Measurement of the Microwave Conductivity of a Polymeric Material with Potential Applications in Absorbers and Shielding

Krishna Naishadham and Prasad K. Kadaba

Abstract —**The microwave conductivity of a new material, the polymer PBT made conductive by ion-implantation doping with iodine, is measured at 9.89 GHz as a function of temperature using the cavity perturbation technique applicable to thin films of arbitrary shape. The dc and microwave conductivities of PBT are seen to approach asymptotically the low-temperature limit predicted by Mott's energy-dependent hopping model. The potential utilization of conductive polymers in microwave absorbers and EMI shielding is examined.**

I. Introduction

A NUMBER of lightweight polymers, intrinsically nonconductive but made conductive upon doping, have been studied in recent years [1]–[3]. These materials have several potential applications, for example, in electromagnetic interference (EMI) shielding, microwave absorbers, gas sensors, display units, and junction devices [4], [5]. The polymer poly-p-phenylene-benzobis-thiazole (PBT), reported in this investigation, has been doped using the technique of ion implantation [6]. Chemical or electrochemical methods of doping were not possible for lack of a suitable solvent. The microwave conductivity of the doped polymer, in the form of a thin film, has been measured over a wide range of temperatures using the cavity perturbation method [7]. The possibility of utilizing the material as a microwave absorber and as an EMI shield is investigated.

Although the potential utilization of conductive polymers in EM applications such as shielding has been envisaged in previous publications (e.g., [3]), experimental data and analyses for the microwave characterization of these polymers are sparse. Of the highly conductive polymers, only polyacetylene seems to have received considerable attention from the standpoint of microwave conductivity [2]. Thus, besides providing measured data on the microwave conductivity of a new material, PBT, this paper examines, for the first time, the shielding and absorption capabilities of conductive polymers. Where applicable, our results on PBT will be compared with polyacetylene [2] to evaluate the usefulness of both of these polymers in EM applications.

Section II outlines the ion-implantation procedure and describes the cavity perturbation technique for the measurement of microwave conductivity. Measured results on the conductivity of doped PBT as a function of temperature are presented in Section III. It is seen that the conductivity of PBT increases by about seven orders of magnitude upon doping and varies significantly with temperature. The temperature variation of conductivity is expected to be of interest in aerospace applications. The potential utilization of PBT, as well as polyacetylene, in microwave absorbers and EMI shields is explored in Section IV by appealing to layered media EM theory.

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K. Naishadham is with the Department of Electrical Engineering, Wright State University, Dayton, OH 45435.
P. K. Kadaba is with the Department of Electrical Engineering, University of Kentucky, Lexington, KY 40506.
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II. Measurement of Conductivity

Pristine (undoped) PBT 2002-2, available in the form of a thin film of thickness in the range of 5–100 μm, is an *insulator*, characterized by an extended system of double conjugated bonds. It has a low specific gravity (1.31) and a high mechanical strength (flex strength of 12500 lb/in^2). It becomes conducting only after doping.

A. Ion Implantation

Ion implantation is a process by which a dopant can be injected into the near-surface region of a solid by means of a beam of high-velocity ions striking a target mounted in a vacuum chamber [6]. The PBT samples used in this investigation have been implanted with iodine, an electron acceptor, to a fluence of 10^{16} ions/cm^2 in an Extrion series 400 ion implanter at the Oak Ridge National Laboratory. Ion energies and corresponding doses are determined using an analytical version of the TRIM code to achieve a uniform implant distribution through the sample [8]. Iodine is chosen to dope PBT because a relatively high conductivity has been reported for polyacetylene after iodine doping [2]. The PBT film sample (typical dimensions: 5 cm×2.5 cm×6 μm) is fastened to an aluminum plate and mounted on the target block in the sample chamber of the linear accelerator, where it is bombarded with a beam of high-energy iodine ions to the desired fluence. The physical characteristics of the beam, i.e., beam diameter, beam symmetry, and beam current, are determined using the TRIM code. In order to avoid localized heating of the sample, the beam current has been maintained at less than 10 μA. After the desired fluence is achieved, the sample is annealed in argon at 200°C for 20 min to ensure stable iodine content.

B. Cavity Perturbation Method

The experimental technique for microwave conductivity measurements is similar to that used by Bauhofer [9], which is a cavity perturbation technique for thin samples of irregular shape. Compared with the original development of Buranov and Shchegolev [7], Bauhofer's technique involves a fitting procedure (see subsection II-B-2), which eliminates the need to evaluate the sample depolarization factor. This factor has a large uncertainty for arbitrarily shaped samples and, if not known exactly, results in large errors in the determination of the conductivity. The depolarizing factor can be determined exactly only for a sphere or an ellipsoid [10].

