Measurement of the Dielectric Constant and Loss Tangent of Thallium Mixed Halide Crystals KRS-5 and KRS-6 at 95 GHz

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Abstract—The dielectric constants and loss tangents of KRS-5 and KRS-6 thallium halide mixed crystals have been measured at 95 GHz using both the shorted waveguide (SWG) reflection method and the Fabry–Perot (F–P) transmission method on samples filling standard WR-10 waveguide. The results—KRS-5: $\epsilon_r' = 31$, $\tan\delta = 1.8\times10^{-2}$; KRS-6: $\epsilon_r' = 29$, $\tan\delta = 2\times10^{-2}$—agree reasonably well with a simple theoretical fit to the far-infrared lattice absorptions of TlBr and TlCl centered at about 1400 GHz. The dielectric samples were hot-pressed into copper wafers with dimensions matching WR-10 waveguide, and then machined and polished to obtain flat, parallel air–dielectric interfaces.

I. Introduction

The mixed crystal thallium bromide–iodide (KRS-5) has long been known as an infrared transmitting window material for the wavelength range 0.6–40 μm. However, little was known about its microwave transmission properties, and nothing of its properties in the millimeter wave range. Recently, long fibers of KRS-5 have been fabricated, and their infrared transmission has been reported [1]. Soon afterward, propagation in a KRS-5 fiber at 95 GHz was demonstrated [2], thus raising the possibility of waveguide applications in the millimeter-wave range. The low-frequency dielectric constant of KRS-5 is given by von Hippel [3] as 32, which would imply a very small fiber diameter for such a millimeter-wave guide (less than 1 mm diameter) and allow a wide range of dielectrics for cladding material, for example, Teflon or polyethylene. Von Hippel also reports a loss tangent of 2×10^{-3} in KRS-5 at 10 GHz [3], while Popa and Johnson [2] measured a value of 2.3×10^{-3} at 37 GHz. The losses are expected to be larger at higher frequencies due to lattice absorption, but no literature values are available. The reported low-frequency losses in KRS-6 (thallium bromide–chloride) are also quite low [3]. Accordingly, we undertook a study of the dielectric properties of KRS-5 and KRS-6 at 94 GHz to assess the potential of these materials in a practical flexible waveguide.

Our measurement techniques utilize samples mounted in standard metal waveguide, in contrast to past work at 95 GHz, which was based primarily on quasi-optical techniques. Our preference arises from the simplicity and accuracy of waveguide techniques and a novel sample mounting configuration which eliminates gaps between sample and wall. Two different waveguide measurement techniques were used with the same samples: 1) measurement of the transmission through or reflection from a planar slab of dielectric, taking into account the multiple reflections between the two faces—a Fabry–Perot (F–P) resonator (our method is a modification of that described by Redheffer [5]); 2) measurement of the reflection from a sample backed by a short (a well-known technique; see Roberts and von Hippel [4]).

In addition, samples of Teflon and Rexolite were measured by these same two techniques as a check on the validity and accuracy of the methods.

II. Sample Preparation

In preparing samples for any waveguide measurement, it is very important that a tight fit be obtained to the waveguide walls. The errors introduced by any gap between the wall and the sample increase as the dimensions of the waveguide and sample decrease and as the dielectric constant increases. In order to obtain the best fit for the 95-GHz measurements, the samples of KRS-5 were hot-pressed into a waveguide-shaped opening in a copper wafer. The cross section of the opening was 2.54×1.27 mm, corresponding to standard WR-10 waveguide. This opening was formed by electroplating a thick layer of copper onto a precision machined aluminum mandrel, and then etching away the mandrel. Before the copper electroplating, a thin (5-μm) layer of gold was evaporated on the mandrel; after electroplating and etching, this gold layer remains on the interior surfaces of the waveguide and prevents oxidation during the hot-pressing procedure.

Samples of KRS-5 and KRS-6 were machined from commercial stock[1] into billets which were slightly undersize in both thickness and transverse dimensions. A sample was then inserted into a wafer opening and pressed with an

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[1]The sources of the materials were: KRS-5, Harshaw Chemical Co., Solon, OH; KRS-6, British Drug House, Poole, England.

