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DIELECTRIC MEASUREMENTS OF SELECTED CERAMICS AT MICROWAVE FREQUENCIES

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KEY WORDS

Dielectric relaxation, ceramics, phase change, perturbation, frequency shift, Q-change.

OBJECTIVES

1. To understand the basic concepts of dielectric relaxation.
2. To study the process of dielectric measurement of strontium titanate and lead titanate zirconate ceramics at microwave frequencies.

SUMMARY

Dielectric measurements of strontium titanate and lead titanate zirconate ceramics are conducted at microwave frequencies using a cylindrical resonant cavity in the TE_{011} mode. The perturbations of the electric field are recorded in terms of the frequency shift and Q-changes of the cavity signal. Slater's perturbation equations are used to calculate ε' and ε'' of the dielectric constant as a function of temperature and frequency.

INTRODUCTION

A microwave resonant cavity has become a standard technique to study dielectric relaxation in a compound of interest. It is a definite improvement over the waveguide techniques in which the sample under investigation was placed in a hole in the waveguide itself to study dielectric behavior. In a resonant cavity the perturbations of the electric and magnetic fields are well defined depending on the structure of the cavity.

For this experiment a cylindrical resonant cavity in the TE_{011} mode was designed to study the dielectric behavior of strontium titanate ceramic at microwave frequency of 9.7 GHz at different temperatures. The dielectric behavior of lead titanate zirconate was studied as a function of frequency from 8.8 to 9.7 GHz. The strong perturbations of the electric field were recorded in terms of the frequency shifts and Q-changes at different temperatures. Using Slater's perturbation equations (Ref. 4), the real and imaginary parts of the complex permittivity were calculated at different temperatures. The relaxation times for these compounds were calculated using Debye's theory for polar molecules. Dielectric behavior of a mixture of barium and strontium titanates and lead titanate zirconate has been studied at different frequencies (Ref. 1-3). The resonant cavity technique has been very successfully used to study dielectric relaxation in a number of compounds of interest (Ref. 4-7).

EXPERIMENTAL PROCEDURE, THEORY AND RESULTS

The details of the microwave spectrometer used in this investigation operating in the x-band of frequencies were given in a number of papers (Ref. 4 and 7). The block diagram of such a spectrometer is shown in Figure 1. The signal was produced by a tunable klystron and transmitted through the waveguides to the resonant cavity where the material under investigation was placed into the cavity. The modulated signal as displayed on the oscilloscope was of the form of a butterfly. A part of the signal from the directional coupler was mixed with a standard frequency taken from an oscillator. A radio receiver was used to detect the change in the frequency of these signals in the form of two markers also displayed on the oscilloscope. These markers were used to find the positions of frequency shifts and Q-changes as the sample under investigation goes through a dielectric change. A true copy of the signal as taken from the oscilloscope is shown in Figure 2.

When a dipole is placed in an electric field $\vec{E}$, it experiences a torque given by

$$\vec{\tau} = \vec{p} \times \vec{E} \qquad (1)$$

where **p** is the dipole moment vector. The dipole rotates in the electric field and stores potential energy given by

$$U = -\vec{p} \cdot \vec{E} \qquad (2)$$

At lower frequencies, the dipole can follow the orientations of the applied field without any phase difference. At higher frequencies, there is a phase lag between the orientation of the field and the dipole and the dielectric constant becomes a function of frequency. As a matter of fact, the dielectric constant becomes a complex quantity

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \tag{3}$$

With ε' and ε'' being the real and imaginary parts of the dielectric constant and ε'' being necessarily the loss term for the dielectric constant. The value of the dielectric constant basically depends upon the number of dipoles that can follow the orientations of the applied field. At higher frequencies the number of dipoles following the orientations of the field goes down and hence a lower value for the dielectric constant. The ratio of ε' and ε'' is expressed as a loss tangent as given by