Bauhofer's method consists in measuring the *change* in the resonant frequency:

$$\delta = \frac{f_0' - f_0''}{f_0'} \quad (1)$$

and in the loss factor:

$$\Delta = \frac{1}{Q''} - \frac{1}{Q'} = \frac{1}{Q_s} \quad (2)$$

of a microwave cavity, caused by a *perturbation* of the cavity field by the film sample. In (1) and (2), a single prime denotes the resonant frequency or the quality factor of the unloaded cavity, and a double prime denotes these quantities for the sample-loaded cavity. The factor Q_s^{-1} is the loss caused by the sample. The complex permittivity $\epsilon^* = (\epsilon' - j\epsilon'')\epsilon_0$, where $\epsilon_0 = 8.854 \times 10^{-12}$ F/m, can be determined from measurements of δ and Δ at various temperatures, as shown in subsection II-B-2. The conductivity of the material is then given by $\sigma = 2\pi f_0''\epsilon''\epsilon_0$. Measurement of δ and Δ over a wide temperature range is necessary to eliminate the depolarization factor [9].

1) Experimental Setup: The experimental setup for microwave conductivity measurements on thin samples is shown in Fig. 1. The X-band microwave signal is generated in an HP 8350 synthesized sweep oscillator supplying microwave power of about 25 mW in the CW mode. A small portion of this power is coupled out for frequency measurement. The frequency is stored in the HP PC-308 Vectra personal computer, which also controls the measurement process. The main power couples into and out of the rectangular cavity, which is isolated at its input and output ports, through small circular irises (4 mm in diameter). The small diameter of the irises ensures that the cavity field is weakly perturbed. The cavity resonates at a frequency of 9.89 GHz in the TE_{103} mode. The double-walled quartz dewar of bore diameter 8 mm penetrates the cavity and serves to maintain a constant ambient temperature inside the cavity in the vicinity of the sample. The unloaded Q of the cavity plus dewar is about 2000. The sample is heated (cooled) with air heated through a coil (boiling liquid nitrogen). The temperature is measured and controlled with a 100 Ω platinum resistor and a temperature controller.

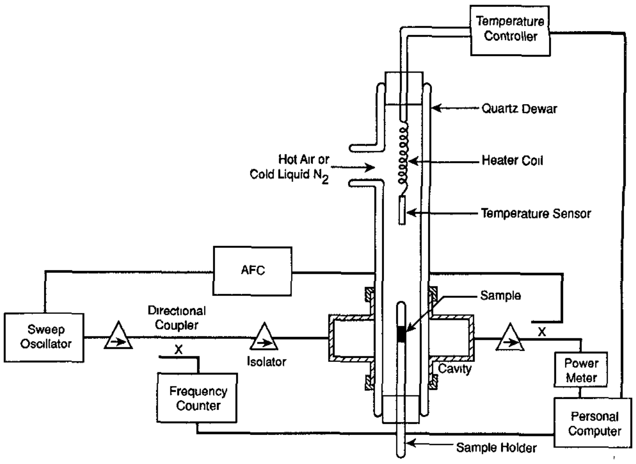

Fig. 1. Experimental setup to measure the temperature-dependent microwave conductivity of thin polymer films using the cavity perturbation technique.

The power coupled out of the cavity is measured with an HP 436 power meter using the HP 8481A bolometer head. The maximum power transmitted through the *unloaded* cavity is about 0.5 mW. A small portion of the output power is coupled out and fed into the automatic frequency control (AFC) loop, which ensures that the sweep oscillator continues to operate at the center of the cavity resonance curve when the sample is inserted.

The sample is inserted at the maximum of the electric field in the cavity. After the temperature has stabilized, the resonant frequency f_0'' and the transmitted power P_0'' at resonance are measured. Then the sample is removed, and f_0' and P_0' are measured. The parameter δ is then obtained using (1) and Δ is calculated from

$$\Delta = \frac{1}{Q'}\left(\sqrt{\frac{P_0'}{P_0''}} - 1\right) \quad (3)$$

where

$$Q' = \frac{f_0'}{F}. \quad (4)$$

The resonant frequencies f_0' and f_0'' are measured in the frequency counter, and the half-power bandwidth F is determined from the resonance curve of the unloaded cavity.

