

Microwave measurements upon uniaxial anisotropic dielectrics - theory and practice.

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1 Introduction

A wide variety of dielectric materials can be anisotropic, ranging from composite materials with woven or fibrous structure, through biological tissues and optically active crystals (eg. quartz) to materials with 'frozen-in' stress. In such materials the complex permittivity is a tensor quantity. The simplest form of anisotropy occurs in *uniaxial* materials in which the tensor can be reduced to a diagonal matrix having only two independent permittivities to characterise the material fully: there is one permittivity for electric fields parallel to the optic axis of the material, $\epsilon_{\parallel}$, and another, $\epsilon_{\perp}$, for electric fields orthogonal to the optic axis. Many materials from the classes mentioned above exhibit this simplest form of anisotropy. NPL has had a limited capability for anisotropic measurements for some time. The NPL open resonators can measure degrees of anisotropy, $|1 - \epsilon_{\parallel}/\epsilon_{\perp}|$, as low as parts in 10^4 . However, they are suitable only for millimetre-wave low-loss dielectrics. By contrast, conventional waveguide transmission-cell measurements can detect anisotropy in higher loss dielectrics but a number of different specimens are needed, which can be costly.

NPL has recently completed a DTI-funded project to broaden coverage of anisotropic measurements. A method has been developed in which a single square uniaxial specimen is enclosed in a cell at the end of dielectrically loaded waveguide. The specimen is rotated into two orthogonal orientations in order to obtain sufficient measurement information to characterise its anisotropy. Uncertainties of $\pm 2\%$ in permittivity are possible but degree-of-anisotropy can be measured more sensitively. The measurement system has been modelled by a novel modal analysis of the fields inside anisotropically loaded waveguide. This has highlighted the drawbacks of applying isotropic-dielectric theory to measurement of anisotropic materials, even as an approximation. The technique has been tested in the frequency range 3.5 GHz - 12 GHz. A complementary 'slab-line' method has also been evaluated for measurements at lower frequencies.

2. Open-ended Waveguide Probes.

Open-ended coaxial sensors for non-invasive measurements of complex permittivity have been in use at NPL for over a decade [1]. The main technique described in this paper was originally conceived as an extension of this convenient method. Coaxial sensors cannot measure anisotropy because of the circular symmetry of the fields they launch into the dielectric-under-test. However, an open-ended waveguide in which fundamental-mode radiation is propagating can launch a near-linearly polarised field into the region of the dielectric close to the sensor face. The use of open-ended waveguide for dielectric probes is not in itself novel, a number of authors have published papers on this and related techniques for measuring *isotropic* dielectrics [eg. 2, 3]. However, the application to anisotropic dielectrics requires a more comprehensive numerical model which has been provided in the work described here. While the use of open-ended transmission line (whether coaxial or waveguide) is convenient, an analysis in which the probe opens into a potentially 'semi-infinite space' of the dielectric-under-test is only relevant for lossy materials, because of the effects of reflections from sample boundaries in less lossy dielectrics [1]. The analyses followed here are all for a rectangular waveguide feeding into a rectangular or square cell or cavity, see Figure 1. This analysis does not exclude probe-type measurements because a large cavity can be used to model an infinite half-space of lossy dielectric.

Measurements were required to be performed up to 6 GHz, but two considerations led to the adoption of sealed dielectrically-loaded British Waveguide-16 (WG16 - normal working range 8.2 - 12.4 GHz) as the basis for the sensor. Firstly, it was required (if only for calibration) to operate with liquid dielectrics, which

must not be allowed actually to enter the waveguide. Secondly, it is always an advantage in dielectric measurements to design techniques for smaller, rather than larger specimens. These considerations led to the design of Figure 1. By using BK7 glass as the dielectric infill ($\epsilon' \approx 6.3$), working frequencies for WG16 fall to 3.5 - 6.0 GHz. Note, however, that all of the techniques described here also apply to air-dielectric waveguide and measurements were also performed with an air-filled WG16 probe in the frequency range 7-12.4 GHz. The same dielectric samples can be used for the glass-filled probe, 3.5 - 6 GHz, and the air-filled probe, 7 - 12.4 GHz.