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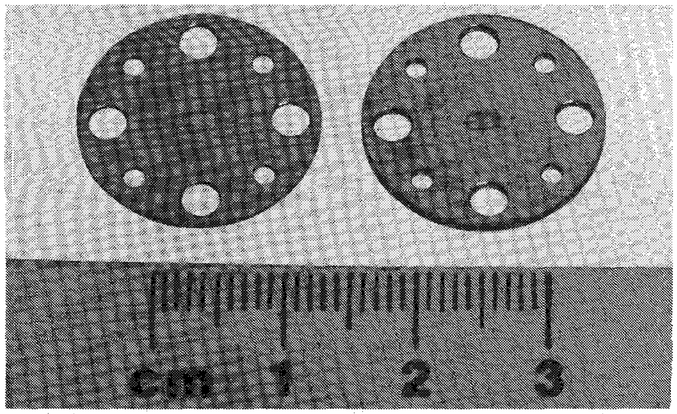

Fig. 1. Photograph of the copper-wafer-mounted samples of KRS-5.

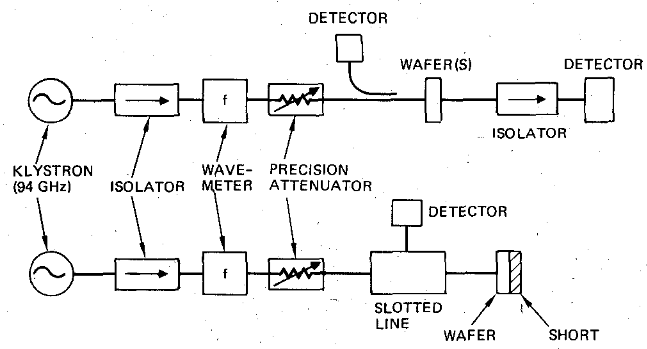

Fig. 2. Experimental arrangements used for the waveguide measurement of complex dielectric constant. (a) F–P resonances in reflection and transmission. (b) SWG method.

undersized mandrel at an elevated temperature until it expanded laterally to fill the opening. The best results were obtained by applying $\sim 2\times10^6$ kg/m^2 for periods of 6 h at a temperature of 250°C. The wafers with the sample in place were then machined to the desired thickness and lapped to obtain a flat polished surface.

The KRS-5 samples prepared in this manner were free from cracks or voids under inspection by microscope. Typical samples are shown in Fig. 1. KRS-6 is substantially less ductile than KRS-5, and the pressed samples of this material were not as free from defects. Waveguide wafers containing samples of Teflon and Rexolite were also prepared by hot pressing. Because of the high ductility of Teflon, lower values of pressure and temperature were used when pressing the material.

III. Waveguide F–P Measurements

The first measurement technique used the wafers as F–P resonators in a waveguide; different combinations of samples are inserted to vary the length of the resonator. The arrangement shown in Fig. 2(a) was employed to measure the transmission and reflection from the dielectric wafers. A waveguide isolator was used to prevent frequency pulling of the klystron source by reflections from the samples; a second isolator was used in front of the transmission detector to eliminate reflections from any detector mismatch. A reference transmission level was first established with no wafers in the system. Transmission and reflection coefficients with the wafers in place were then determined by changing the precision attenuator until the detector signals were equal to the reference level, thus eliminating detector nonlinearity as a source of error.

