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Evaluation of a VNA-based Material Characterization Kit at Frequencies from 0.75 THz to 1.1 THz

Ata Khalid, David Cumming
School of Engineering,
University of Glasgow,
Glasgow, UK
Email: ata.khalid@glasgow.ac.uk

Roland Clarke
School of Electronic and Electrical Engineering,
University of Leeds,
Leeds, UK

Chong Li, Nick Ridler
Electromagnetic Technologies Group, National Physical Laboratory,
Teddington, Middlesex, UK

***Abstract*—This paper describes an initial assessment of a commercially available THz material characterization kit (MCK). The assessment is based on the measurement of several material samples. The MCK comprises two conical waveguide horn transitions and two sections of low-loss corrugated waveguide. A gap between the two corrugated waveguides allows the material samples to be inserted into the system during measurement. The MCK is attached to a THz Vector Network Analyzer (VNA), which measures S-parameters, in the frequency domain, of a material under test (MUT). A computer-based algorithm employing an iterative calculation derives values for material parameters (e.g. permittivity) from the measured S-parameters of the MUT. A MCK has been evaluated over the frequency range 0.75 THz to 1.1 THz, to assess the plausibility of results that can be obtained using such a technique. Two VNAs utilizing frequency extender heads were used for the investigation, with measurements being made with reference to a range of different calibration techniques and different calibration standards. Whilst some of the results obtained look reasonable, a significant proportion of the results were either difficult to interpret or showed inexplicable (i.e. non-physical) behavior. This indicates that much work is still needed before this technique can be used routinely for the measurement of material parameters at these very high frequencies.**

Keywords—Vector Network Analyzer; Calibration; Terahertz; Material characterisation

I. Introduction

Recent advances in optical and electronic systems have enhanced the development and application of the so-called 'terahertz region' (THz) of the electromagnetic spectrum [1]. The application of THz technology has expanded from the traditional area of astronomy to newer application areas such as imaging, security, communications, pharmaceuticals, and many more. For measurements of the dielectric properties of materials at THz frequencies, methods have traditionally been based on spectroscopy techniques. More recently, operating frequencies of vector network analyzers (VNA) have been extended into the terahertz region (i.e. to 1.1 THz). In addition, a material characterization kit (MCK) and associated software has recently been developed by SWISSto12 that interfaces with these VNAs to enable S-parameter measurements made by a VNA to be converted into the equivalent material parameters (e.g. permittivity, loss tangent, etc) [2]. This is similar to free-space material characterization approaches that have been used widely at microwave frequencies (see, for example, [3]). However the use of these VNA-based techniques at higher frequencies (i.e. at millimeter-wave and terahertz frequencies) has not yet been fully evaluated.

In this paper, we present some preliminary results from an initial investigation into the performance of a MCK operating at frequencies from 0.75 THz to 1.1 THz. Measurements were made of several different types of material. A selection of these results are presented in this paper.

II. Experimental details

The investigation was carried out using a MCK supplied by SWISSto12. The MCK was used in conjunction with two VNAs. Both VNAs were fitted with waveguide extended heads that enabled measurements to be made in the WM-250 waveguide band (which operates from 0.75 THz to 1.1 THz). One of the VNA / extender heads configurations was situated at the University of Leeds, UK (as shown in Fig. 1a); the other VNA / extender heads configuration was situated at the University of Glasgow, UK (as shown in Fig. 1b). The use of two different VNA / extended heads arrangements, situated at physically different locations, enabled a preliminary assessment of the sensitivity of the measurement method to different hardware platforms. In addition, several calibration techniques employing different calibration standards were used with both VNAs to explore the sensitivity of the material measurement technique to variations in instrument calibration.

Fig. 1c shows a schematic diagram of the MCK / VNA configuration [2] used by both universities. Each half of the MCK comprises a waveguide transition from rectangular waveguide to corrugated circular waveguide, followed by a section of low loss corrugated waveguide. This design allows the THz test signal to propagate within an enclosed low-loss environment. The MUT is inserted between the two corrugated waveguides. One half of the MCK, shown on the left in Fig. 1c, is fixed, while the other half, on the right in Fig. 1c, is moveable (to accommodate the insertion of the MUT). This makes it possible to measure MUTs of different thicknesses. However, it is a requirement of the measurement technique that the thickness of the MUT is known in order that the material parameters can be derived from the S-parameters measured by the VNA.