The measurement method for anisotropic dielectrics required that the specimen be rotated to two orthogonal orientations in relation to the waveguide. This led to the adoption of a *square* cross-section cell at the end of the sensor with *square* specimens to fit. Cells of different thicknesses were mounted symmetrically at the end of the waveguide, all nominally having sides of length equal to the waveguide width. Measurements were performed at ambient temperatures via flexible test-cables on an Automatic Network Analyser (ANA). A computer-based least-squares ANA calibration method was employed because it enabled a wider range of standards to be used for cross-checking of calibration validity. It was found that the most reliable calibrations were based on 'standards' which were actually the short-circuited square metal cells themselves, filled with air dielectric. A number of such cells of different thickness gave sufficient spread of calculable reflection coefficient phases for ANA calibration. The thicknesses used were 0.942 mm, 1.981 mm and 5.007 mm, a fourth standard being a short-circuit fitted directly to the face of the sensor. It was established that for dielectric measurements, thicker specimens gave superior results, and so most solid specimens were machined to fit the 5.007-mm cell.

3 Computer Programs

The computational task was tackled in two stages. Firstly a program, called 'RWPA', was written by one of the present authors (G T Symm) to model the probe/cell geometry for *isotropic* dielectrics only. Secondly a program for uniaxial *anisotropic* dielectrics, 'WGANISO', was written, using entirely different principles, by another of the present authors (T E Hodgetts). To help check the operation of these programs with isotropic dielectrics, a third program, 'CORREC', was also purchased from *Antenna Software Ltd*. As in the NPL research on coaxial sensors, *numerical intercomparison* of independently written programs is regarded as the best method of checking the validity of computations and also the most viable method of ensuring that program-based measurements can be fully traceable to national standards.

The isotropic program 'RWPA' was based upon an algorithm originated by Saed *et al.* [4] and adopted at NPL [1] for modelling coaxial lines with cylindrical cavities at the end. The analysis was modified for the TE and TM-modal decomposition associated with rectangular waveguide. The fields in waveguide and cell are formally described by the summation of an infinite number of such modes. In practice, the summation must be truncated to cover a finite number of modes and a major part of the evaluation of 'RWPA' was concerned with how this could be done to produce errors in computation which would be acceptably small when compared with measurement uncertainties. In the coaxial program a quadratic extrapolation algorithm [1, 5] was employed for this. The waveguide case is more complex but a simple linear extrapolation was found to suffice when the maximum TE/TM mode subscript used was 11.

The version of 'CORREC' (V1.01C) employed was specially written by Dr Soe Min Tun [6] (*SMT Consultancies Ltd.*) to an NPL specification to meet the requirements of this project. An advantage of 'CORREC' is that it enables one to model the effects of air gaps in the cells, whereas 'RWPA' and 'WGANISO' assume complete filling of the cells. However, 'CORREC' runs more slowly than 'RWPA' and, as only object code can be purchased, 'CORREC' cannot be included efficiently in an iterative loop to perform 'inverse' computations which derive an unknown ϵ^* from a measured Γ (rather than *vice-versa*).

The third program, 'WGANISO', is the only one which can model uniaxial dielectrics. It represents the fields in the waveguide by *Hertzian vectors* [7] aligned along the optic axis of the dielectric, rather than along the propagation direction or waveguide axis, as is more conventional - and as is done in 'RWPA'. Failure to take this step prevents analytical separation of the 'ordinary' and 'extraordinary' fields in anisotropic materials. In the waveguide/cell geometry shown in Figure 1 the solution is obtained by mode-

matching at the interface plane between the waveguide and cell to arrive at a stationary variational expression. The total electric field at the interface is subjected to the Rayleigh-Ritz treatment [7] to give a sequence of computable approximations, which is extrapolated to its infinite limit to supply the final relationship between Γ , ϵ_1 and ϵ_2 . The entire analysis must be repeated with the optical axis taken in the orthogonal cross-guide direction, to correspond to the physical rotation through 90° of the anisotropic specimen in its second measurement. In 'WGANISO' the waveguide and cell are assumed to have the same width and to be joined symmetrically. It should be noted, however, that the fundamental form of this analysis can potentially be applied to a much wider range of measurement geometries.

