GHz characterisation of dielectric properties of ultra-low temperature co-fired ceramic materials for 5G systems application

Marzena Olszewska-Placha
QWED Sp. z o.o.
Warsaw, Poland
molszewska@qwed.eu

Dorota Szwagierczak
Lukasiewicz Research Network-Institute of Microelectronics and Photonics, Krakow Division
Krakow, Poland
dorota.szwagierczak@imif.lukasiewicz.gov.pl

Beata Synkiewicz-Musialska
Lukasiewicz Research Network-Institute of Microelectronics and Photonics, Krakow Division
Krakow, Poland
beata.synkiewicz.musialska@imif.lukasiewicz.gov.pl

Janusz Rudnicki
QWED Sp. z o.o.
Warsaw, Poland
jrudnicki@qwed.eu

Jan Kulawik
Lukasiewicz Research Network-Institute of Microelectronics and Photonics, Krakow Division
Krakow, Poland
jan.kulawik@imif.lukasiewicz.gov.pl

***Abstract*—This work is dedicated to non-destructive characterization of dielectric properties of novel ultra-low temperature co-fired ceramic materials. With regards to their targeted application to emerging 5G communication systems, high accuracy measurements of complex permittivity are of crucial importance for materials manufacturers and further, 5G components designers. For high system efficiency, the discussed materials are aimed at low dielectric constant, in the range of 4-6.5 and low loss tangent, being within the range of 0.0005-0.005. Complex permittivity extraction is performed with the aid of split-post dielectric resonator method, which has been known for its robustness and high accuracy.**

Keywords—material measurements, non-destructive testing, resonant method, ULTCC, 5G materials

I. Introduction

Continuous progress in research and development observed in materials science is stimulated and aims at responding to rigours expectations defined by new emerging technologies. In the recent years much attention is given to efficient materials for green energy and organic electronics [1] and new, high efficiency communication systems, based on 5G and 6G technologies. For the emerging communication systems, the focus is given to low temperature co-fired ceramics (LTCC) and novel ultra-low temperature co-fired ceramics (ULTCC) [2], which attract continuously growing interest over the last years. With lower sintering temperature, compared to well-established high temperature co-fired ceramics (HTCC) technology, and keeping compatibility with already existing material fabrication methods, the LTCC and ULTCC materials are foreseen to deliver enhanced manufacturing flexibility, miniaturization, and packaging degree, along with lower production cost, higher sustainability, and environmental friendliness.

Precise characterization with regards to complex permittivity constitutes one of the crucial stages in the material development and composition optimisation process, serving as a feedback loop to material science engineer.

Part of this work was funded by the Polish National Centre for Research and Development under M-ERA.NET2/2020/1/2021 contract.

Resonant methods have been widely recognized by the scientific and industrial communities for their high precision of electromagnetic material characterization [3]. Among wide spectrum of possible configurations and designation, dielectric resonator methods are widely known solutions, commonly used for electronic materials measurements. Single-Post Dielectric Resonator (SiPDR) configuration [4] is used for resistivity and surface resistance measurements of low resistivity semiconductors and thin conductive films (e.g. screen printed silver- or carbon-based polymer composites), whereas Split-Post Dielectric Resonator devices (SPDR) [5] are applied to characterization of laminar dielectrics and high-resistivity semiconductors, delivering complex permittivity and resistivity values. The unprecedented accuracy of the SPDR technique [5-6], as high as 0.3% for dielectric constant and 3% for loss tangent, owed to dedicated calibration process, performed for each device unit and supported by rigorous electromagnetic modelling, makes it suitable for high precision characterisation, necessary for novel materials dedicated for emerging 5G and 6G electronics [3].

SPDR technique has been proven to be successfully applied to characterisation of LTCC materials [7]. In this work it is considered for complex permittivity measurements of a set of ULTCC materials samples. In Section II, we discuss ULTCC materials samples rigorously prepared for their characterisation with the aid of SPDR test-fixture, whereas results of SPDR characterisation are evaluated in Section III of this work.