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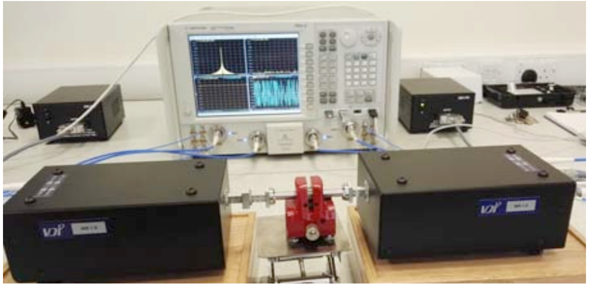

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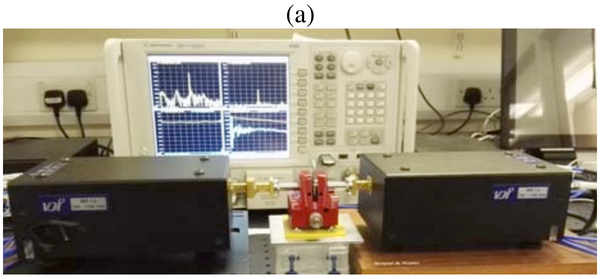

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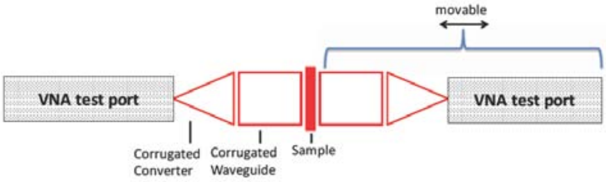


(c)

Fig. 1. Measurements set-ups: (a) University of Leeds; (b) University of Glasgow; (c) Schematic diagram [2]

Similar to other VNA-based free-space material characterization systems, the MCK requires a suitable calibration procedure prior to making any measurements. The calibration method has two tiers; the first tier is used to establish error-corrected reference planes at the VNA frequency extender ports. This can be done by using one of the conventional two-port calibration methods such as thru/reflect/line (TRL) [4], line/reflect/line (LRL) [5] or short/offset-short/load/thru (SOLT) [6]. The second tier calibration removes errors between the input/output of the MCK and establishes the material test port reference planes. The gated-reflect-line (GRL) calibration technique [7] is used for this purpose. This technique utilizes the time-domain capability that is found on most modern VNAs. The GRL method requires a simple zero-length 'Thru' standard and a metallic reflecting plate with known thickness, as the 'Reflect' standard [7]. The 'Thru' standard is measured by closing the gap between the two halves of the corrugated waveguides. The 'Reflect' standard comprises a 1 mm thick metal shim that is placed between the two halves of the corrugated waveguides. After a complete two-tier calibration of the MCK / VNA set-up, a transmission/reflection algorithm is used to compute the complex permittivity of the MUT [3].

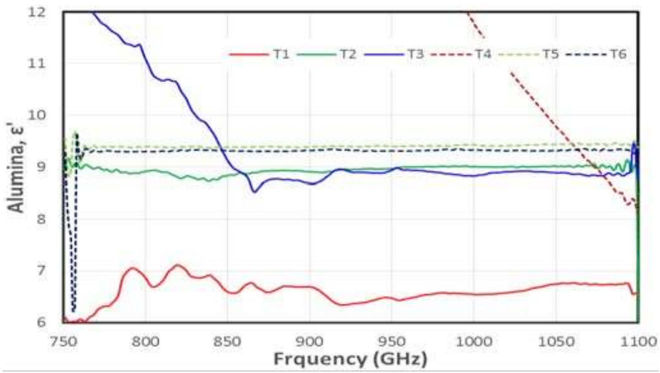
As part of the initial assessment of the performance of the MCK / VNA arrangement, the sensitivity of the measurement results to variations in the first-tier calibration was investigated. This was achieved by using different VNAs, calibration methods and calibration standards. Two VNA / extender head set-ups were used: Set-up A – situated at the University of Glasgow; Set-up B – situated at the University of Leeds. Both VNAs were manufactured by Keysight Technologies and both pairs of extender heads were manufactured by Virginia Diodes, Inc (VDI). In addition, three calibration techniques (and associated calibration standards) were used: Cal 1 – SOLT [6] using standards manufactured by VDI, belonging to the University of Glasgow; Cal 2 – SOLT [6] using standards manufactured by VDI, belonging to the University of Leeds; Cal 3 – LRL [5] using standards manufactured by SWISSto12, belonging to the University of Leeds and NPL. The combination of two different VNAs and three different calibration techniques resulted in 2 × 3 = 6 different test conditions (T1 to T6).