2) Determination of the Conductivity: The conductivity is determined from the measured values of δ and Δ, as outlined in [9]. Since the sample is electrically small, and its conductivity is not too high, it will be assumed that the microwave field penetrates the whole sample, so that quasi-static approximations may be used to relate ϵ' and ϵ'' to δ and Δ [7], [9]:

$$\delta = \frac{1}{2B}\left\{1 - \frac{A}{A^2 + (N\epsilon'')^2}\right\} \quad (5)$$

$$\Delta = \frac{1}{B}\frac{N\epsilon''}{A^2 + (N\epsilon'')^2} \quad (6)$$

where

$$A = 1 + N(\epsilon' - 1) \quad (7)$$

$$B = \frac{N}{2\alpha_F}. \quad (8)$$

It is also assumed that $\epsilon'' \gg \epsilon'$ or, equivalently, that the temperature variation of ϵ' can be neglected, in arriving at (5)–(8) [7]. Our measurements will prove that these assumptions are correct. Here, α_F is the filling factor, defined as [9]

$$\alpha_F = \frac{2V_s}{V_c} \quad (9)$$

with V_s and V_c denoting the sample and cavity volumes, respectively. The sample depolarization factor, N, can be eliminated from (5) and (6), as shown next. The limiting values of $\delta(\epsilon'')$ for large ϵ'' and small ϵ'' may be determined from (5) as

$$\delta_{\max} = \frac{1}{2B}, \qquad \epsilon'' \gg \frac{A}{N} \quad (10)$$

$$\delta_{\min} = \frac{1}{2B}\left(1 - \frac{1}{A}\right), \qquad \epsilon'' \ll \frac{A}{N}. \quad (11)$$

The loss factor $\Delta(\epsilon'')$ in (6) has a maximum at $\epsilon'' = A/N$, given by

$$\Delta_{\max} = \frac{1}{2AB}. \quad (12)$$

After inverting (6) for ϵ'' and substituting (10) and (12), we obtain

$$\epsilon''(T) = \left\{1 \pm \left[1 - \left(\frac{\Delta(T)}{\Delta_{\max}}\right)^2\right]^{1/2}\right\} \bigg/ \left\{\frac{\alpha_F \Delta(T)}{\delta_{\max}^2}\right\} \quad (13)$$

where the positive sign of the square root is valid for $T \geqslant T\Delta_{\max}$, the negative sign holding otherwise. The above equation does not involve the depolarization factor, N, and contains only the measured parameters α_F, $\Delta(T)$, $\Delta_{\max}$, and $\delta_{\max}$. To determine $\epsilon''(T)$ using (13), then, we measure δ and Δ at various temperatures and find $\Delta_{\max}$ and $\delta_{\max}$.

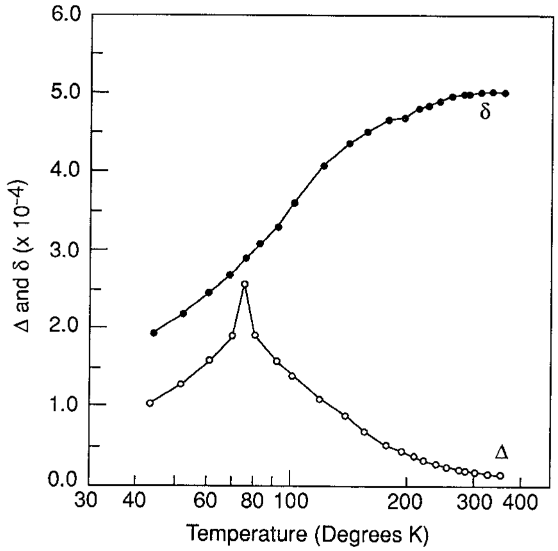

Fig. 2. Temperature dependence of the measured resonance shift δ and the loss factor Δ for a 5.8-μm-thick iodine-doped PBT film at 9.89 GHz.

III. Measured Results

Fig. 2 shows a plot of the loss factor Δ and the resonance shift δ of the cavity as a function of temperature ranging from 44 K to 350 K for an iodine-doped PBT film of 5.8 μm thickness. Several samples were measured, and only one sample which showed a loss maximum in this temperature range was selected for further analysis. When the temperature is increased, as a consequence of the temperature dependence of ϵ'', δ varies smoothly from $\delta_{\min}$ to $\delta_{\max}$, whereas Δ goes through a maximum. For this sample $\Delta_{\max}$ occurs at considerably below room temperature (300 K), and $\delta_{\max}$ occurs close to room temperature. The microwave absorption Δ peaks when the sample becomes strongly polarized by the increase in its conductivity with temperature [9]. Equations (10)–(12) yield $\delta_{\max} - \delta_{\min} = \Delta_{\max}$, which is observed to be true (within measurement tolerances) from our experimental data in Fig. 2. Thus, our initial quasi-static, or "thin film," assumption that the microwave field penetrates the whole of the sample and that $\epsilon'' \gg \epsilon'$ is validated. The permittivity of the sample computed using the procedure in subsection II-B-2 is given by $\epsilon^* = (3 - j838)\epsilon_0$ at 300 K.

