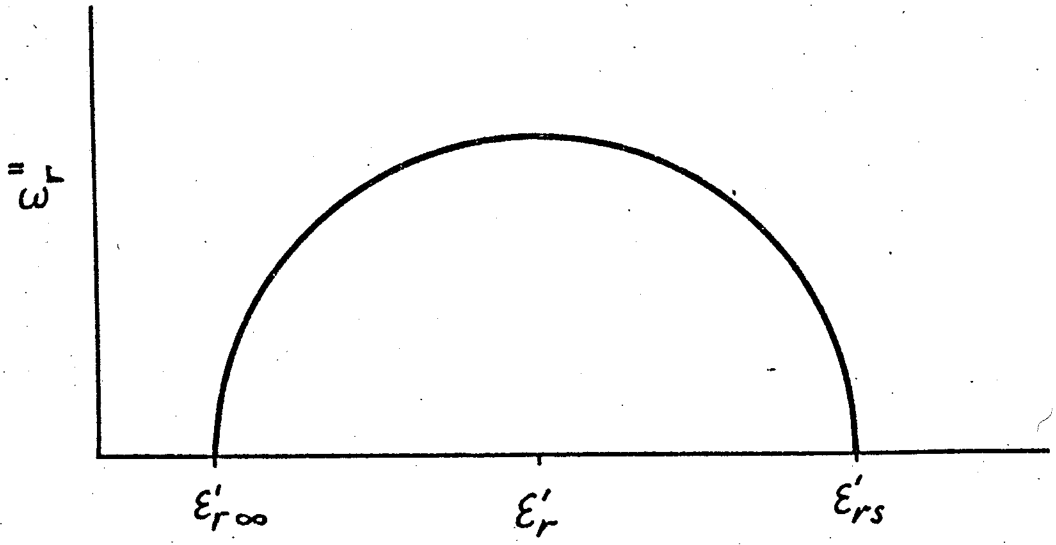


Fig. 2.4 Cole-Cole plot for Debye relation

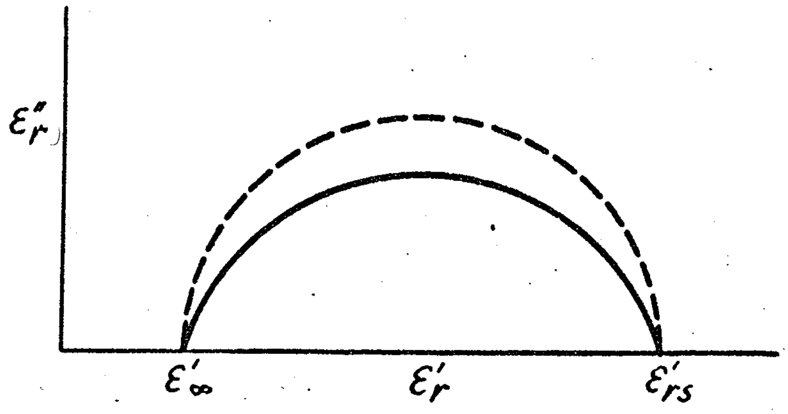


Fig. 2.5 Cole-Cole circular arcs

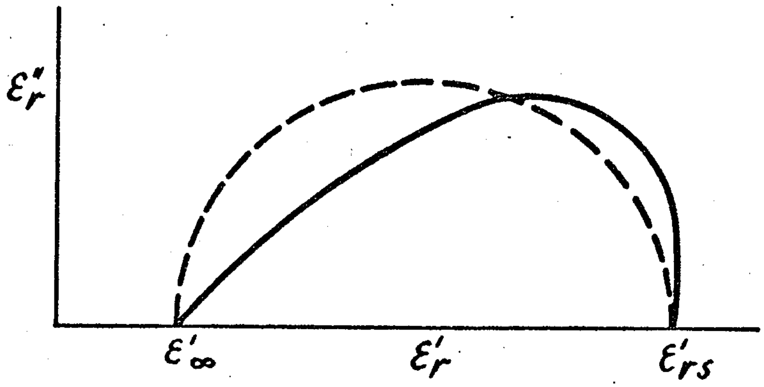


Fig. 2.6 Cole-Davidson skewed arcs

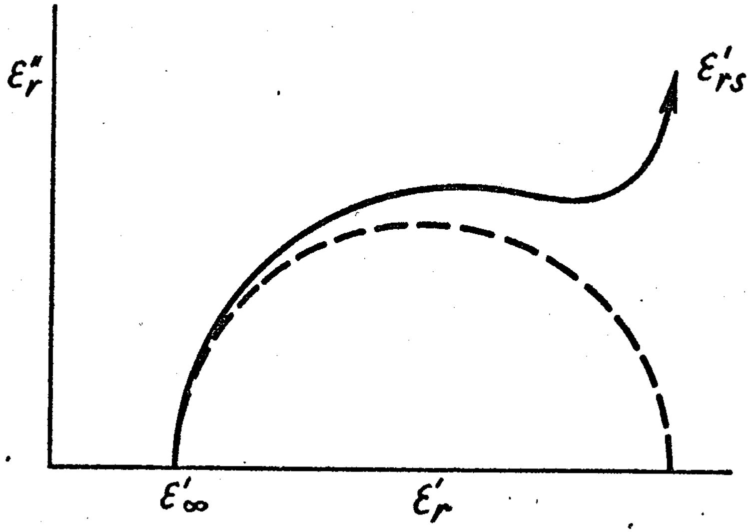


Fig. 2.7 Cole-Cole plot for dielectric
with d-c conductivity

CHAPTER 3

EXPERIMENTAL TECHNIQUES

For many applications some knowledge is essential concerning dielectric properties of materials being considered. Since the dielectric properties vary with frequency, their frequency dependence may be necessary for a particular appplication.

Dielectric measurement may be made over a very wide range of frequency, from the microwave region, through the radio- and audio- frequency regions, down to very low frequencies.

Many methods have been developed for determining dielectric properties of materials depending on the frequency range. For the microwave region /up to 18×10^9 Hz/, a time-domain technique performed by means of fast rise time pulse, and high frequency sampling oscilloscop, is very useful/11/. Resonant circuit are used successfully at the radiofrequencies /10^5 - 10^8 Hz/. Lower frequencies can be investigated using two basic approaches to the problem, the steady-state technique, and the transient technique.

3.1 Steady-state method

