

EMPLOYMENT OF BROADBAND DIELECTRIC SPECTROSCOPY (BDS) IN STUDIES OF ANTENNA MATERIALS AND LIQUIDS

Malgorzata Holynska⁽¹⁾, Virginie Cesar-Auguste⁽¹⁾, Christopher Semprimoschnig⁽¹⁾

(1) ESA-ESTEC, Keplerlaan 1, 2201 AZ Noordwijk, The Netherlands
malgorzata.holynska@esa.int, virginie.cesar-auguste@esa.int, christopher.semprimoschnig@esa.int

ABSTRACT

This paper focuses on the applications of BDS (Broadband Dielectric Spectroscopy) to study materials for antenna structures and liquids, showing the applied experimental setups and methodology.

As an example of employment of BDS / RF (Radio Frequency) dielectric spectroscopy, studies of glass-reinforced composites used to manufacture antenna structure, performed at cryogenic temperatures, will be presented.

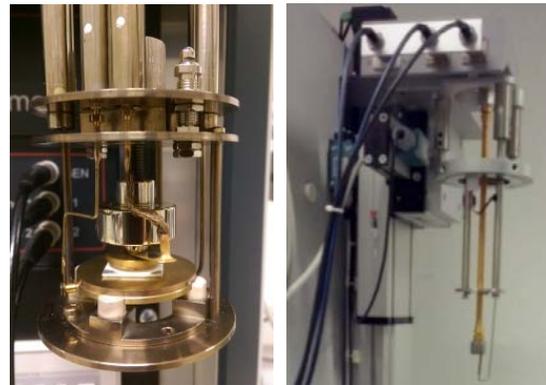
Employment of the setup for testing liquids will be demonstrated on the example of water and ice. Determination of dielectric constants of ice at low temperatures in the presence of representative contaminants is relevant as support for studies of electrostatic discharge in thin ice layers.

ABBREVIATIONS

| | |
|-------|---|
| AC | Alternating Current |
| BDS | Broadband Dielectric Spectroscopy |
| CFRP | Carbon-Reinforced Polymer |
| DC | Direct Current |
| ESA | European Space Agency |
| ESTEC | European Space Research and Technology Centre |
| GFRP | Glass-Reinforced Polymer |
| RF | Radio Frequency |
| RT | Room Temperature |

INTRODUCTION

Broadband Dielectric Spectrometry (BDS) records non-destructively the time-resolved response of a material to an applied alternating electric field (Figure 1). The frequency of the alternating field is varied and the current which passes through the sample is measured. The measurement is based on the interaction of an external electric field with the electric dipole moment and charges of the medium [1]. Setups to employ radio frequencies (RF) are also available and, as a rule, require more extensive calibration.



(a)

(b)



(c)

Figure 1 Novocontrol setup with alpha-analyzer for dielectric spectroscopy at ESTEC laboratories: (a) the BDS, (b) the RF sample cell. (c) The main unit with alpha-analyzer.

From the output different related dielectric properties, such as impedance, capacity, complex permittivity and conductivity, can be obtained. These properties may support studies aiming at characterization of structural phase transitions (e. g. glass transition), charge mobility, diffusion coefficients, activation energies, molecular relaxations (in terms of time, strength, shape parameters) [2].

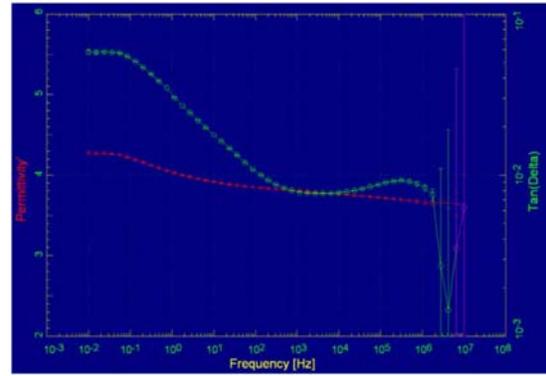
In particular, in the context of aerospace materials, dielectric properties of the structure are relevant for antenna components. For these materials typically measurements at high frequencies are relevant, from 1

MHz until 1.8 GHz, and can be used for the purpose of characterization or quality control at broad temperature range, enveloping the relevant space mission conditions.

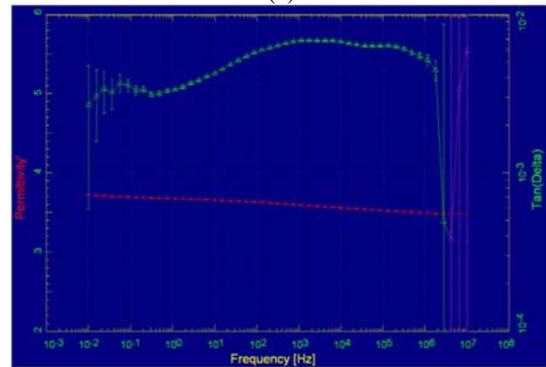
ANTENNA MATERIALS

GFRP sample was chosen for testing with BDS/RF dielectric spectroscopy. The choice of this material is driven by structural and electrical performances, including resistance to extreme environmental conditions. In non-standard setups impact of mechanical stress on the dielectric properties can also be studied.

