Meas. Sci. Technol. **9** (1998) 1751–1756. Printed in the UK PII: S0957-0233(98)93119-8

A dielectric resonator for measurements of complex permittivity of low loss dielectric materials as a function of temperature

Jerzy Krupka†, Krzysztof Derzakowski‡, Bill Riddle§ and James Baker-Jarvis§

† Instytut Mikroelektroniki i Optoelektroniki PW, Koszykowa 75, 00-662 Warszawa, Poland
‡ Instytut Radioelektroniki PW, Nowowiejska 15/19, 00-665 Warszawa, Poland
§ National Institute of Standards and Technology, Radio Frequency Technology Division, 325 Broadway, Boulder, CO 80303, USA

Received 7 April 1998, in final form 20 July 1998, accepted for publication 30 July 1998

Abstract. An application of a $TE_{01\delta}$ mode dielectric resonator is described for precise measurements of complex permittivity and the thermal effects on permittivity for isotropic dielectric materials. The Rayleigh–Ritz technique was employed to find a rigorous relationship between permittivity, resonant frequency, and the dimensions of the resonant structure, with relative computational accuracy of less than 10^{-3}. The influence of conductor loss and its temperature dependence was taken into account in the dielectric loss tangent evaluation. Complex permittivities of several materials, including cross-linked polystyrene, polytetrafluoroethylene, and alumina, were measured in the temperature range of 300–400 K. Absolute uncertainties of relative permittivity measurements were estimated to be smaller than 0.2%, limited mainly by uncertainty in the sample dimensions. For properly chosen sample dimensions, materials with dielectric loss tangents in the range of 5×10^{-7} to 5×10^{-3} can be measured using the $TE_{01\delta}$ mode dielectric resonator.

Keywords: complex permittivity, dielectric resonator, Rayleigh–Ritz technique, alumina, polytetrafluoroethylene, cross-linked polystyrene, variable temperature

1. Introduction

The most accurate methods of measuring low loss dielectric materials at microwave frequencies are resonance methods employing cavity [1, 2], dielectric [3], or open resonators [4]. The presence of air gaps is one of the most important factors limiting the measurement accuracy of high-permittivity solid materials, except when the mode of interest of the electric field does not have a component perpendicular to the surfaces of the sample. This is the situation for TE_{0np} or quasi-TE_{0np} ($TE_{0\nu\delta}$) modes of cylindrical cavities and dielectric resonators, so methods employing these modes are considered to be among the most accurate [1, 3]. The quasi-TE_{011} mode of operation (often called the $TE_{01\delta}$ mode) is the mode most commonly used by manufacturers of dielectric materials for making dielectric loss tangent measurements [5]. In this work we used a resonant structure operating in the $TE_{01\delta}$ mode having geometry as shown in figure 1. With respect to former work on this type of resonator, we considered additional phenomena: (a) the influence of changes in surface resistance versus temperature on the evaluation of dielectric loss tangent, (b) the influence of thermal expansion of the sample and the cavity on permittivity evaluation, and (c) spurious modes in the frequency spectrum.

2. Theory

The Rayleigh–Ritz method is one of several rigorous techniques used in computing resonant frequencies of axially symmetric, multilateral dielectric structures [6, 7]. We have employed this technique to find a relationship between the permittivity and the $TE_{01\delta}$ resonant frequencies of a cylindrical cavity containing a cylindrical dielectric sample and a dielectric support. Empty cavity TE_{0mn} mode electromagnetic fields were used as a basis. These modes are purely rotational, so the system of linear equations with respect to unknown field expansion coefficients assumes the