The microwave conductivity of the PBT sample at 9.89 GHz is calculated using (13) and is plotted in Fig. 3 against temperature. The room-temperature conductivity is 460 S/m (compared with $\sigma \approx 10^{-5}$ S/m for the undoped sample). An interesting feature of the sample is a significant change in the conductivity (by a factor of 5–6) with temperature.

The absolute experimental error in the measurement of the microwave conductivity is expected to be around $\pm 30\%$ owing to uncertainties in the geometrical dimensions of the sample (which manifest themselves through the filling factor α_F—see (9)). Reproducibility errors in positioning the sample inside the cavity and uncalibrated errors in temperature measurement also contribute to

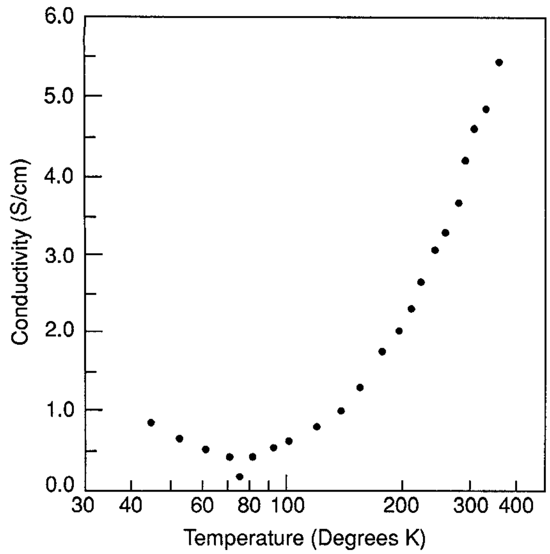

Fig. 3. Temperature dependence of the microwave conductivity at 9.89 GHz of the PBT film, computed using (13) with a filling factor $\alpha_F = 3 \times 10^{-5}$.

degradation of the measurement accuracy of the microwave conductivity. The reader is referred to [9] for a qualitative error analysis of the various factors affecting the measurement accuracy.

The dc conductivity, measured using a four-probe technique, is plotted in Fig. 4 over a temperature range of 20 K to 400 K. Also, replotted for comparison is the microwave (9.89 GHz) conductivity. The abscissa is scaled in terms of $T^{-1/4}$ (T being the absolute temperature of the sample in kelvins) to corroborate our experimental results with Mott's variable range hopping (VRH) model of conduction in polymers [11]. According to this model, log $\sigma_{\rm dc}$ is linearly related to $T^{-1/4}$ at low temperatures, which is found to be in good agreement with the experimental data in Fig. 4. Furthermore, the microwave conductivity becomes progressively higher than the dc conductivity at low temperatures, in accord with Mott's theory [11]. Similar results have been obtained for iodine-doped polyacetylene by Ehinger *et al.* [2]. The difference between the dc and microwave conductivities at room temperature is within a few percent, so that the frequency dependence of the conductivity of PBT (say, within the X band) may be neglected at room temperature. Theoretical details on the corroboration of our experimental results on PBT with Mott's VRH model can be found in [12].

IV. Applications

Potentially useful applications of conductive polymers are in microwave absorbers and EMI shielding. We consider below some implementations of these two applications, appealing to EM theory for reflection and transmission caused by a planar multilayer. Since these details can be found in any standard EM text (e.g., [13]), we relegate to the Appendix a summary of the equations relevant to our computations.

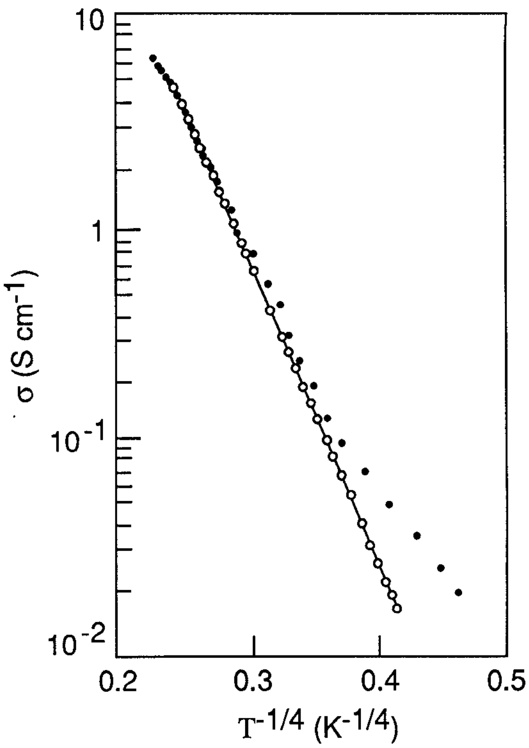

Fig. 4. Temperature dependence of dc (circles) and 9.89 GHz microwave (dots) conductivities of iodine-doped PBT.