The power transmission coefficient at normal incidence through a plane-parallel dielectric sample filling the waveguide cross section is easily derived by the standard techniques for handling multiple beam interference in optics (see, for example, the text by Hecht and Zajac [12]). The transmission is given by

$$\frac{P_{\text{transmitted}}}{P_{\text{incident}}}=\frac{1}{\dfrac{[1-R\exp(-2\alpha L)]^2}{(1-R)^2\exp(-2\alpha L)}+\dfrac{4R}{(1-R)^2}\sin^2\beta L} \tag{1}$$

where R is the power reflection coefficient of a single air–dielectric interface and $\alpha+j\beta$ is the complex propagation constant for TE_{10} waves in the dielectric-filled region

$$\alpha=\frac{\pi}{\lambda}\cdot\frac{\epsilon_r''}{\sqrt{\epsilon_r'-\left(\dfrac{\lambda}{2a}\right)^2}}=\frac{\pi}{\lambda}\tan\delta\frac{\epsilon_r'}{\sqrt{\epsilon_r'-\left(\dfrac{\lambda}{2a}\right)^2}} \tag{2}$$

$$\beta=\frac{2\pi}{\lambda}\sqrt{\epsilon_r'-\left(\frac{\lambda}{2a}\right)^2} \tag{3}$$

where λ is the free space wavelength, a is the width of the waveguide, $\epsilon_r'-j\epsilon_r''$ is the complex relative dielectric constant, and $\tan\delta$ is the loss tangent. These expressions are valid for low-loss materials ($\tan\delta\ll1$).

The reflection coefficient R in (1) is simply the Fresnel reflection from an air–dielectric interface, modified by the change in phase velocity resulting from the presence of the metallic waveguide walls

$$R=\left[\frac{\sqrt{\epsilon_r'-\left(\dfrac{\lambda}{2a}\right)^2}-\sqrt{1-\left(\dfrac{\lambda}{2a}\right)^2}}{\sqrt{\epsilon_r'-\left(\dfrac{\lambda}{2a}\right)^2}+\sqrt{1-\left(\dfrac{\lambda}{2a}\right)^2}}\right]^2. \tag{4}$$

For $\epsilon_r'=32$ and $\lambda=3.0$ mm, $R\simeq0.76$ in a WR-10 waveguide.

Equations (1)–(4) assume, of course, that all the power remains in the TE_{10} mode as the wave passes through the dielectric-filled section, despite the fact that many higher order modes are above cutoff in that section. We argue for this simplification by noting that the planar, normal air–dielectric interfaces and the constant physical cross section of the metallic boundaries do not encourage mode conversion. Nevertheless, this could be a source of error in long sample sections.

Waveguide wall losses in the sample length L are indis-

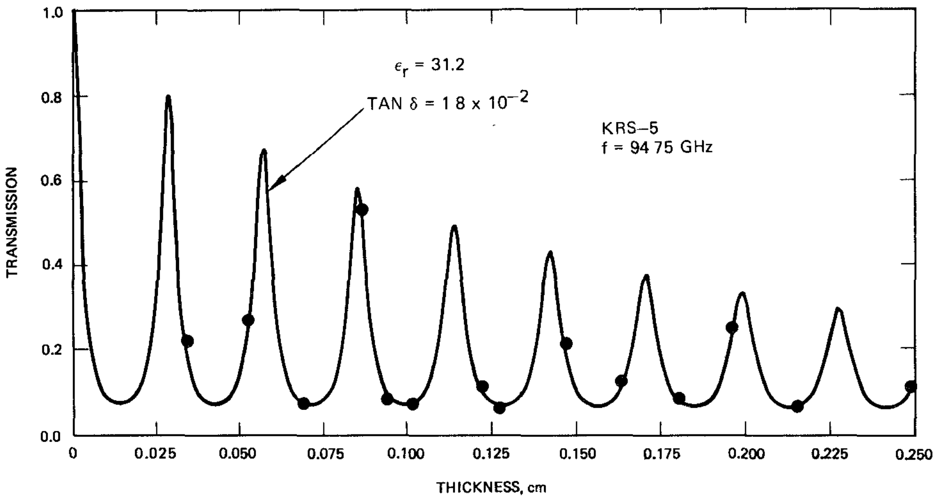

Fig. 3. Measured transmission coefficients and fitted F–P curve for KRS-5 wafer samples.