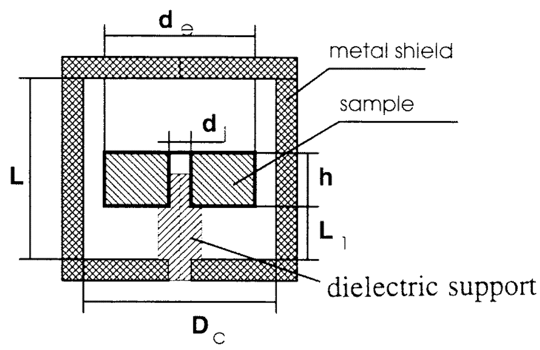

Figure 1. Geometry of $TE_{01\delta}$ mode dielectric resonator.

form

$$\left(A_{ij} - \delta_{ij}\frac{1}{\omega^2}\right)\alpha_j^{(H)} = 0 \qquad i, j = 1, 2, \ldots, N \qquad (1)$$

where $\{\alpha_j^{(H)}\}$ is the set of coefficients of the magnetic field expansion to be determined, N equals the number of basis functions, and ω is the angular frequency to be determined. The matrix $[A]$ contains elements given by the expression

$$A_{ij} = \frac{\langle \varepsilon(\rho, z)\boldsymbol{E}_i, \boldsymbol{E}_j\rangle}{\omega_i\omega_j}$$

where

$$\langle \varepsilon(\rho, z)\boldsymbol{E}_i, \boldsymbol{E}_j\rangle = \iiint_V \varepsilon(\rho, z)\boldsymbol{E}_i\boldsymbol{E}_j^* \,\mathrm{d}v$$

$\{E_i\}$ defines the set of TE_{0mn} mode normalized electric fields of the empty cavity, $\{\omega_i\}$ is the set of resonant frequencies of the TE_{0mn} mode of the empty cavity, and $\varepsilon(\rho, z)$ denotes the spatially dependent permittivity.

To find the permittivity of the sample, the eigenvalue problem shown in (1) has to be solved. Since for a given permittivity resonant frequencies of (1) can be found directly as eigenvalues of the matrix $[A]$, we used an iterative method to evaluate the permittivity for a given resonant frequency. At the last iterative step, field expansion coefficients were found as the eigenvector for the final permittivity value and given resonant frequency. The size of matrix $[A]$ depends on the number of terms used in the series expansion of the fields. In practice, we used a matrix with 126×126 elements to accommodate computer storage in one 64 kilobyte memory block in order to speed up computations, although in principle the number of basis functions can be increased if necessary. The relative numerical accuracy of the resonant frequency computations using 126 basis functions was less than 0.1%.

After the permittivity is determined, the dielectric loss tangent can be computed based on the following general expression defining the unloaded Q-factor of the resonant structure [8]:

$$Q_u^{-1} = p_{es}\tan\delta_s + p_{ed}\tan\delta_d + R_s/G \qquad (2)$$

where Q_u defines the unloaded Q-factor of the resonant structure, R_s equals the surface resistance of the conducting shield, and the terms $\tan\delta_s$ and $\tan\delta_d$ are the dielectric loss tangents of the sample and dielectric support, respectively. The geometrical factor G is defined as

$$G = \omega\frac{\iiint_V \mu_0|\boldsymbol{H}|^2\,\mathrm{d}v}{\oint\!\!\oint_S|\boldsymbol{H}_\tau|^2\,\mathrm{d}s}. \qquad (3)$$

An expression for p_{es}, the electric energy filling factor of the sample, is given by

$$p_{es} = \frac{W_{ES}}{W_{ET}} = \frac{\iiint_{V_S} \varepsilon_S\boldsymbol{E}\cdot\boldsymbol{E}\,\mathrm{d}v}{\iiint_V \varepsilon(v)\boldsymbol{E}\cdot\boldsymbol{E}\,\mathrm{d}v} \qquad (4)$$

where W_{ES} is the electric energy stored in the sample, W_{ET} is the total electric energy stored in the resonant structure, ε_S is the permittivity of the sample, and $\varepsilon(v)$ is the permittivity in the resonant structure (spatially dependent).