This method is very widely used at present time. Generally, it employs a specially constructed sample holder capacitor, which is used with an impedance bridge and generator, to measure changes in capacitance and dissipation factor due to presence of the sample material. From these electrical measurements and the volume of the sample in the sample holder, the dielectric properties are calculated. Fig. 3.1 shows basic diagram of the measuring circuit. It is usual for such measurements to be made at discrete frequencies only, and a trained operator is almost always needed to execute some crucial adjustment procedure at each frequency.

Until fairly recently, the lowest frequency, obtainable with conventional bridges had been about 10 Hz using Schering bridge /2/. Special bridges supplied with a motor driven potentiometer have been used down to about 0.1 Hz /6/. The frequencies of interest are often so low, that steady-state measurement becomes extremely time consuming, as the rate of sweeping oscillator must be slow, in comparison with the period of the exciting wave. The scarcity of skilled operators, and the time taken in manipulations, make this method increasingly incompatible with the needs, when the results of a variation of the dielectric properties may have to be evaluated quickly and often.

3.2 Transient method

The transient method or step response method does not suffer from these problems. The principle of operation, is to record a time response to a voltage step of a capacitor containing a sample of measured material /Fig.3.2/.
It should be ephasised, that the excitation is by a simple voltage step function, and the response measured within duration of the single step is used as a data, to cover the whole range of frequencies.