TABLE I
EXPERIMENTAL VALUES OF ϵ_r' AND TAN δ BY THE WAVEGUIDE F–P METHOD AT 94.75 GHz

Run	Material	ϵ_r'	Tan δ	Method[a]	Fit[b]	Wafer Thicknesses (mm)	Comment
1	KRS-5	31.2	1.8×10^{-2}	T	0.01	0.335, 0.526, 0.686, 0.940	
2	KRS-5	30.5	2×10^{-2}	T	0.075	0.315, 0.516, 0.678, 0.932	wafers of run #1 machined and repolished
3	KRS-5	30.4	1.9×10^{-2}	T	0.019	Same as run 2, plus 0.414, 0.947	only combinations up to 3 wafers at a time taken
4	KRS-6	28.5	2.3×10^{-2}	T	0.11	0.310, 0.358, 0.483, 0.777, 0.973	
5	KRS-6	28.9	2.3×10^{-2}	T	0.11	0.307, 0.357, 0.483, 0.775, 0.968	wafers of run #4 repolished
6	KRS-6	25.5	1.4×10^{-2}	T	0.0014	0.307, 0.357, 0.483	only thinnest 3 wafers of run #5 used
7	Teflon	2.04	9×10^{-3}	R	0.0029	0.818, 1.288, 1.849	
8	Rexolite	2.56	2.6×10^{-3}	R	0.0061	0.812, 1.285, 1.88	

Notes: (a) T = transmission, R = reflection measured to fit to theory

(b) Root mean square deviation of data points from theory.

tinguishable from bulk dielectric losses and could constitute a source of error. However, standard WR-10 waveguide loss is usually quoted as 4 dB/m, which would yield an apparent loss tangent of 4.4×10^{-4} if the dielectric filling were completely lossless. This turns out to be at least an order of magnitude smaller loss than the samples measured, and we have not corrected for it.

The transmission and reflection for all possible combinations of wafer thicknesses were measured at a fixed frequency of 94.75 GHz. These data were then used as input to a computer program that systematically varied the complex dielectric constant to yield a least-squared-error fit of the theoretical transmission or reflection coefficient to the data. To reduce the data with this program the user specifies a range of complex dielectric constant to be explored for a possible fit by specifying maximum and minimum values of α and β and the step size for each. Starting at one corner of the (α, β) space, the program computes the sum of the squared differences between the theoretical expression (1) and the measured transmissions for all samples lengths. The program repeats this calculation, stepping α through its complete range, and stores the minimum rms error found and the value of α that gives the minimum. The program then steps β and repeats this procedure. If the new minimum is less than the previous minimum, it continues to step β; if not, it prints out the previous minimum and the corresponding values of α and β. These are converted to ϵ_r' and $\tan\delta$ by (2) and (3). Some idea of the sensitivity of this method can be gained by tracking the minimum rms error as the program runs; an increment of 5–10 percent in either ϵ_r' or $\tan\delta$ away from the final value typically doubled the rms error for KRS-5 or KRS-6.

Several sets of measurements were made with the KRS-5 and KRS-6 wafers under different conditions as specified in Table I. Fig. 3 shows the data points corresponding to a specific run for KRS-5 and illustrates the quality of the fit to the theoretical transmission (solid curve). The reduced accuracy for the KRS-6 measurement is presumed to be due to sample imperfections, which had an especially strong effect when several samples were stacked together to give large thicknesses.

In order to check on the accuracy of this technique and its ability to measure still lower values of loss tangent, we also measured the dielectric constant and loss tangent of Rexolite and Teflon. The resulting values were

$$\begin{aligned} \epsilon_r' &= 2.56 \qquad \tan\delta = 3\times 10^{-3}, \qquad \text{for Rexolite} \\ \epsilon_r' &= 2.04 \qquad \tan\delta = 9\times 10^{-3}, \qquad \text{for Teflon.} \end{aligned} \tag{5}$$