The electric energy filling factor of the dielectric support with permittivity ε_d is given by

$$p_{ed} = \frac{W_{ED}}{W_{ET}} = \frac{\iiint_{V_{di}} \varepsilon_d\boldsymbol{E}\cdot\boldsymbol{E}\,\mathrm{d}v}{\iiint_V \varepsilon(v)\boldsymbol{E}\cdot\boldsymbol{E}\,\mathrm{d}v} \qquad (5)$$

where W_{ED} is the electric energy stored in the dielectric support and ε_d is the permittivity of the dielectric support. In principle, the electric energy filling factors can found from equations (4) and (5); however, in our computations, we have used an incremental frequency rule [9] given by

$$p_{ed(s)} = 2\frac{\partial f}{\partial\varepsilon_{d(s)}}\frac{\varepsilon_{d(s)}}{f} \qquad (6)$$

where f is the resonant frequency.

To achieve the highest accuracy in the determination of the dielectric loss tangent based on Q-factor measurements, conductor losses and losses in the dielectric support should be kept as low as possible with respect to losses in the sample. The determination of these losses requires rigorous computations of the geometrical and electrical energy filling factors and knowledge of the material properties, such as surface resistance of the metal surfaces and the loss tangent of the dielectric supports for the whole temperature range of interest. For the resonant fixture used in our experiments, resolution of the dielectric loss tangent measurements is limited mainly by conductor losses since the support can be made small and constructed of low loss dielectric material. Equation (2) shows that conductor losses are low when the surface resistance is low and the geometrical factor is large. One well known way to minimize conductor loss is to remove the dielectric sample under test from the immediate vicinity of any conducting walls, as indicated in figure 1. Figure 2 is a graph of the computations of the geometrical factors versus the relative dimensions of the shield for the $TE_{01\delta}$ mode dielectric resonator for a sample having an aspect ratio of 2:1. This figure shows that the locations of the maxima of the geometrical coefficients depend on the value of the permittivity.

Optimum shield dimensions for dielectric loss tangent measurements of low loss materials vary with the

Table 1. Dominant mode spectra for different samples in the cavity (shield) having 35.55 mm diameter and 21.5 mm height. The cross-linked polystyrene support has a 3 mm diameter and is 8.2 mm in height.

Sample	No	Mode	Frequency (GHz)	Remarks
Cross-linked polystyrene ε_r = 2.54	1	quasi-TM_{010}	5.52	weakly coupled
d = 25.402 mm	2	quasi-HE_{111}	6.68	well coupled
h = 8.117 mm	3	quasi-TM_{011}	8.62	well coupled
	4	quasi-EH_{111}	8.78	weakly coupled
	5	quasi-HE_{211}	8.94	well coupled
	6	quasi-TE_{011}	8.98	well coupled
Alumina ε_r = 10.01	1	quasi-TM_{010}	5.40	weakly coupled
d = 11.615 mm	2	quasi-HE_{111}	7.00	well coupled
h = 8.405 mm	3	quasi-TE_{011}	7.53	well coupled
Ceramic ε_r = 34.46 d = 14.867 mm h = 4.626 mm	1	quasi-TE_{011}	4.14	well coupled

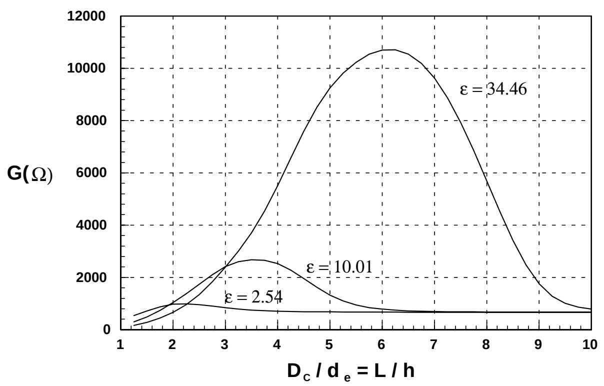

Figure 2. Geometrical factors versus relative dimensions of metal shield for $TE_{01\delta}$ mode dielectric resonators having aspect ratio $D_c/d = L_c/h = 2$.