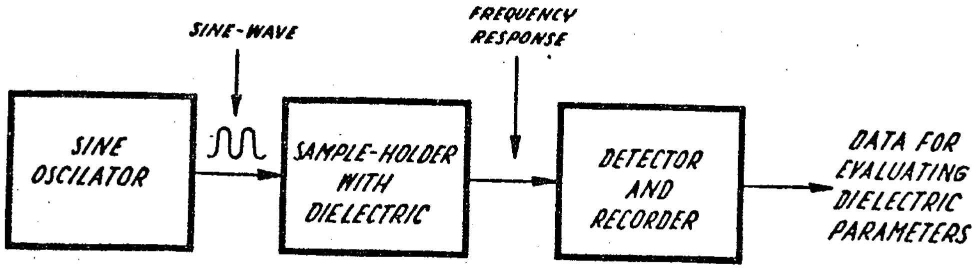
For a capacitor having unity vacuum capacitance, filled with the material as dielectric, the current response i/t/ to a unit voltage step, is connected with complex permittivity by the Fourier transform /3/.

$$\varepsilon^{*}/\omega/ = \varepsilon'/\omega/ - j\,\varepsilon''/\omega/ = \int_{0}^{\infty} i/t/\exp/-j\omega t/dt \qquad /3.1/$$

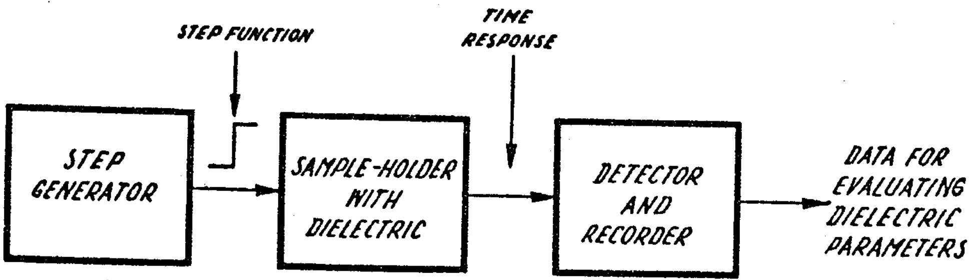
A major practical difficulty in using this method to obtain steady-state information is recording of the i/t/ at both small and large values of time due to some technical reasons.

It means, that any experimental data will cover only a finite range of time, and errors will therefore arise owing to cutting off of the Fourier transform integral at both sides. The magnitude of such errors will depend on the time range of available data, and the value of ω in relation to the minimum and maximum values of time. Hence, it may be inferred, that the wider range of time that the data cover, the wider range of ω is described by, and the $\varepsilon(\omega)^*$ is known more accurately. Ignoring other benefits, this alone emphasises the importance of obtaining time response data over as wide a range of time as possible.

A disadvantage of the transient method is that it can not be applied easily to materials which show significant nonlinearity; fortunately, using even moderately high amplitude of step voltage, most of them are not intended do so.



3.1 Basic diagram of the measuring circuit for steady-state technique



3.2 Basic diagram of the measuring circuit for transient technique

CHAPTER 4

DEVELOPMENT OF EXPERIMENTAL EQUIPMENT

4.1 General design approach

The design and development of the measuring system requires consideration of many different aspects.

The experimental system should provide fast measurement of dielectric properties of many materials. Further, the system should be characterized by simplicity of operation and computer compatible output for easy data processing. Comercially available equipment should be utilized wherever possible.

The construction of a sample-holder for dielectric specimens should facilitate its filling with granular materials.

The design of the system has been based on principles of transient method, as the frequency range of interest is from kHz region down to very low frequencies.

4.2 Measuring system

A simplified block diagram of the system which has been developed is shown in Fig.4.1.

A step voltage produced by HP 8003A pulse generator is, applied to the sample of a dielectric specimen placed in the sample-holder capacitor. The output pulse exciting the dielectric is triggered from the control circuit.
This pulse has 5ns rise time and its duration can be adjusted up to 3 sec. Maximum output amplitude is ± 5 V across a 50 ohms load impedance.