The measured values of dielectric constant are in good agreement with literature values [6]–[8] for Rexolite (2.47–2.58) and Teflon (2.0–2.1), while the measured values for loss tangent are larger than the literature values [6]–[8] for Rexolite (1.2×10^{-3}) and Teflon (2×10^{-4}–3×10^{-3}). We should note, however, that the literature values cited cover the frequency range 70–400 GHz, and that the $\tan\delta$ values do not exhibit a simple increase with frequency over this range; thus it is somewhat difficult to cite an "accepted" value for $\tan\delta$ at 95 GHz. The sensitivity of the curve-fitting program was also somewhat reduced for the low-dielectric constant materials; a 10- to 20-percent change in ϵ_r' or $\tan\delta$ away from the final value doubled the rms error. In any case, our measured $\tan\delta$ values are high, and we do not known if this discrepancy is due to metallic waveguide wall loss or sample imperfection. However, since our measured values of $\tan\delta$ for KRS-5 and KRS-6 are larger still, we feel the method should be reasonably accurate for those materials.

From our experience, it appears that in the case of high-dielectric constant material, the best data are obtained from the measurement of the transmission coefficient, whereas for low-dielectric constant material the reflection coefficient should be used, and a fit made to the reflection equation analogous to (1)

$$\frac{P_{\text{reflected}}}{P_{\text{incident}}} = \frac{\dfrac{R[1-\exp(-2\alpha L)]^2}{(1-R)^2\exp(-2\alpha L)} + \dfrac{4R}{(1-R)^2}\sin^2\beta L}{\dfrac{[1-R\exp(-2\alpha L)]^2}{(1-R)^2\exp(-2\alpha L)} + \dfrac{4R}{(1-R)^2}\sin^2\beta L}. \tag{6}$$

IV. Waveguide Reflection Measurements

In a second experiment we measured the complex reflection coefficient from a single wafer inserted at the shorted end of a waveguide. An experimental arrangement similar to the one described by Roberts and von Hippel [4] was used for the reflection measurements, as shown in Fig. 2(b).

A short was placed at the end of the empty waveguide, creating a reference standing wave pattern. The position of a node was determined with a slotted line (TRG Model W740). A wafer was then inserted between the end of the waveguide and the short, and the position and magnitude of the standing wave were again determined with the slotted line. As before, the precision attenuator was used to return the detector output to the reference level, so that the VSWR accuracy depended solely on the attenuator calibration, and not on the detector linearity. The measurements were made at a frequency of 94.75 GHz.

The theoretical equation relating the shift of the VSWR minimum from the reference position and VSWR magnitude to the complex dielectric constant of the sample is transcendental and implicit in dielectric constant

$$\frac{\tanh[(\alpha+j\beta)L]}{[(\alpha+j\beta)L]} = -\frac{j\lambda_g}{2\pi L}\,\frac{\mathrm{VSWR}^{-1} - j\tan\left(\dfrac{2\pi S}{\lambda_g}\right)}{1 - j\mathrm{VSWR}^{-1}\tan\left(\dfrac{2\pi S}{\lambda_g}\right)} \tag{7}$$

where λ_g is the wavelength in the air-filled guide and S is the distance from the dielectric interface to the first node of the standing wave in the air-filled sections; S is also equal to the shift in position of the standing wave mode when the sample is inserted. As before, the assumption is that the power remains in the TE_{10} mode throughout, even though higher order modes can exist in the dielectric-filled section.

The right-hand side of (7) contains the measured quantities and is evaluated, resulting in a single complex number. The propagation constant $\alpha + j\beta$ is then determined numerically from this complex number and $\epsilon_r' - j\epsilon_r''$ from $\alpha + j\beta$ by (2) and (3). A computer program to solve these equations was written along the lines of the program used by Nelson *et al.* [9].

The values of complex dielectric constant obtained for the samples of KRS-5 and KRS-6 are given in Table II. The agreement between the various samples is quite good and provides an increased level of confidence in the results.