Direct numerical comparisons of output from programs provides one of the best methods available for ensuring that they are individually operating correctly. A number of computations with cells of various geometries were performed to compare 'CORREC' and 'RWPA'. Generally, computational discrepancies were at least three-times better than expected measurement uncertainties. Similar agreement was obtained between 'RWPA' and 'WGANISO' for isotropic dielectrics. Table 1 provides just one example:

Computations for $\epsilon^* = 20.0 - j 20.0$				
Frequency (GHz)	Program 'RWPA'		Program 'WGANISO'	
	Magnitude: $ \Gamma $	Phase: $\arg(\Gamma)^\circ$	Magnitude: $ \Gamma $	Phase: $\arg(\Gamma)^\circ$
3.5	0.388	166.08	0.387	165.88
4.0	0.353	170.92	0.354	170.72
4.5	0.371	174.31	0.364	174.13
5.0	0.399	173.78	0.398	173.61
5.5	0.423	170.96	0.421	170.76
6.0	0.435	167.30	0.434	167.08
6.5	0.437	163.72	0.436	163.45

Table 1. Computed reflection coefficients from 'RWPA' and 'WGANISO' for a $22.9 \times 22.9 \times 5.007$ -mm cell filled with a dielectric having complex permittivity $\epsilon^* = 20.0 - j 20.0$.

4. Comparison of Measurements with Program Predictions.

Initial measurements were performed upon isotropic materials as a check. Some results at 5 GHz are summarised in Table 2. The measured and estimated permittivities agree within estimated uncertainties. Note, however, that the quoted uncertainties do not include a contribution for the effect of air-gaps between specimen and sensor face for solid specimens. For *higher* permittivity solid specimens, measurements systematically gave lower permittivity values than are estimated because of these air-gaps - see BK7 glass in the table. One way to avoid this problem is to use liquid-immersion measurements [1] in which the gaps are filled with liquid of similar permittivity to the solid. When this was done for BK7 glass, using ethanol filling liquid, a permittivity, ϵ_m^* , of $(6.34 \pm 0.13) - j (0.22 \pm 0.02)$ was measured by the probe, compared with an estimated value, ϵ_e^* , of $(6.30 \pm 0.05) - j (0.04 \pm 0.01)$.

Similar isotropic comparisons were performed over a range of frequencies from 3.5 - 12 GHz with air-filled and dielectrically loaded probes. As with the measurements in the table, they demonstrated the basic soundness of the technique. When one wishes to repeat such comparisons with *anisotropic materials*, there is a limited number of 'standard' materials to be used. One suitable material, however, is quartz crystal. This is a uniaxial material with two orthogonal permittivities, the real parts of which are $\epsilon_1' = 4.43$, $\epsilon_2' = 4.63$. Quartz has been well characterised at 10 GHz and above in NPL cavities and open resonators [8]. It has a low loss ($\tan \delta < 40.10^{-6}$), well below the resolution of the probe technique. The reflection coefficient magnitude from the specimen, $|\Gamma|$, is virtually unity and the sole information in the measurement is carried by the phase

Material Measured (isotropic materials only)	Complex Permittivities at 5 GHz		
	Estimated ϵ_r^*	Notes on ϵ_r^*	As measured ϵ_m^*
High density polyethylene (HDPE)	2.365 ± 0.005 $-j 0.000 \pm 0.001$	I T R	2.34 ± 0.04 $-j 0.01 \pm 0.02$
'Acetal' polymer	2.88 ± 0.04 $-j 0.10 \pm 0.02$	I T	2.91 ± 0.06 $-j 0.10 \pm 0.02$
BK7 glass	6.30 ± 0.05 $-j 0.04 \pm 0.01$	I R	6.17 ± 0.13 $-j 0.05 \pm 0.02$
ethanol liquid at 23.7°C	5.05 ± 0.04 $-j 3.59 \pm 0.03$	T R L	5.05 ± 0.05 $-j 3.66 \pm 0.04$
methanol liquid at 23.7°C	12.60 ± 0.08 $-j 12.15 \pm 0.08$	T R L	12.8 ± 0.09 $-j 12.0 \pm 0.09$