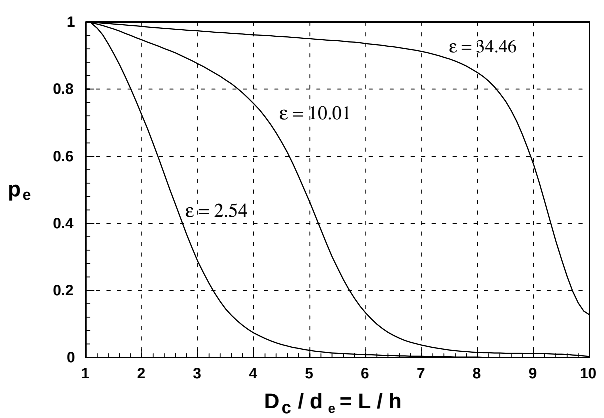

Figure 3. Electric energy filling factors versus relative dimensions of metal shield for $TE_{01\delta}$ mode dielectric resonators having aspect ratio $D_c/d = L_c/h = 2$.

permittivity of the sample and can be found as abscissa values corresponding to those maxima. For shield dimensions greater than the optimum, the electric energy filling factors decrease rapidly, and the field distribution

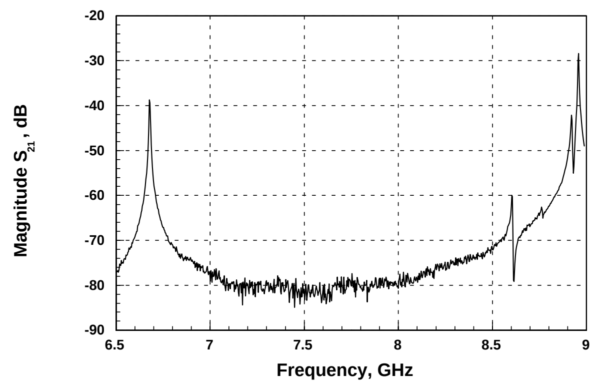

Figure 4. Measured dominant mode spectrum for cross-linked polystyrene sample.

converges to that of an empty TE_{011} cavity, as seen in figure 3. Also, $TE_{01\delta}$ mode dielectric resonators without complete shielding cannot be employed for dielectric loss tangent measurements, due to the large radiation loss.

Values of the geometrical factors for large shield dimensions and all permittivities converge to the geometrical factor of an empty cavity. As figure 2 shows, the geometrical factor for an empty cavity with an aspect ratio of 2:1 is approximately 667 Ω. In the other limit, where the dimensions of the shield equal the dimensions of the sample, geometrical factors become equal to the empty cavity value divided by the square root of the permittivity of the sample. This makes the geometrical factor small, especially for high-permittivity materials, and significantly reduces the resolution of the dielectric loss measurement. For optimum shield dimensions and a surface resistance value of 30 mΩ (copper at 10 GHz), the Q-factor due to conductor loss for a relative permittivity of 36 can be as large as 330 000. To measure the dielectric loss of the sample with good accuracy, it should be at least 10% of the overall loss. Considering a material with a relative permittivity of 36, the resolution of the dielectric loss tangent measurement is approximately 3×10^{-7} using the $TE_{01\delta}$ mode dielectric resonator technique with optimum shield dimensions.