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \tag{4}$$

ε' and ε'' are further related to the frequency shifts and Q-changes as expressed in the Slater's perturbation equations (Ref. 4).

$$\frac{\Delta f}{f_o} = -\frac{\varepsilon'-1}{2}\,\frac{\int E_s \cdot E\, dv}{\int E \cdot E_a\, dV} \tag{5}$$

and

$$\Delta\left(\frac{1}{Q}\right) = \varepsilon''\,\frac{\int E_s \cdot E\, dv}{\int E \cdot E_a\, dV} \tag{6}$$

where E is the field of the unperturbed cavity, E_a is the microwave field as applied to the cavity and E_s is the field of the sample itself, v and V are the volumes of the sample and cavity respectively.

The perturbations of the sample under investigation were studied by experimentally recording the frequency shifts and the width changes of the microwave resonant signal by placing a fixed length of the sample into the resonant cavity. The width change of the signal is directly related to the Q-change of the cavity. These changes in width and frequency of the signal were recorded at different temperatures as the substance under investigation goes through a phase change. The frequency of the signal was kept constant for one set of data taken from one temperature to the other for strontium titanate. Dielectric relaxation in lead titanate zirconate was studied as a function of frequency. Dry nitrogen gas at the liquid nitrogen temperature from a heat exchanger was allowed to circulate around the cavity containing the sample under investigation. A thermal equilibrium was maintained by controlling the amount of cold air circulating around the resonant cavity and this method is very useful in maintaining a certain temperature while the material under investigation goes through a phase change.

Figures 3 and 4 show the dielectric behavior of strontium titanate as a function of temperature at a microwave frequency of 9.7 GHz. Figure 3 shows the behavior of Q-change as a function of temperature. From Slater's equations, Q-change corresponds to the dissipation term (ε'') in the dielectric constant. This curve shows a peak around 20°C and after that there is a sharp drop in the dissipation factor. The frequency shift vs temperature behavior as displayed in Figure 4 shows a dramatic change in the real part of the dielectric constant around -57°C. The strontium titanate ceramic undergoes a phase change at this temperature and polarization of the material follows the orientation of the field. The relaxation times using Debye's theory are of the order of 10^{-12}- 10^{-14} sec.

The dielectric behavior of lead titanate zirconate as a function of frequency is displayed in Figures 5 and 6 with Figure 5 showing the Q-change vs applied frequency and Figure 6 shows the behavior of $\Delta f/f_o$ at different frequencies. The dissipation factor (ε'') as displayed in Figure 5 shows an increase as the frequency increases up to around 9.6 GHz and after that has a decreasing trend. The real part of the dielectric constant decreases as the frequency increases and then levels off around 9.6 GHz. This behavior is shown in Figure 6. The dielectric behavior of strontium titanate and lead titanate zirconate ceramics has been studied very successfully by using the microwave resonant cavity as a probe. This method will also be applied to study the mass dependence of these materials at different microwave frequencies and the free energy of activation calculated at these frequencies.