Table 2. Measurements on isotropic dielectric materials using the glass loaded sensor with a cell of size $23.02 \times 23.02 \times 5.007$ -mm. Tabulated values are averaged over a number of measurements. **Key to the 'Notes' column:** 'T': ϵ_r^* was measured on an ANA in coaxial transmission line at frequencies close to 5 GHz, 'I': the estimated value is interpolated between higher (≥ 10 GHz) and lower (≤ 3 MHz) frequencies ('I T' signifies a value estimated by both techniques). Materials labelled 'R' are regarded as standard reference materials for dielectric traceability. 'L' refers to liquids, which do not suffer from air-gap problems during measurement.

of Γ , $\arg(\Gamma)$. The two dashed curves in Figure 2 trace out $\arg(\Gamma)$, when ϵ_1' and ϵ_1'' are used, respectively, in the *isotropic* program 'RWPA' as an *isotropic* permittivity for the material in a $23.02 \times 23.02 \times 5.007$ -mm cell. The continuous traces are obtained from 'WGANISO', which preserves the explicitly anisotropic nature of the specimen. 'RWPA' clearly fails to function adequately because it assumes that *all three* orthogonal permittivities change from 4.43 to 4.63 when the sample is rotated - clearly an unphysical model.

Figure 3 compares the predicted phases for quartz crystal with actual measurements. The agreement with the predictions of 'WGANISO' is excellent, especially for the change in phase between the two crystal orientations, $\Delta(\arg(\Gamma))$. Between 4 and 6 GHz the average predicted difference is $1.51^\circ \pm 0.04^\circ$, whereas the average measured difference is $1.49^\circ \pm 0.025^\circ$. The reproducibility of phase for a given orientation can be better than $\pm 0.05^\circ$ for a specimen like quartz crystal. Similar agreement was obtained in the frequency range 7.5 - 12 GHz with the air-filled sensor. The quartz measurements provide the most compelling evidence that the waveguide sensor technique operates correctly for anisotropic materials. A range of other anisotropic materials was measured by this method, but the full benefit of intercomparisons, as in Table 4 and as for quartz crystal, was not always possible. These measurements will be summarised at the conference.

5. Discussion of Findings and of Other Methods

The comparison between 'RWPA' and 'WGANISO' in Figure 2 demonstrates how important it is to use a full anisotropic analysis when anisotropic materials are being measured. In that figure, and in Figure 3, it was shown that $\Delta(\arg(\Gamma))$ is significantly smaller than the figure predicted by the isotropic program. A related counterintuitive finding of 'WGANISO', confirmed by measurement, is that at some frequencies $\Delta(\arg(\Gamma))$ can actually fall to zero: ie. the anisotropic specimen may be rotated by 90° and *no difference* in Γ can be measured. It is therefore recommended that all measurements of anisotropy by this and related methods be carried out over a significant frequency range to ensure that adequate resolution is obtained.

Hodgetts' anisotropic analysis employed in 'WGANISO' is, as far as the present authors are aware, original and it is important to emphasise that it can be extended to other measurement geometries, for example, to transmission measurements upon solid anisotropic specimens which fill a rectangular waveguide.

The method described above is limited in frequency range by its being a waveguide method, subject to a low frequency cut-off. This sensor method was therefore complemented by a feasibility study on a 'slab-line' sensor. This is effectively a transformed coaxial sensor with a 'slab' inner conductor which supports near-linearly polarised electric fields on either side. TEM propagation is possible in this sensor, which therefore has no low frequency cut-off. The geometry is too complex for analytic modelling and so Finite Difference Time Domain Modelling (FDTD) was employed to relate Γ to ϵ^* . The results of the 'slab line' measurements upon anisotropic materials will be reported at the conference.

Full evaluation of the implications of the work on both sensors is an ongoing task, but the findings so far demonstrate that both methods can usefully complement measurements in open resonators, transmission cells and by free-wave transmission techniques for the evaluation of anisotropic dielectrics.