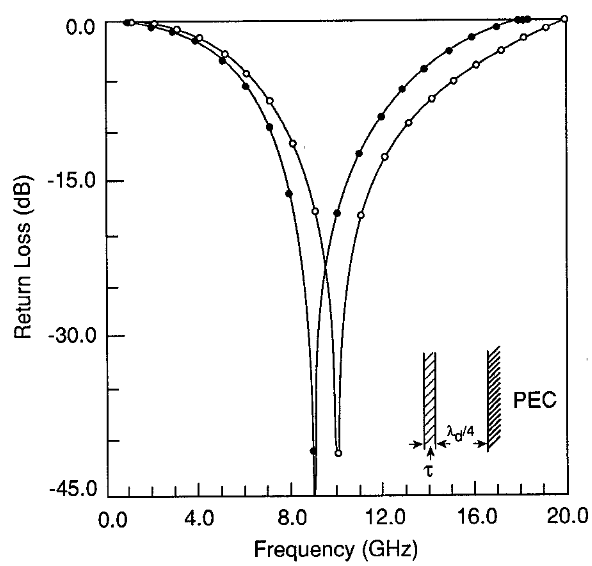

Fig. 5. Frequency dependence of return loss at normal incidence of a single layer electric Salisbury screen comprising a 377 Ω/square resistive sheet of (a) polyacetylene (dots) designed to resonate at 9 GHz and (b) PBT (circles) designed to resonate at 10 GHz. The dielectric substrate is Duroid ($\epsilon_r = 2.1$) backed by a perfect electric conductor (PEC). A wavelength in the dielectric is denoted by λ_d. The thickness τ of the polyacetylene and PBT sheets are, respectively, 8.8 μm and 5.8 μm.

A. Microwave Absorbers

A simple implementation of a microwave absorber is an electric Salisbury screen [14], which consists of a thin resistive sheet (of thickness τ and conductivity σ) deposited on a lossless quarter-wavelength-thick dielectric substrate backed by a perfect electrically conducting

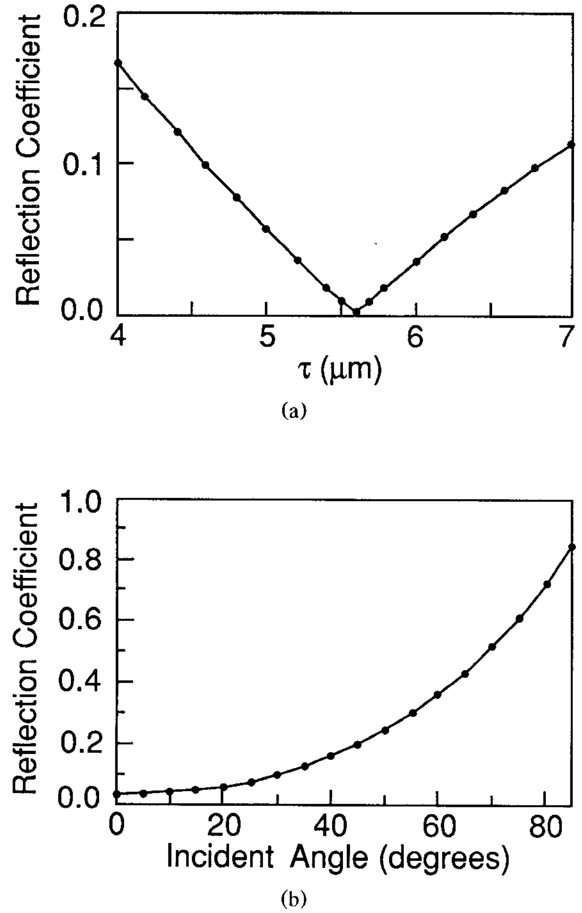

Fig. 6. (a) Reflection coefficient (magnitude) at normal incidence of a PBT Salisbury screen absorber as a function of thickness of the resistive sheet. (b) Magnitude of the reflection coefficient of a 377 Ω PBT Salisbury screen as a function of angle of incidence for perpendicular and parallel polarizations.