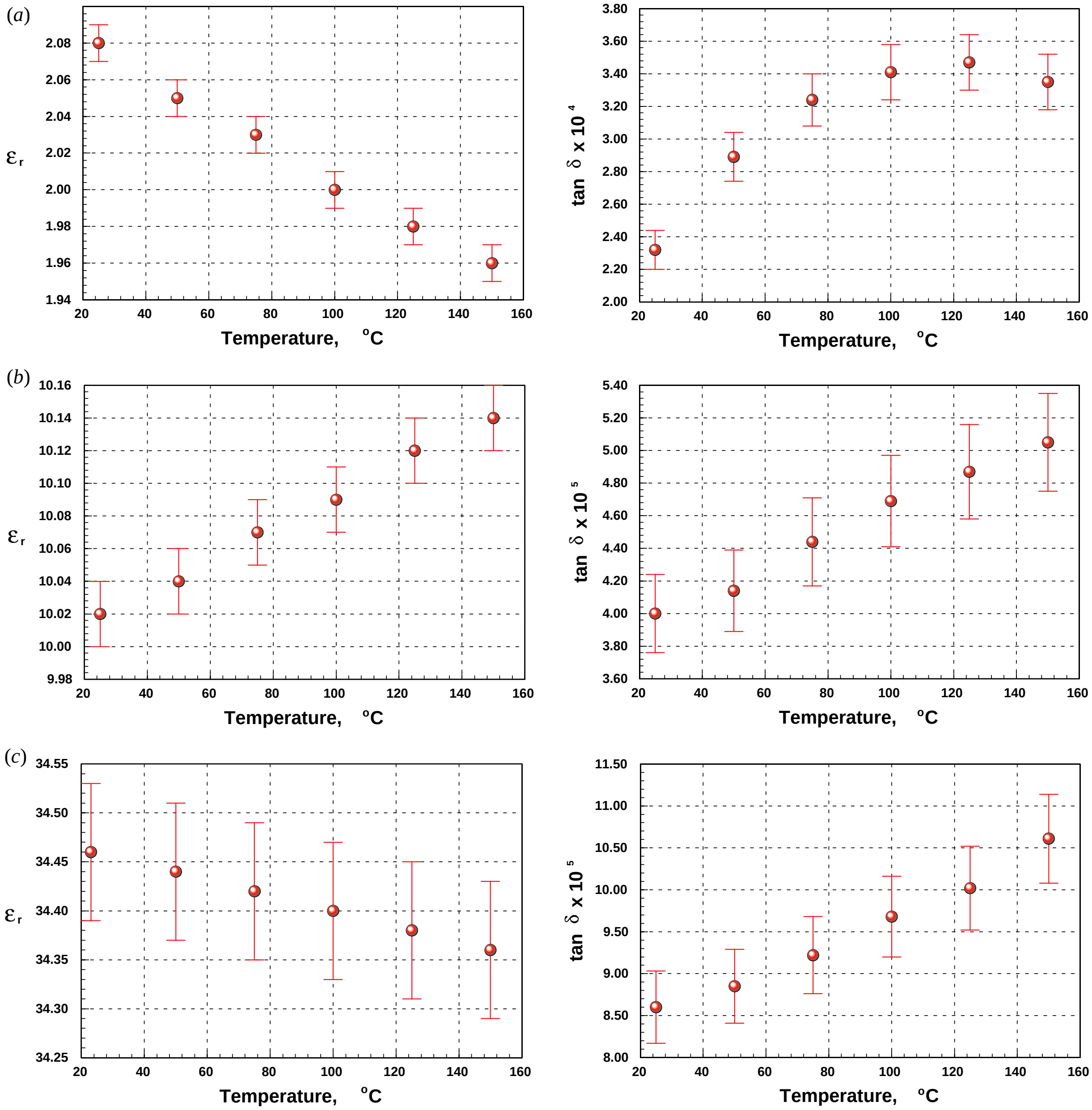

Figure 5. Relative permittivity and dielectric loss tangent of the measured samples. (*a*) PTFE, $f = 10.1$ GHz. (*b*) Alumina, $f = 7.53$ GHz. (*c*) Commercial Ceramic, $f = 4.14$ GHz.