6. References

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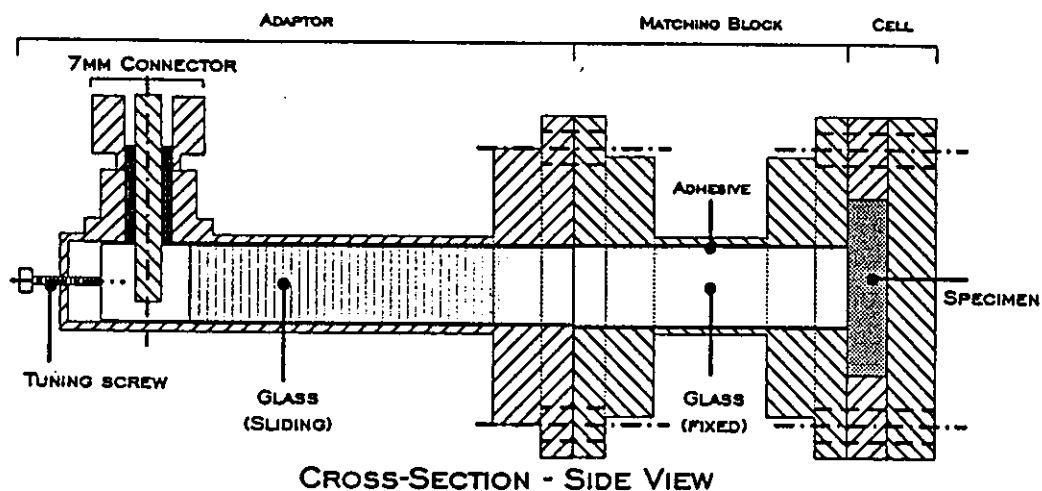


Figure 1. The dielectrically-loaded waveguide sensor and cell.