A HP 3480A digital voltmeter combined with HP 5055A digital recorder have been used as the recording equipment. These instruments provide a high performance economical method of making permanent records of digital data.
The voltmeter has 950 μs reading time and ± /0.01% of reading + 0.01% of range/ voltage accuracy. An external trigger

from control circuit assures reading, when the sample-hold system is in the hold mode. Output information from the voltmeter is available in the BCD and together with print command is interfaced with the recorder. Permanent records of data are printed on paper tape.

The part of the system which has been designed and built consists of:

- Sample-holder Capacitor
- Input Amplifier and Sample-hold Circuit
- Control Circuit

These units have been developed to cope with the requirements for the experimental system, and more detailed discussion is devoted to them.

It should be pointed out that the use of the system is very simple. All the control signals are performed automatically in order to obtain fast measurement and simplicity of operation.

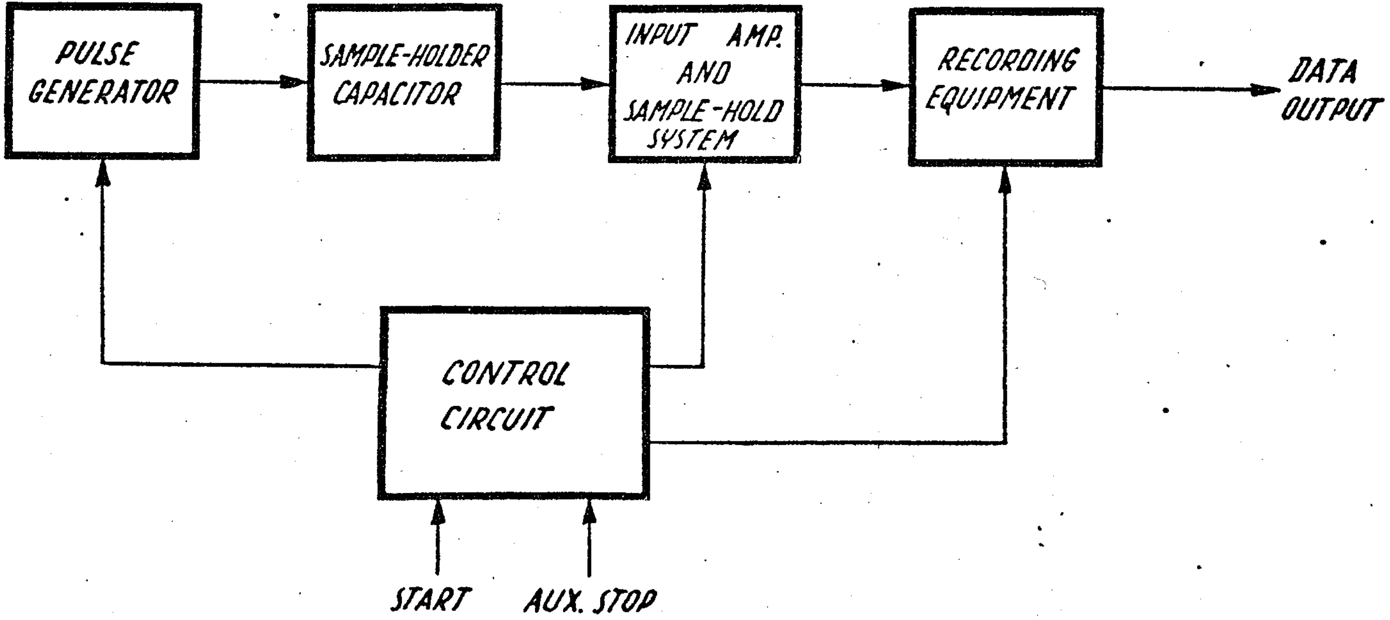


Fig. 4.1 Block diagram of the measuring system