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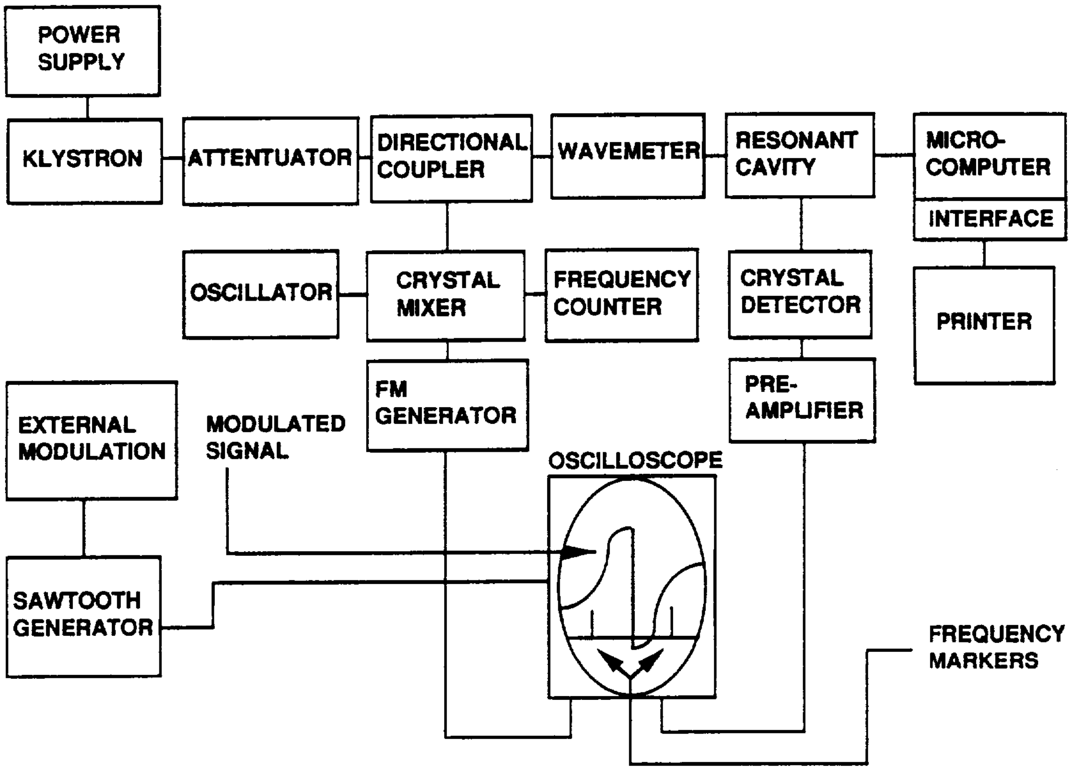
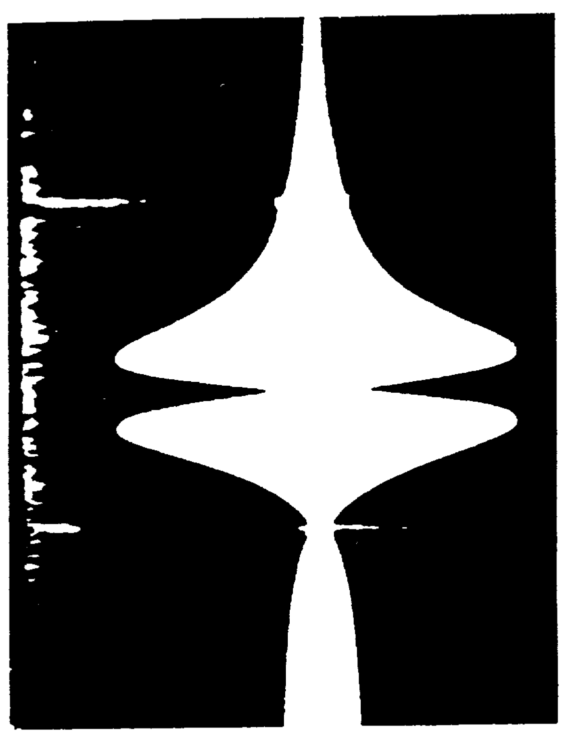
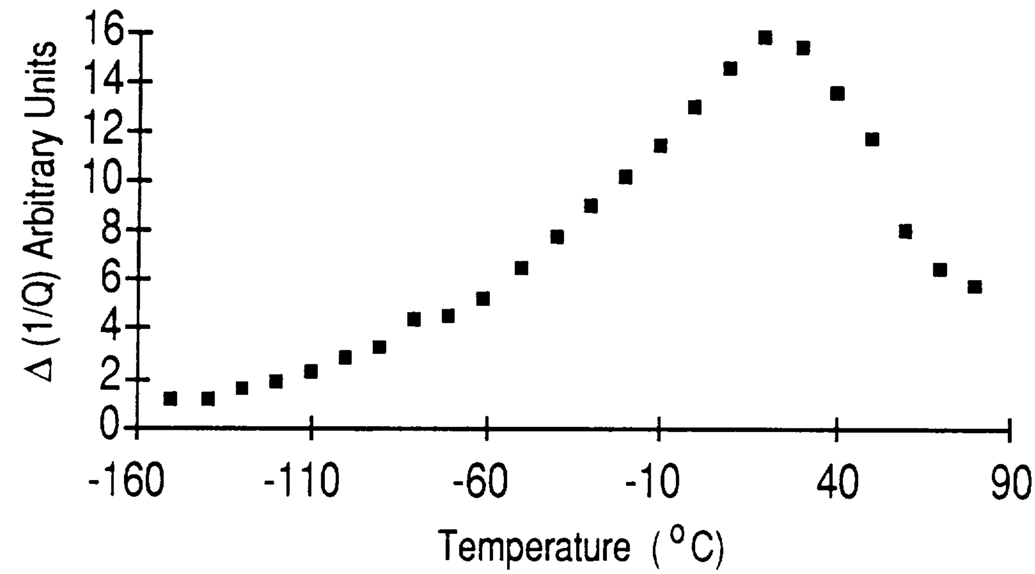