In order to provide a further check on our experiments, we measured the dielectric properties of Rexolite and Teflon at 95 GHz and obtained values similar to those obtained with the F–P technique. Unfortunately, the wafer-mounted samples of Teflon and Rexolite were not thick enough to yield good results. In the case of very low-loss low dielectric constant materials, it is desirable to use samples that are significantly larger physically because the additional losses when the dielectric is introduced in the waveguide must be larger than the losses due to the metallic walls. Accordingly, we cut longer samples of Teflon and Rexolite ($\sim$13 mm) for a slip fit in WR-10 waveguide from the same lots of Teflon and Rexolite used for the wafers. Our

TABLE II
EXPERIMENTAL VALUES OF ϵ_r' AND TAN δ AT 94.75 GHz BY THE SWG METHOD

Sample Thickness (mm)	Material	ϵ_r'	Tan δ
0.942	KRS-5	31.7	1.7×10^{-2}
0.940	KRS-5	31.9	1.7×10^{-2}
0.686	KRS-5	31.1	1.9×10^{-2}
0.414	KRS-5	31.5	1.6×10^{-2}
0.973	KRS-6	30.8	1.1×10^{-2}
0.777	KRS-6	31.0	3.3×10^{-2}
0.483	KRS-6	30.8	3.6×10^{-2}
0.358	KRS-6	30.8	1.0×10^{-2}
12.532	Rexolite	2.41	3.4×10^{-3}
12.517	Rexolite	2.41	3.2×10^{-3}
14.030	Teflon	1.94	4.1×10^{-3}
13.872	Teflon	1.98	4.7×10^{-3}

results with these samples were

$$\epsilon_r' = 2.4 \qquad \tan\delta = 3.3\times10^{-3}, \qquad \text{for Rexolite}$$

$$\epsilon_r' = 1.9 \qquad \tan\delta = 4\times10^{-3}, \qquad \text{for Teflon.}$$

As a check on the 10-GHz values of ϵ_r' and $\tan\delta$ quoted without reference by von Hippel [3] for KRS-5, we also made a waveguide reflection measurement at 10 GHz, using a setup similar to the one depicted on Fig. 2(b). In this case, the samples were machined to size and slipped into the end of a standard X-band waveguide. The average values for the complex dielectric constant of KRS-5 at 10 GHz were

$$\epsilon' = 30.6 \qquad \tan\delta = 4\times10^{-3}.$$

V. COMPARISON OF THE TWO METHODS

Two different methods of measurement were used primarily to gain added confidence in the results. However, it may be useful to make some comparison between the two techniques. The shorted waveguide (SWG) method requires a slotted line or other means of determining the shift in standing wave position while the F–P method does not; since slotted lines are increasingly expensive and difficult to make at shorter wavelengths, this is a definite advantage for the F–P method. On the other hand, the SWG method requires only a single sample and no curve fitting, while the F–P method requires several samples to vary βL in order to remove ambiguity and obtain reasonable accuracy by curve fitting. (The F–P method could perhaps be used with a single sample if a wide range swept frequency source were available to vary β rather than L, but this is another expensive item at millimeter wavelengths.) The SWG method has "preferred" lengths of samples (see [5]) that give more accurate results; the F–P method also should yield more accurate results with fewer sample points if the lengths happen to be resonant. The two methods should be comparable in their sensitivity to wall losses, sample finish, flatness, fit in the waveguide, etc. However, the SWG method has the added problem of a possible gap in the fit between the sample and the end short.

VI. FREQUENCY DEPENDENCE OF DIELECTRIC PROPERTIES

As stated earlier, no measurements of the dielectric properties of KRS-5 or KRS-6 above 10 GHz have been reported previously. However, measured values are available at lower frequencies, especially for KRS-5. Our measured values of ϵ_r' at 10 and 94 GHz for KRS-5 are essentially the same as the values reported by von Hippel [3] at 10^2–10^7 Hz and 10^{10} Hz. In order to compare our measured values of loss tangent for KRS-5 with the other values, we have plotted all measurements as a function of frequency in Fig. 4. It is clear that the frequency variation can be divided into two separate regimes. Below $\sim 10^8$ Hz, ionic conductivity dominates and the loss tangent varies as

$$\tan\delta = \frac{1}{2\pi f \rho \epsilon_r' \epsilon_0} \tag{8}$$

where f is the frequency and ρ is the resistivity. As expected, the data points closely follow a $1/f$ variation, corresponding to $\rho = 2\times10^8\ \Omega\cdot\text{cm}$ and $\epsilon_r' = 31$. The absorption at microwave and millimeter wavelengths appears to be dominated by the low-frequency tail of the strong lattice absorption centered at ~ 1400 GHz. If we model the lattice vibration as a single harmonic oscillator, the loss