II. ULTCC Materials

Four ULTCC materials samples, as shown in Fig. 1, have been prepared for the purpose of complex permittivity measurements with SPDR test-fixtures. ULTCC samples are based on new ceramic materials with a low dielectric permittivity: Li_2WO_4 with 4 wt.% $CuBi_2O_4$, Li_2WO_4 with 4 wt.% AlF_3-CaB_4O_7, $LiBO_2$ with 4 wt.% $CuBi_2O_4$, $LiBO_2$ with 4 wt.% AlF_3-CaB_4O_7, and have been prepared according to the conventional ceramic procedure. The fabrication procedure comprised solid state synthesis of oxide components, ball milling, uniaxial pressing of pellets, and sintering at 610-650°C for 1 - 2h. The material composition of each fabricated and investigated sample, with reference to Fig. 1, has been given in Table I. The considered ULTCC materials are expected to have low dielectric constant and loss tangent, being in the range of 4 – 6.5 and 0.0005 – 0.005, respectively, making them good candidates for laminar substrates for components dedicated to 5G systems.

With the available technological process for bulk ULTCC composites fabrication, all samples have been prepared in a circular shape with maximum diameter of ca. 20 mm, obeying requirements for minimum lateral size for SPDR 10 GHz [6], and thickness of 1-1.5mm. After fabrication, all the samples were manually polished to fit their thickness into SPDRs limitation of 0.6 – 0.7 mm. Eventually, due to manual processing, ULTCC material samples gained thickness variation as reported in Table II.

III. Materials Characterisation

Dielectric properties of ULTCC materials samples, as of Table I, have been measured with SPDR test-fixture with a nominal resonant frequency of an empty device of 10GHz. Technologically limited maximum size of the samples under test and their thickness determined maximum operating frequency of the SPDR device. Measurement frequency of 10GHz lands between 5G microwave frequency bands however, with a known value of QF extracted at 10GHz (QF=const), their dielectric properties may be extrapolated towards bands of interest.

Measurement procedure consists of two stages, where resonant frequency and Q-factor are measured firstly, for an empty and then, sample-loaded SPDR device. The measured quantities are input parameters for the SPDR dedicated conversion software, extracting complex permittivity (dielectric constant and loss tangent) of a sample under test (SUT).

Fig. 1. ULTCC materials samples under investigation (Sample 1 to 4 – arranged from left).

TABLE I. ULTCC materials samples

Sample name	Material description
Sample 1	Li_2WO_4 + 4% $CuBi_2O_4$
Sample 2	Li_2WO_4 +4% AlF_3-CaB_4O_7
Sample 3	Li_2BO_2 + 4% $CuBi_2O_4$
Sample 4	Li_2BO_4 +4% AlF_3-CaB_4O_7

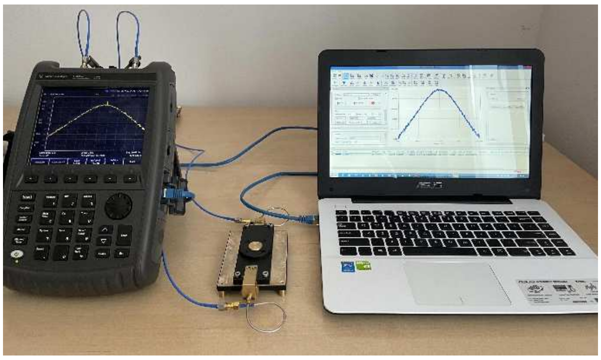

Fig. 2. Measurement setup for complex permittivity extraction, comprising of 10GHz SPDR device, handheld VNA, and measurement control computer application.

TABLE II. Parameters of ULTCC materials measured with 10 GHz SPDR

Sample name	Thickness [mm]	Dielectric constant	Loss tangent (±3%)
Sample 1	0.485 ± 0.015	5.40 ± 2.5%	0.00200 ±3%
Sample 2	0.64 ± 0.030	6.52 ± 4%	0.00233 ±3%
Sample 3	0.6 ± 0.030	5.12 ± 4%	0.00195 ±3%
Sample 4	0.55 ± 0.020	4.48 ± 3%	0.00328±3%

The measurement setup is shown in Fig. 2 and consists of 10GHz SPDR test-fixture, handheld Keysight VNA, and measurement control application (Material Measurement Suite), allowing for fully automated measurement process, (controlled and invoked from a laptop), having a built-in functionalities for enhanced resonant frequency and Q-factor extraction, decreasing measurement uncertainties.