(PEC) plate (see the inset of Fig. 5). The reflection coefficient of this absorber can be found by tailoring (A9) to four layers (with $\sigma_4 \to \infty$). A necessary condition for zero reflection under the assumption $\epsilon'' \gg \epsilon'$ is $1/\sigma\tau = \eta_0$ [14], where $\eta_0 = 377$ Ω is the free-space intrinsic impedance. A resistive sheet with parameters which satisfy this condition is said to have an impedance of 377 Ω per square [14]. Both polyacetylene ($\epsilon_r^* = 5.1 - j607$ at 8.895 GHz [2]) and PBT ($\epsilon_r^* = 3 - j838$ at 9.89 GHz) satisfy the requirement $\epsilon'' \gg \epsilon'$. The corresponding free-space resistive sheet thicknesses are 8.8 and 5.8 μm, respectively. Fig. 5 plots the return loss of polyacetylene and PBT single-layer Salisbury screens designed to resonate for normal incidence at 9 and 10 GHz, respectively. In both cases the bandwidth for 90% absorption (−20 dB return loss) of incident microwave power is observed to be about 2 GHz. Considering that the conductivity/weight ratio of conductive polymers is typically 100–150 times that of carbon-impregnated nonmetallic composites [3], it appears that conductive polymers are advantageous as radar absorbable materials (RAM's).

Fig. 6 plots the reflection coefficient of a single-layer PBT Salisbury screen absorber as a function of the thickness of the resistive material (Fig. 6(a)) and as a function of incident angle for perpendicular polarization (Fig. 6(b)). It is seen that very thin sheets (thickness in the range 4.5–7 μm) provide 90% absorption. This translates to a sheet resistivity of 310–385 Ω per square. The reflection coefficient is small (less than 0.1) for angles of incidence up to 35°. The same angular dependence is observed for parallel polarization also.

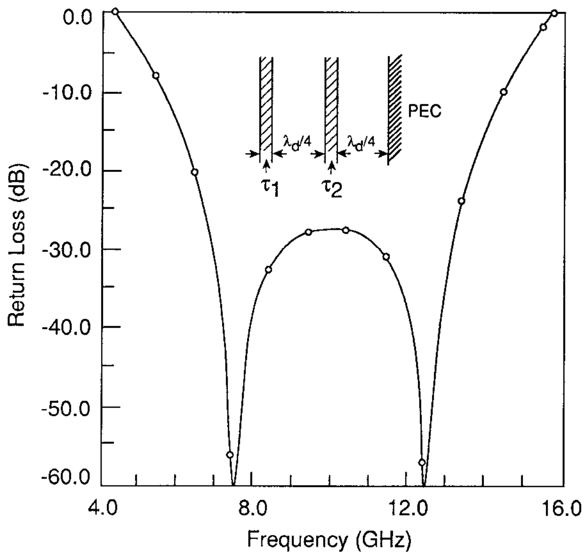

Fig. 7. Frequency dependence of return loss at normal incidence of a double layer electric Salisbury screen comprising polyacetylene and PBT resistive sheets of thickness $\tau_1 = 3$ μm (surface resistivity 1.13 kΩ/square) and $\tau_2 = 7.7$ μm (surface resistivity 282.8 Ω/square). The substrate layers ($\epsilon_r = 2.1$) are a quarter wavelength thick at $f_0 = 10$ GHz. The absorber is designed to produce zero reflection at 7.5 GHz and 12.5 GHz using a maximally flat characteristic [14].

The bandwidth of a Salisbury screen absorber can be improved by constructing a multiple screen absorber, which is analogous to a number of coupled parallel resonant circuits [14]. Fig. 7 shows the return loss of a two-layer polymer absorber, comprising thin resistive sets of PBT and polyacetylene. The bandwidth for 90% absorption is observed to be 7 GHz (in contrast to 2 GHz for a single layer). An absorber with a characteristic such as Fig. 7 can be designed using concepts in network optimization and synthesis [14].

B. EMI Shielding

In Fig. 8, the shielding effectiveness, defined by

$$\mathrm{SE} = 20 \log_{10} |T| \tag{14}$$

where T is the transmission coefficient (see eq. (A10)), of a 64-mil-thick free-standing polymer film, is plotted against frequency. Both PBT and polyacetylene films have $\mathrm{SE} < -40$ dB ($|T| < 0.01$) for frequencies greater than 4 GHz. Thus, conductive polymers have the potential for lightweight shielding of cabinets housing high-data-rate electronics, such as in supercomputers. Researchers at Los Alamos Laboratories are investigating ways of synthesizing conductive latexes that, for example, could be