To achieve this resolution for the dielectric loss tangent, the geometrical factor must be accurately obtained through computations. In test fixtures used by manufacturers of dielectric ceramics for measurements of the dielectric loss tangent, the shield is typically about three times greater than the dimensions of the resonator [10]. Also, the dielectric loss tangent is usually assumed to be the inverse of the measured unloaded Q-factor of the structure; this means that the influence of conductor losses on the Q-factor is negligible. For dielectric loss tangents on the order of 10^{-4}, this is an appropriate assumption, resulting only in about 10% error of the dielectric loss tangent. However, for measurements requiring higher accuracy, conductor losses cannot be neglected. For dielectric materials having dielectric loss tangents smaller than 10^{-4}, errors resulting from neglected conductor losses significantly increase with a decrease of dielectric losses, so for accurate measurements they should be taken into account, as shown in the work of Takamura *et al* [10].

3. Experimental results

Experiments have been performed in a cylindrical cavity with a diameter of 35.55 mm and a height of 21.5 mm. The cavity was constructed of silver-plated copper. Cylindrical samples with an internal hole diameter of 2 mm were placed on a cross-linked polystyrene support 8.2 mm from the base of the cavity. Two variable horizontal loops provided coupling of the resonant structure to a network analyser through semirigid coaxial cables. The cavity