III. RESULTS

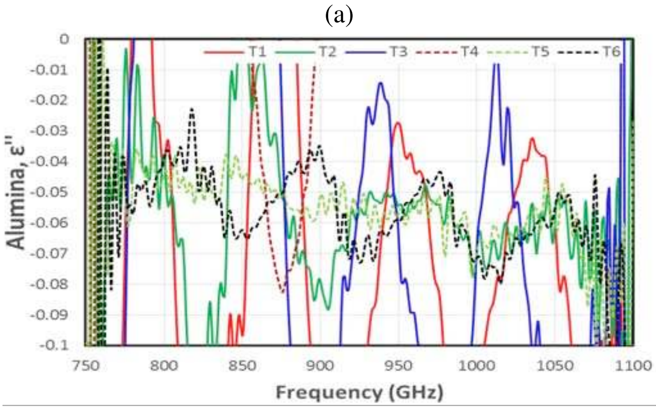
Table 1 summarizes the test plan using the different VNA set-ups and calibration schemes. Figs. 2a and 2b show some typical results obtained using the different set-ups and calibration schemes for measurements of a sample of alumina. The thickness of the alumina sample is 0.63 mm.

TABLE I. SUMMARY OF TEST PLAN, SHOWING TESTS T1 TO T6

VNA set-up	**Calibration scenarios**		
	Cal 1	**Cal 2**	**Cal 3**
A	T1	T2	T3
B	T4	T5	T6



(a)



(b)

Fig. 2. Measured relative permittivity of alumina using the MCK: (a) real part, ε'; (b) imaginary part, ε''

IV. Discussion

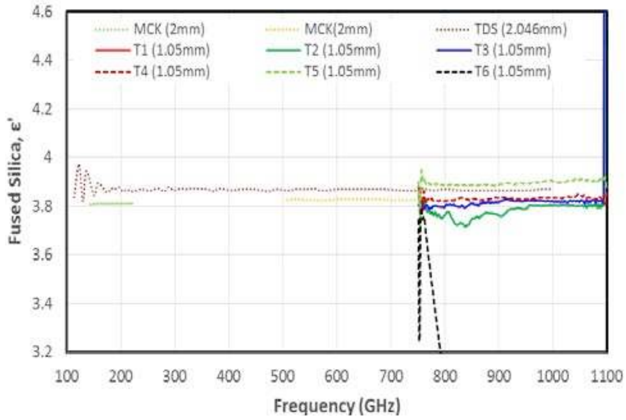
Figs. 2a and 2b indicate considerable variation between the results obtained under the various different test conditions, T1 to T6. Values for the real part of the relative permittivity, ε', for alumina given in reference data (see, for example, [8]) are in the range between 8 and 11. Therefore, some of the measured values shown in Fig. 2a (i.e. T2, T5, T6) show reasonable agreement with such reference data, across most of the frequency range. However, some of the results shown in Fig. 2a (i.e. T1, T3, T4) do not agree with these reference values. In addition, T3 and T4 show a dramatic variation with frequency over the lower part of this frequency range. It is considered that this behavior is non-physical (i.e. not real) and therefore indicative of significant problems with the measurement process.

Fig. 2b (which shows the imaginary part of the relative permittivity, ε''), reveals considerable variation between all the results. Under these circumstances, it is difficult to discern a reliable consensus value for ε'' from any of these measurements – there is also 'ringing' (i.e. oscillating) in this data, as a function of frequency, which again is likely to be due to errors and instabilities in the measurement process, rather than due to the physical behavior of the MUT.