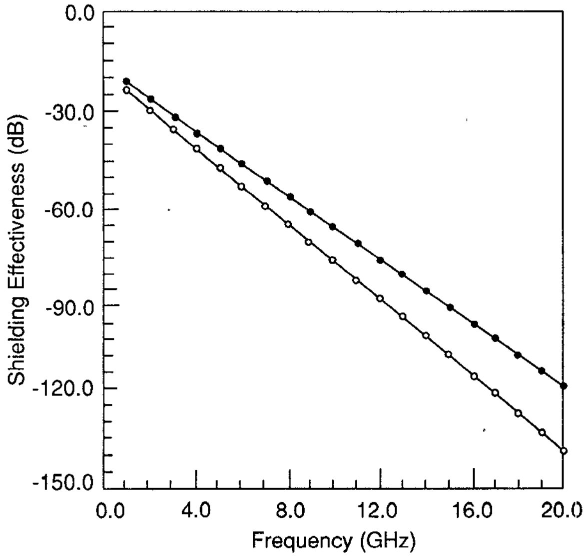

Fig. 8. Shielding effectiveness as a function of frequency for a 64-mil-thick free-standing film of polyacetylene (dots) and PBT (circles).

sprayed onto computer cabinets to prevent EMI and leakage [4].

V. Conclusions

The dc and microwave conductivities of a new material, the polymer PBT made conductive by ion-implantation doping with iodine, have been measured over a wide temperature range. A cavity perturbation technique [9] suitable for thin films of arbitrary shape is used to measure the temperature-dependent microwave conductivity of PBT at 9.89 GHz. A significant change in conductivity is observed with increase in temperature, and the conductivity of the doped sample is seen to be six to seven orders of magnitude higher than that of the undoped film. At low temperatures, the dc conductivity approaches asymptotically Mott's VRH limit of conduction in polymers [11] and is lower than the microwave conductivity. At room temperature, the dc and microwave conductivities are within a few percent, suggesting that the frequency dependence of the microwave conductivity of PBT is perhaps insignificant. Because of the narrow measurement bandwidth of the cavity technique, however, the microwave measurements reported in this paper are strictly valid in the range 9.5–11 GHz (which is the frequency range of the TE_{103} mode). As an alternative, microwave conductivity of PBT films may be measured using a network analyzer to study its frequency dependence.

The potential usefulness of conductive polymers as microwave absorbers is evaluated by studying implementations of polyacetylene and PBT thin films as multilayer electric Salisbury screens. Relatively thin films deposited on a lossless substrate such as Duroid ($\epsilon_r = 2.1$) provide near-zero reflection over a substantial bandwidth. Owing to their higher conductivity/weight ratio over carbon-impregnated nonmetallic composites, conductive polymers appear to be attractive as RAM's. The shielding effectiveness of thin films of polyacetylene and PBT is computed over a wide frequency range, and indicates that conductive polymers have the potential for lightweight shielding in an environment containing high-speed electronic circuits.

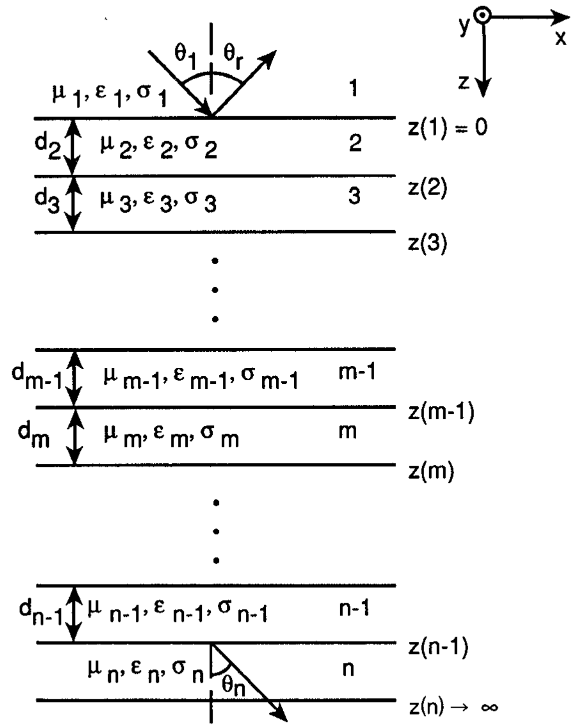

Fig. 9. Oblique incidence of a TE or TM polarized plane wave on a planar dielectric multilayer. The constitutive parameters of the jth layer are denoted by ϵ_j, μ_j, and σ_j and the thickness by d_j.