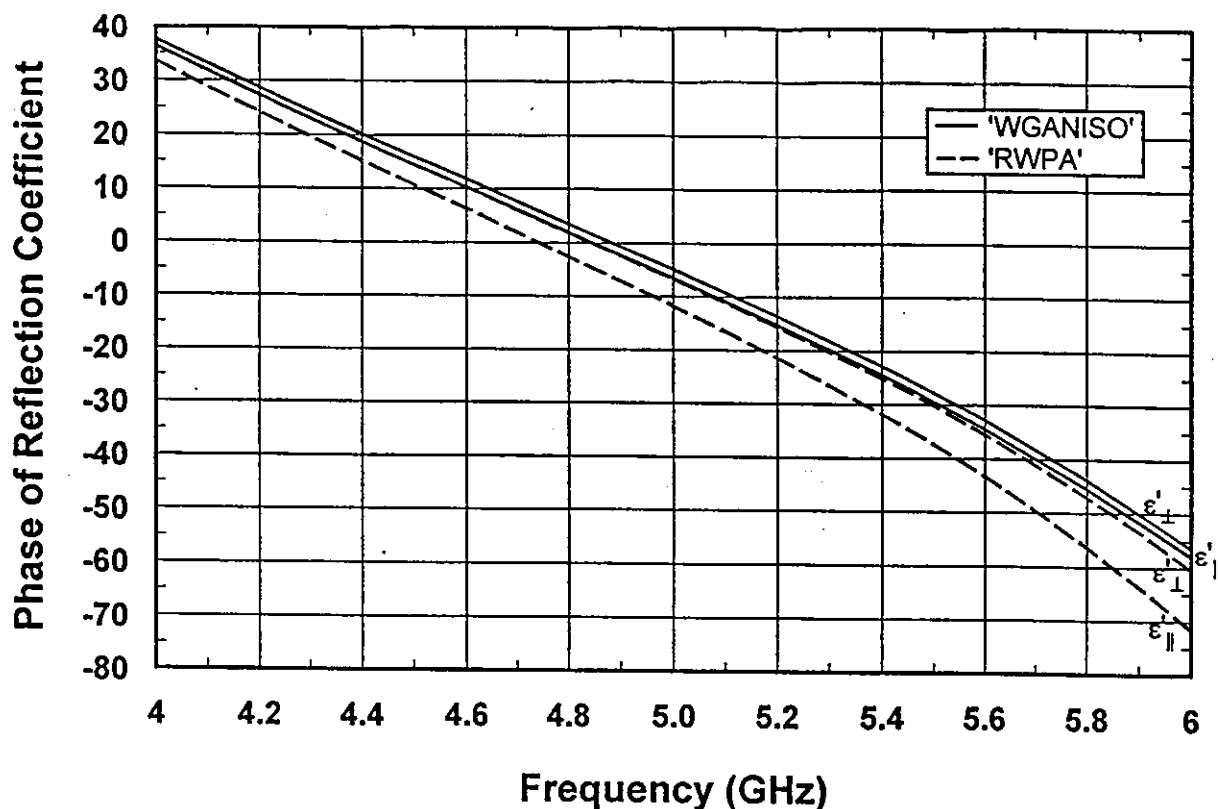


Figure 2. Comparison of predicted waveguide probe reflection coefficients from quartz crystal computed by the two programs 'RWPA' and 'WGANISO'. BK7-glass loaded WG16 probe with 5.007-mm cell.

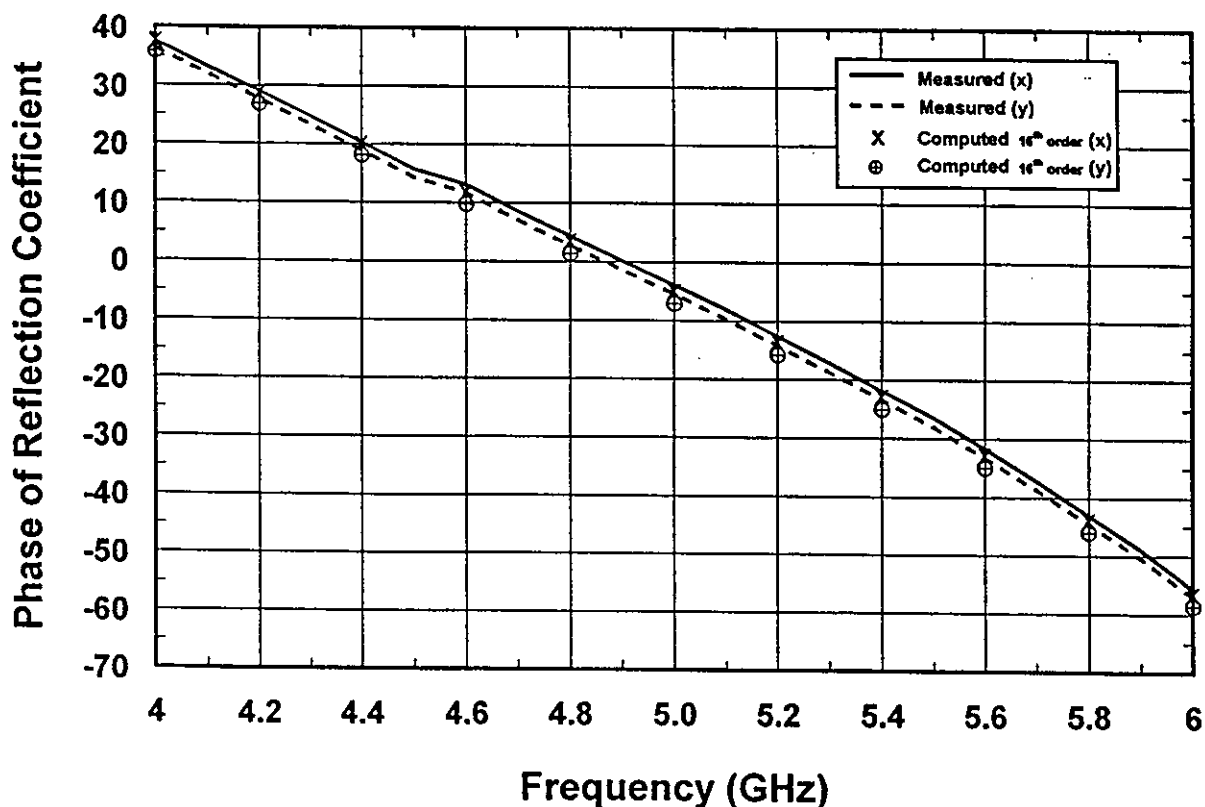


Figure 3. Comparison of predicted and measured reflection coefficients from quartz crystal for the same waveguide/cell geometry as Figure 2. The small absolute offset in phase is caused by small air-gaps but the measured phase difference, $\Delta(\arg(\Gamma))$, agrees well with that predicted by 'WGANISO'.