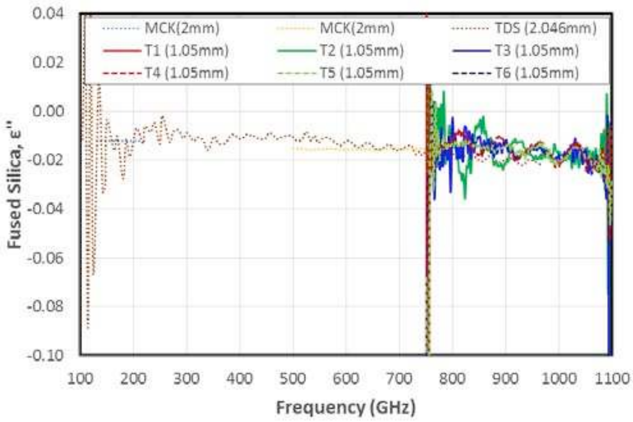
For comparison, Fig. 3 shows the measured results for a sample of fused silica (Fig. 3a shows results for ε' and Fig. 3b shows results for ε''). The thickness of the sample is 1.05 mm. Also included on these graphs are values obtained by other researchers, on previously occasions, [9] over different frequency ranges. Note the thickness of the fused silica sample used in [9] was 2 mm. The previous work involved using MCKs manufactured by SWISSto12 at WR-05 (140 GHz to 220 GHz) and WM-380 (500 GHz to 750 GHz), and, a THz Time-Domain Spectrometer (TDS) manufactured by MenloSystems [9]. The results for fused silica using the MCK at frequencies from 0.75 THz to 1.1 THz show some agreement with these other measured values, but, as before, some of the results show both erratic and non-physical behavior. Note that T1 in both Fig.3a and Fig.3b and T6 in Fig.3b are off the scale in these graphs.

Results obtained for other materials that were measured during the investigation exercise reported in this paper (i.e. TPX, indium phosphide and high resistivity silicon), also showed similar behavior to that observed above for alumina (Fig. 2) and fused silica (Fig. 3). However, examination of all this data showed that there was no obvious correlation between the occasions when the observed results were very poor, and, the different VNA configurations – i.e. the choice of VNA / MCK (i.e. Set-ups A or B); or, the choice of calibration technique and associated calibration standards (Cals 1, 2 or 3). This indicates that the unusual behavior observed during this investigation exercise is unlikely to be due to the VNA instrumentation and calibration, at least at the first-tier calibration stage.

Some further investigations indicated that the calculation procedure that is used to determine permittivity (both ε' and ε'') from the measured S-parameters is very sensitive to the assumed thickness of the MUT. However, there are likely to be additional factors that contribute significantly to the erratic behavior observed in the results obtained during this evaluation exercise. This requires further investigation – for example, by undertaking a full measurement system sensitivity analysis.



(a)



(b)

Fig. 3. Measured relative permittivity of fused silica: (a) real part, ε'; (b) imaginary part, ε''. Also included are results obtained by other researchers using MCK systems at different frequencies and the TDS method

V. Conclusions

This paper has presented some preliminary results obtained from an initial investigation into the performance of a commercially available material characterization kit that is used in conjunction with a THz vector network analyzer. The investigation concentrated on measuring material properties (i.e. permittivity, ε' and ε'') of several different types of sample in the frequency range from 0.75 THz to 1.1 THz (i.e. corresponding to the WM-250 waveguide band). The investigation has produced some interesting observations:

1. With regard to determinations of ε', the MCK seemed capable of producing measured values that are within a plausible range (e.g. as given in reference data [8]). However, the MCK can also produce measured values that are considered to be unrealistic (i.e. non-physical) and erratic (exhibiting excessive variation).
2. With regard to determinations of ε'', the measured values produced by the MCK lacked repeatability and so it was difficult, with any degree of confidence, to

derive consensus values for ε" from the various sets of measured values.

3. An investigation into the effect of changes in the VNA instrumentation – specifically, the choice of VNA, VNA calibration, and calibration standards – on the performance of the MCK did not lead to any firm conclusions regarding correlation between these variations in the VNA instrumentation and the resulting quality achieved for the materials measurements.

In summary, the MCK is capable of achieving plausible results. However, more work is needed (both experimental and analytical) in order to understand the conditions required under which reliable results may be obtained from the MCK.

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