Table II reports measurement results for all four ULTCC materials samples. The obtained values of both, dielectric constant and loss tangent, prove that the developed compositions of ULTCC materials meet the expectations with regards to dielectric properties. However, with regards to results of Table II, a wider discussion on uncertainty of dielectric constant measurements is needed.

As discussed in [5], uncertainty of dielectric constant extraction is directly related to uncertainty of resonant frequency measurement and determination of SUT's thickness. For the investigated ULTCC materials samples, a dominant factor influencing uncertainty of dielectric constant extraction is SUTs thickness variation, which according to data given in Table II is non-negligible, and prevents accurate thickness evaluation. Dielectric properties of SUTs have been retrieved for an average thickness values and thickness variation has been used to determine a related uncertainty of dielectric constant evaluation, which in this case varies between ± 2.5 to ± 4%. The accuracy of thickness determination is much less pronounced in uncertainty of loss tangent extraction and becomes negligible compared to inherent accuracy of SPDR method defined as 3%.

IV. CONCLUSIONS

In this paper, novel ULTCC materials, envisaged to be used for design and fabrication of communication components for 5G systems, have been discussed. Four ULTCC materials samples, varying in chemical composition, have been prepared in sintering process and their dielectric properties have been measured with high-accuracy SPDR technique. The obtained measurement results have been discussed with regards to uncertainty of complex permittivity extraction, which, for the investigated ULTCC materials samples, is determined mainly by SUTs thickness variation, having a major impact on uncertainty of dielectric constant evaluation. In the view of this discussion, it is concluded that conducting uncertainty study for measured dielectric properties becomes crucial, specifically when samples, due to e.g. technological constraints, are needed to be mechanically postprocessed or are known to be of limited flatness. The extracted values of dielectric constant and loss tangent, with evaluated uncertainty bounds, show good correlation with targeted dielectric properties, implying the investigated materials are promising for laminar substrates for 5G systems designers.

REFERENCES

[1] M. Celuch, O. Douheret, P. Korpas, R. Michnowski, M. Olszewska-Placha, J. Rudnicki, “Portable low-cost measurement setup for 2D imaging of organic semiconductors,” IEEE MTT-S International Microwave Symposium 2020, Los Angeles, 4-6 August 2020.

[2] D.Szwagierczak, B. Synkiewicz-Musialska, J. Kulawik, N. Palka, “LTCC and Bulk Zn4B6O13-Zn2SiO4 Composites for submillimeter Wave Applications”, Materials 2021, vol.14 no. 1014, Feb. 2021.

[3] M. Celuch, M.J. Hill, T. Karpisz, M. Olszewska-Placha, S. Phommakesone, U. Ray, B. Salski, “Benchmarking of GHz resonator techniques for the characterisation of 5G/mmWave materials,” 51st European Microwave Conference April 2021, pp. 568-571.

[4] J. Krupka and J. Mazierska, “Contactless measurements of resistivity of semiconductor wafers employing single-post and split-post dielectric-resonator techniques,” IEEE Trans. Instr. Meas., vol. 56, no. 5, pp. 1839-1844, Oct. 2007.

[5] J. Krupka, A. P. Gregory, O. C. Rochard, R. N. Clarke, B. Riddle, and J. Baker-Jarvis, “Uncertainty of complex permittivity measurements by split-post dielectric resonator technique,” J. Eur. Ceramic Soc., vol. 21, pp. 2673-2676, 2001.

[6] (2022) Split-Post Dielectric Resonator specification. [Online]. Available: https://qwed.eu/resonators_spdr.html

[7] J. Mazierska, M.V. Jacob, A. Harring, J. Krupka, P. Barnwell, T. Sims, “Measurements of loss tangent and relative permittivity of LTCC ceramics at varying temperatures and frequencies,” J. Eur. Ceramic Soc, vol. 23, no. 14, pp. 2611-2615, December 2003.