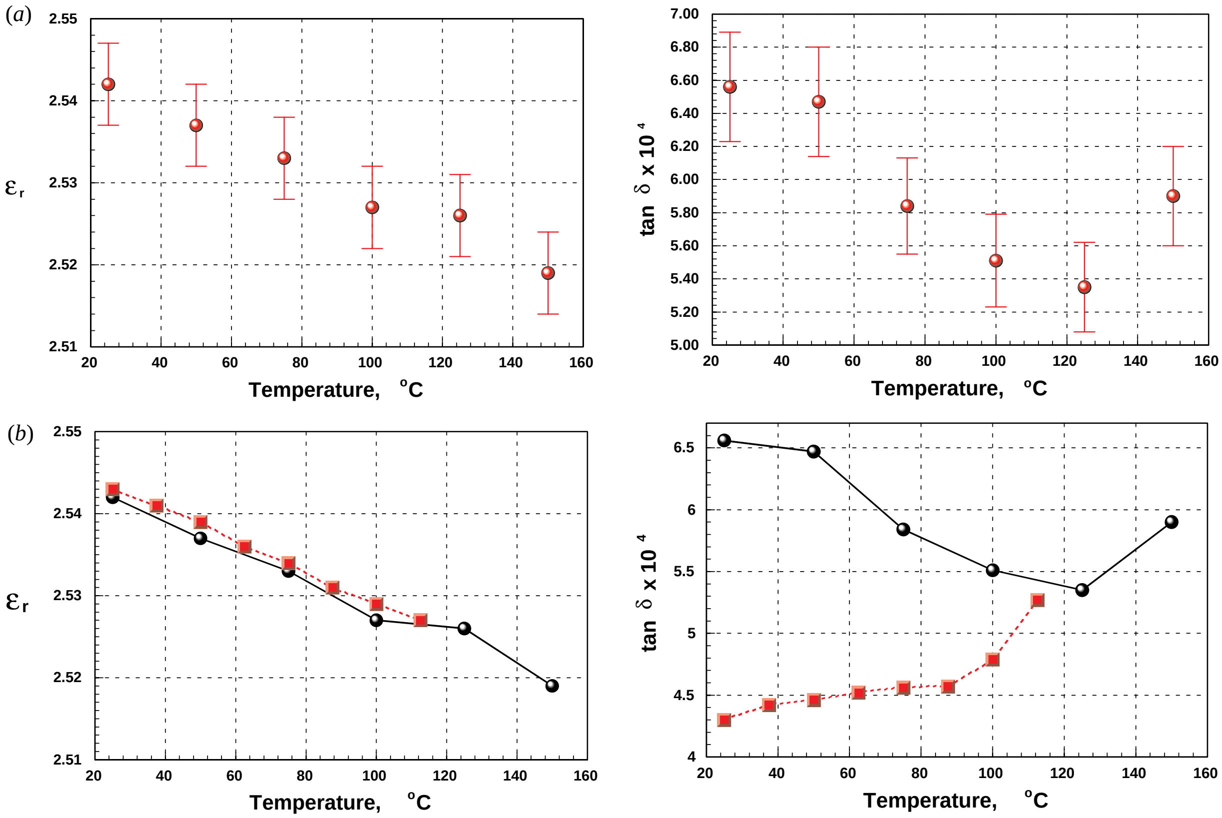

Figure 6. Relative permittivity and dielectric loss tangent of cross-linked polystyrene; f = 8.98 GHz. (*a*) Data for the initial heating cycle. (*b*) Comparison of results for two consecutive heating cycles; the circles/full lines for the first cycle, and the squares/broken lines for the second cycle.

was placed into a variable temperature-controlled chamber and the resonant frequencies and unloaded Q-factors were measured in the temperature range of interest. One of the important factors at the initial stage of the measurement was proper mode identification of the correct resonance. The $TE_{01\delta}$ mode resonance can be dominant (lowest) for high-permittivity samples or higher for low-permittivity samples. Its sequence on the frequency axis depends on the size of the sample and its permittivity value. In table 1, the results of resonant frequency computations for all modes lower than the $TE_{01\delta}$ mode are presented. The $TE_{01\delta}$ mode is lowest for the high-permittivity ceramic sample, while it is the sixth resonant mode for the cross-linked polystyrene sample. One of the physical reasons for such behaviour is the depolarization effect that takes place for all modes except the $TE_{0\upsilon\delta}$. For the $TE_{0\upsilon\delta}$ mode, the electric field has only tangential components with respect to the surface of the sample, so there is no depolarization and the resonant mode occurs at the lowest frequency. For low-permittivity samples, depolarization factors are weak, so the mode sequence is similar to that of an empty cavity. This explains why the $TE_{01\delta}$ mode of the cross-linked polystyrene sample is the sixth resonance appearing on the frequency axis. A graph of the mode spectrum measurements of the cross-linked polystyrene sample is shown in figure 4.

After the $TE_{01\delta}$ mode was identified, its resonant frequency and unloaded Q-factor were measured. To obtain precise measurement data of sample permittivity versus temperature, we calculated the dimensions of the metal (copper) cavity and the sample at each temperature based on the thermal expansion coefficients of copper and the sample material. For dielectric loss tangent calculations, we took into account surface resistance changes of the silver plating versus temperature based on unloaded Q-factor measurements of an empty silver-plated cavity versus temperature. Results of measurements of permittivities and the dielectric loss tangents versus temperature for different materials are shown in figures 5 and 6.

In addition to the materials listed in table 1, we also measured a sample of polytetrafluoroethylene (PTFE). Temperature variable measurements made by Ehrlich [11] at lower frequencies indicate a similar negative slope for ε_r versus temperature, providing a qualitative check for our results. The two ceramic materials measured, alumina and a commercial ceramic, provided data for determining the repeatability of the measurement system since the electrical properties of these materials were not affected by repeated exposure to the high temperatures. Measurements made previously by Courtney [3] on an alumina sample over the same temperature range show very good agreement with our results.

The sample of cross-linked polystyrene exhibited a significant change after being heated, as indicated in figure 6. These graphs show measured data of the sample for two sequential cycles of heating, starting from ambient room temperature. These data imply that heating cross-linked polystyrene creates irreversible physical changes that affect the electrical properties of the material. The significant change observed in the loss tangent data could be due to chemical changes induced by the high temperatures, or possibly the evaporation of bound water in the material. Published data at lower frequencies [12] provide a qualitative comparison in the sense that the dielectric loss tangent values follow a similar trend. Values for the thermal coefficients of expansion for the various materials were obtained from published values in the literature [13–15].