Appendix
Reflection and Transmission Caused by Planar Multilayer [14]

Consider the reflection and transmission of EM waves in a planar dielectric multilayer, shown in Fig. 9, for any incident angle θ_1. The layers are assumed to be homogeneous and isotropic, with constitutive parameters of the jth layer denoted by ϵ_j, μ_j, and σ_j and thickness denoted by d_j. The tangential fields in adjacent layers are related by the boundary conditions, through a recursive matrix equation of the form

$$\begin{bmatrix} E_{i-1} \\ H_{i-1} \end{bmatrix} = [M_{i-1}] \begin{bmatrix} E_i \\ H_i \end{bmatrix} \qquad (i = 2, 3, \cdots, n) \quad \text{(A1)}$$

where $E_i = E_y$, $H_i = H_x$ for perpendicular (TE) polarization and $E_i = E_x$, $H_i = H_y$ for parallel (TM) polarization at $z = z(i)$. The overall characteristic matrix $[M]$, which relates the tangential field at $z = 0$ to that at $z = z(n-1)$, is given by the product

$$[M] = [M_1][M_2] \cdots [M_i] \cdots [M_{n-1}] \qquad \text{(A2)}$$

where

$$[M_i] = \begin{bmatrix} \cos\alpha_i & -jZ_i \sin\alpha_i \\ \frac{-j}{Z_i}\sin\alpha_i & \cos\alpha_i \end{bmatrix} \tag{A3}$$

$$\alpha_i = k_i d_i \cos\theta_i \tag{A4}$$

$$Z_i = \begin{cases} \eta_i / \cos\theta_i, & \text{TE polarization} \\ \eta_i \cos\theta_i, & \text{TM polarization.} \end{cases} \tag{A5}$$

By Snell's law,

$$\cos\theta_i = \left[1 - (k_1/k_i)^2 \sin^2\theta_1\right]^{1/2} \tag{A6}$$

where, in the ith medium, θ_i is the angle of refraction, the wavenumber is

$$k_i = \omega\left[\mu_i(\epsilon_i + \sigma_i/j\omega)\right]^{1/2} \tag{A7}$$

and the intrinsic impedance is

$$\eta_i = \left[\mu_i/(\epsilon_i + \sigma_i/j\omega)\right]^{1/2}. \tag{A8}$$

The overall reflection and transmission coefficients can be calculated from $[M]$ as

$$R = \frac{(M_{11}Z_n - M_{12}) - Z_1(M_{22} - M_{21}Z_n)}{(M_{11}Z_n - M_{12}) + Z_1(M_{22} - M_{21}Z_n)} \tag{A9}$$

$$T = \frac{2[M_{22}(M_{11}Z_n - M_{12}) + M_{12}(M_{22} - M_{21}Z_n)]}{(M_{11}Z_n - M_{12}) + Z_1(M_{22} - M_{21}Z_n)}. \tag{A10}$$

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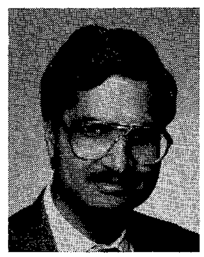

Krishna Naishadham received the M.S. degree from Syracuse University and the Ph.D. degree from University of Mississippi, both in electrical engineering, in 1982 and 1986, respectively.

From August 1986 to June 1990, he was with the University of Kentucky, Lexington, as an Assistant Professor of Electrical Engineering. In August 1990, he joined the Department of Electrical Engineering, Wright State University, Dayton, OH, where he is currently an Assistant Professor. His research interests are numerical and asymptotic methods for electromagnetic radiation and scattering problems and the microwave characterization of conductive polymers for EMC applications.

Dr. Naishadham received a Presidential Summer Faculty Research Fellowship from the University of Kentucky in 1989 and a Special Merit Scholarship from the Goverment of India in 1971. He is a member of the American Geophysical Union (AGU), Phi Kappa Phi, and Eta Kappa Nu.

Prasad K. Kadaba was born in Bangalore, India, on February 14, 1928. He received the M.Sc. degree from the University of Mysore, India, in 1947, the M.S. degree in electrical engineering from the California Institute of Technology, Pasadena, in 1949, and the Ph.D. degree in physics from the University of California, Los Angeles, in 1954.

Since 1964, he has been a Professor in the Electrical Engineering Department, University of Kentucky, Lexington. He was a Senior Fulbright Fellow at the Institute of Jozef Stefan, Ljubljana, Yugoslavia, from 1973 to 1974 and again in 1989 and he was a Visiting Professor at the Wright-Patterson Air Force Base, OH, from 1981 to 1983. His research activities are in the areas of dielectric and magnetic relaxation studies, the application of conductive polymers to microwave shielding, applications of microwave spectroscopy, nuclear double resonance techniques for pollution studies, and, currently, millimeter-wave measurement techniques.

Dr. Kadaba is a member of Sigma Xi, Eta Kappa Nu, and Tau Beta Pi.