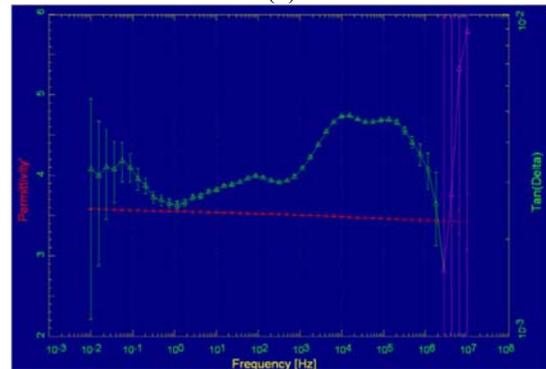
The GFRP sample capacity could be adjusted to the range of 50 to 200 pF at 10^5 to 10^7 Hz (optimum for the device) through the use of 30 mm diameter electrode and metallization of the sample. Permittivity and AC conductivity in the frequency domain are plotted along with the dielectric dissipation factor ($\tan(\delta)$) in Figure 2 at RT, -50 and -150°C, respectively. The conductivity values are low, as expected for an insulating material, especially at 0.01 Hz which is closer to the DC case. The data obtained by BDS for frequencies higher than 1.15 MHz appear to be affected by high experimental errors. Access to these higher frequencies is granted by RF spectroscopy (Figure 2d). In this case the obtained mean dielectric constant values appear to be systematically higher than as obtained from BDS (e. g. at RT, 1 MHz RF: 4.08; 1.15 MHz BDS: 3.79). In principle, the curves obtained by BDS and RF should be superimposable, but this is rarely possible due to high uncertainties associated with the BDS high-frequency range and differences in sample preparation / contact to electrodes. At RT the ϵ' values appear to decrease with increase in frequency which could be expected based on literature [3]. This tendency is less clear at -50 and -150°C. Also, as expected [3], the ϵ' values increase with increase in temperature. This effect is connected with water content in the studied material. Apart from determination of basic dielectric properties, such as dielectric constant relevant for modelling for antenna application, BDS can be used for monitoring of moisture uptake. This uptake affects physical, chemical and mechanical properties of the CFRP-based materials, as a result of matrix cracking, chain scission / residual cross-linking, hydrolysis, oxidation, softening and plasticization [4]. Water molecules in the GFRP matrix may be unbound (diffused from the outer surface) or bound with the GFRP matrix [4].



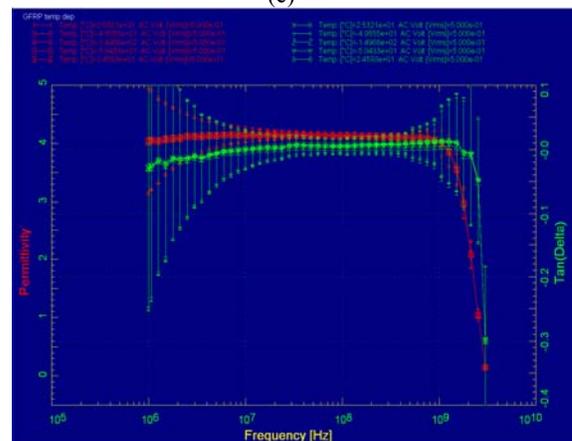
(a)



(b)



(c)



(d)

Figure 2 Dielectric constant / tan(delta) for one of the GFRP samples studied at (a) RT, (b) -50, (c) -150°C with BDS. (d) RF data at RT, -50 and -150°C.

LIQUIDS

BDS technique can be used to test dielectric properties of liquids which can be also frozen in a dedicated parallel-plate sample cell. The sample holders available for BDS and RF testing at ESTEC are shown in Figure 3. Several drops of the tested liquid are introduced in such sample holders, along with quartz spacers to control the liquid layer thickness. Some results will be shown on the example of water/ice. Water formed as a by-product of combustion may condense on cold outer surfaces of spacecraft as amorphous or crystalline form of ice. The expected low conductivity of pure ice may be affected by the presence of nitrates from plume impingement.



Figure 3 Sample holders available for BDS (a) and RF (b) testing at ESTEC.

In Figure 4 RF permittivity / conductivity is shown for water in the high frequency domain. The dielectric constant is in the expected [5] range with ca. 77 at 1 MHz. On the other hand, BDS results (Figure 5) are more affected by the electrodes polarization. This is true for any polar samples, such as aqueous solutions, ionic liquids or biological systems. In general, these lead to higher values of dielectric constant and sudden decrease in conductivity at low frequencies [6]. These effects can be avoided via modification of the experimental setup or included in the results modelling.