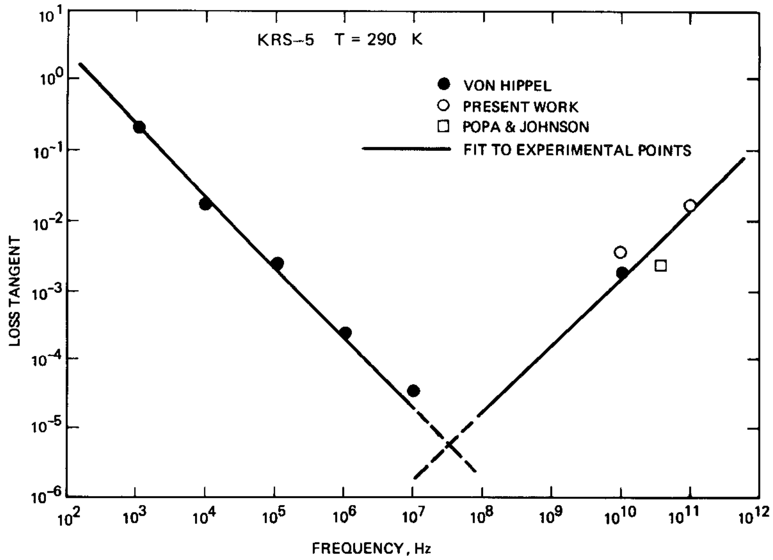

Fig. 4. Measured values of the loss tangent of KRS-5 and theoretical behavior due to ionic conductivity (10^3–10^7 Hz) and lattice absorption (above 10^7 Hz).

tangent is expected to vary as

$$\tan\delta = \frac{\gamma f}{2\pi f_0^2} \tag{9}$$

where γ is the damping coefficient (rad^{-1}) and f_0 is the resonant frequency. In Fig. 3 we have drawn a line through the data points that gives a value of $\gamma/2\pi f_0^2 = 1.5\times10^{-13}$ s. Separate measured values of γ and f_0 for KRS-5 are unavailable, but literature values [10] for TlCl and TlBr (based on dielectric constant measurements in the far infrared) give $\gamma/2\pi f_0^2 = 1.2\times10^{-13}$ and 1.1×10^{-13} s, respectively. No value is available for TlI, but we would expect it to be similar, and thus the admixture of TlI to TlBr (giving KRS-5) is also expected to be comparable. It appears that our measured value for KRS-5 may be slightly high due to extrinsic factors such as impurities and imperfections.

VII. Temperature Dependence

All of the measurements were performed at room temperature 290 K, so we can say nothing first-hand about the temperature dependence of ϵ_r' and $\tan\delta$. However, Von Hippel [3] shows ϵ_r' for KRS-5 decreasing about 5 percent from 290 K to 530 K while the loss tangent roughly doubles. We know of no data on KRS-5 below room temperature, but Lowndes and Martin [10] cite an increase in dielectric constant for TlBr from 30.4 at 290 K to 35.1 at 2 K. Lowndes [11] gives the temperature dependence of γ and ω_0 for TlBr from which we estimate that $\tan\delta$ would decrease about 10 percent from 290 K to 4 K if the losses are solely due to the FIR resonance absorption.

VIII. Summary

In summary, we have provided the first measured value of the dielectric properties of KRS-5 and KRS-6 at 95 GHz. Our measurement techniques made use of sample mounting in standard metal waveguide, thus reducing the required sample size and eliminating the diffraction problems present in quasi-optical techniques. Our sample mounting technique insures a perfect fit into the waveguide, and is applicable to a wide range of ductile materials. We are now working to extend the technique to harder materials.

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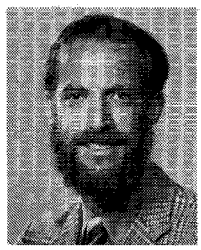

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