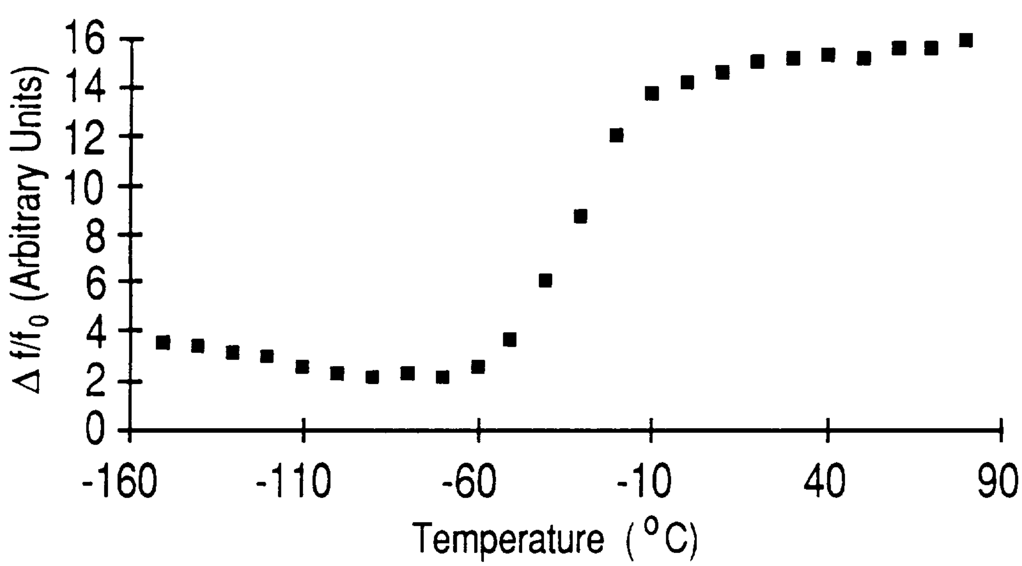
1. Block diagram of the microwave spectrometer used in this investigation.