The estimated uncertainties for the measurements are based only on the dominant sources of error for each parameter, and arrived at through variational calculations with the numerical code. The primary source of error for the relative permittivity measurement is the uncertainty in the sample dimensions, estimated to be 0.1% at all temperatures for all of the materials except PTFE. At higher temperatures, PTFE exhibits unpredictable changes in its shape, which we account for by using a higher-dimensional uncertainty of 0.2%. The contributions of these dimensional errors resulted in estimated permittivity uncertainties of 0.4% for PTFE and 0.2% for the other materials. Errors in the measurement of the dielectric loss tangent are most affected by uncertainties in the Q factor, which we estimated to be less than 5%. This translated into an estimated uncertainty for $\tan\delta$ of 5% for all samples except alumina. Because this material has very low loss, the uncertainty in dielectric loss tangent is affected to some extent by losses in the cavity walls. As a result of this, our estimated uncertainty for the $\tan\delta$ of alumina was 6%. All uncertainty calculations were performed assuming a 25 °C environment, and that the effects of the variable temperatures could be ignored.

4. Conclusions and summary

The $TE_{01\delta}$ mode dielectric resonator technique, with properly chosen shield dimensions, provides a very accurate single-frequency measurement of relative permittivity and dielectric loss tangent for isotropic materials with loss tangents in the range of $5 \times 10^{-3} < \tan\delta < 5 \times 10^{-7}$. To achieve this operating range for loss tangent, conductor losses have to be accounted for in the measurements. The application of thermal expansion coefficients of the conductors and the materials under test are also required to produce an accurate evaluation of relative permittivity versus temperature.

References

[1] Vanzura E J, Geyer R G and Janezic M D 1993 The NIST 60-millimeter diameter cylindrical cavity resonator: performance evaluation for permittivity measurements *Natl Inst. Stand. Technol. Technical Note* 1354

[2] Bussey H E and Steinert L A 1958 Exact solution for a gyromagnetic sample and measurements on a ferrite *IEEE Trans. Micro. Theory Tech.* **6** 72–6

[3] Courtney W E 1970 Analysis and evaluation of a method of measuring the complex permittivity and permeability of microwave insulators *IEEE Trans. Microw. Theory Tech.* **18** 476–85

[4] Cullen A L and Yu P K 1971 The accurate measurement of permittivity by means of an open resonator *Proc. R. Soc.* A **325** 493–509

[5] Dielectric resonators—A designer guide to microwave dielectric ceramics, Trans-Tech Inc. Publication No 50080040, Rev. 2, October 1990

[6] Nikolskij V V 1967 *Variational Methods for Electrodynamic Problems* (Moscow: Science) (in Russian)

[7] Krupka J 1989 Resonant modes in shielded cylindrical and single-crystal dielectric resonators *IEEE Trans. Microw. Theory Tech.* **37** 691–7

[8] Kajfez D and Guillon P (ed) 1990 *Dielectric Resonators* (Vector Fields) pp 327–77

[9] Kobayashi Y, Aoki Y and Kabe Y 1985 Influence of conductor shields on the Q-factors of a TE_0 dielectric resonator *IEEE MTT-S Int. Microw. Symp. Dig.* 281–4

[10] Takamura H, Matsumoto H and Wakino K 1989 Low temperature properties of microwave dielectrics *Proc. 7th Meeting on Ferroelectric Materials and Their Applications (Japanese Journal of Applied Physics 28)* Supplement 28-2, pp 21–3

[11] Ehrlich P 1953 Dielectric properties of Teflon from room temperature to 314 °C and from frequencies of 10^2 to 10^5 c/s *J. Res. Natl. Bur. Stand.* **51**

[12] Aras L and Baysal B M 1984 Dielectric relaxation studies of some linear crosslinked and branched polymers *J. Polym. Sci.* **22**

[13] Swenson C A, Roberts R B and White G K 1985 *Thermophysical Properties of Some Key Solids (CODATA Bulletin 59)* ed G K White and M L Minges (Oxford: Pergamon) ch 4

[14] White G K 1993 Reference materials for thermal expansion: certified or not? *Thermochim. Acta* **218** 83–99

[15] von Hippel A (ed) 1995 *Dielectric Materials and Applications* (London: Artech)