The studied water sample was condensed from air on a cold glass surface. BDS tests were conducted at -50, -100, -120 and -150°C. The maximum and the corresponding dielectric constant at lower frequencies (plateau in Figure 5) is too high, as a result of electrodes polarization. This maximum shifts to the lower frequencies with decrease in temperature which may

correlate with charging effects observed on ice at temperatures lower than -150°C for this layers of ice [7].

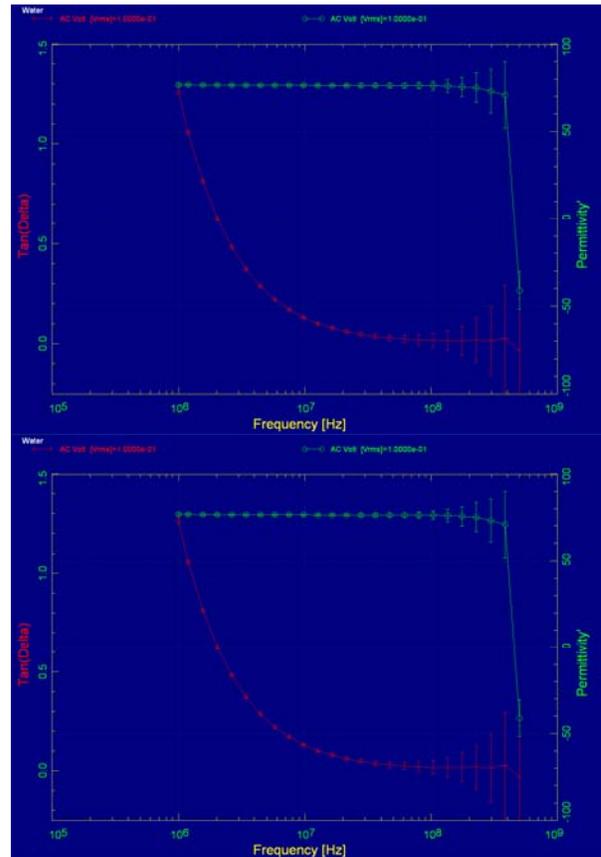


Figure 4 RF dielectric spectra for demineralized water.

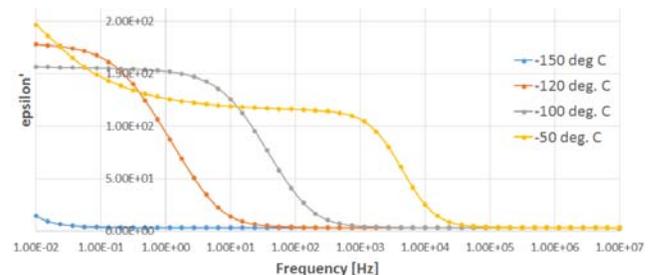


Figure 5 BDS results for water condensed from air with 100 µm spacer.

CONCLUSION

In conclusion, further examples of application of dielectric spectroscopy relevant for testing of space materials have been presented. Use of the liquid chamber gives access to studies of e. g. coolant liquids or any liquid material that may be deposited on structural parts and affect their charging properties. In this paper testing of water relevant for the latter issue in case of some space missions was shown. On the other hand, attachment for tests at radio frequencies (RF) can be used for studies

applicable to dielectric materials, such as GFRPs for the use in antennas. The output values include such basic characteristics as dielectric constants, AC conductivity or dielectric losses, but other experiments, such as monitoring of in situ thermal cycling, may be also conducted.

REFERENCES

- [1] (a) Eds. F. Kremer, A. Schoenhals (2003): Broadband Dielectric Spectroscopy, Springer. (b) V. Cesar-Auguste, E. Amorim, C. Semprimoschnig (2015): Dielectric properties of white ceramic coatings, ISMSE'2015.
- [2] G. Williams, D. K. Thomas (1998): Phenomenological and Molecular Theories of Dielectric and Electrical Relaxation of Materials, Novocontrol Application Note.
- [3] D. Pathania, D. Singh (2009): Int. J. Theor. & Appl. Sci. 1, 34-37.
- [4] (a) S. A. Grammatikos, R. J. Ball, R. G. Jones (2018): Composites: Part A 105, 108-117. (b) M. Chevalier, E. Dantras, C. Tonon, P. Guigue, C. Lacabanne, C. Puig, C. Durin (2009): Non-destructive testing of bonded assemblies during ageing by dynamic dielectric spectroscopy, ISMSE'2009.
- [5] V. G. Artemov, A. A. Volkov (2013): arXiv:1308.1229 [cond-mat.soft]
- [6] S. Emmert, M. Wolf, R. Gulich, S. Krohns, S. Kastner, P. Lunkenheimer, A. Loidl (2011): arXiv:1106.1380v1 [cond-mat.soft]
- [7] A. Polsak - TEC-QEE report on charging of ice – in preparation.