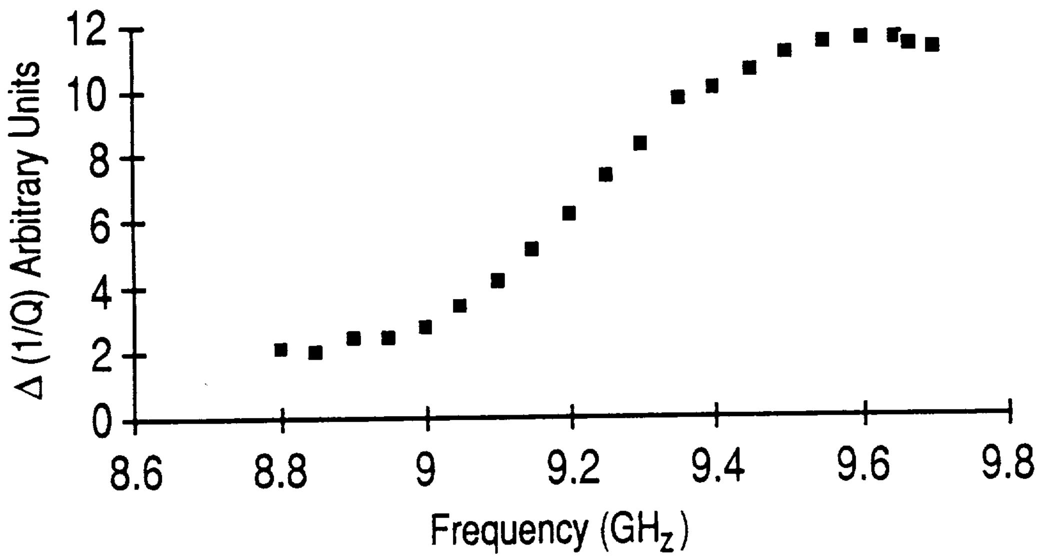
2. Dual trace oscilloscope display for frequency markers and cavity derivative profile for a typical scan. (The marker interval is always twice the radio receiver reading and in this scan the spacing is 8 MHz, with center frequency at 9.7 GHz.)



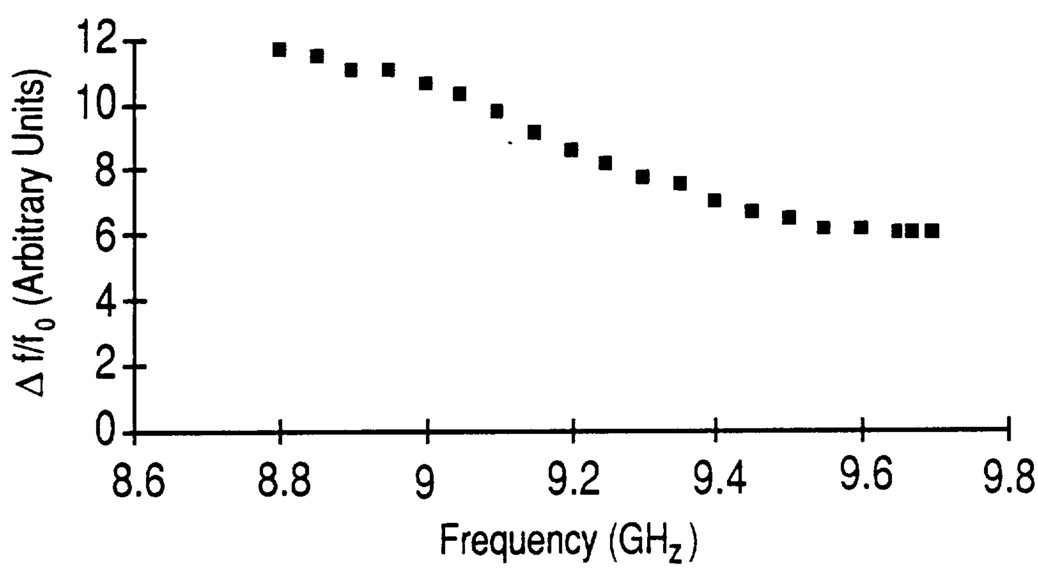
3. Microwave loss Δ(1/Q) as a function of temperature for a sample of strontium titanate.



4. Frequency shift $\Delta f/f_o$ as a function of temperature for a sample of strontium titanate.



5. Microwave loss Δ(1/Q) as a function of frequency for a sample of lead titanate zirconate.



6. Frequency shift $\Delta f/f_o$ as a function of frequency for a sample of lead titanate zirconate.