

Temperature Dependent Dielectric Measurements at 2.45 GHz

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Abstract—The application of high power microwave technology promises innovative and energy efficiency thermal processes in food, textile, automotive and aerospace industries. Selective and volumetric microwave heating may offer significant advantages with respect to energy and time consumption as compared to processing in conventional ovens. For development of microwave systems and processes the microwave properties of processed materials have to be known. Therefore a non-resonant measurement method using transmission and reflection coefficient has been developed to measure complex permittivity of non-magnetic solid and liquid materials. The measurement setup and the calculation method will be described. Temperature dependence measurement of polymers and resins will be presented.

Keywords—Dielectric properties, temperature dependence, waveguide-based method, transmission-reflection method, industrial microwave heating

I. INTRODUCTION

Various conventional heating technologies need excess energy due to inefficient heat transfer to the processed materials, in particular to the materials volume. In contrast to that microwave technology offers energy efficient processing of materials due to selective and volumetric heating. Thus high temperature processing can be implemented in cold furnaces even. The benefit of direct heating in materials volume allows a significant reduction in processing time and finally in energy consumption, in particular for big size products characterized by low thermal conductivity. This motivates a large variety of potential microwave applications such as drying, curing, tempering or sintering and a growing interest in food, chemical or ceramic industry. For successful microwave curing of carbon fiber reinforced polymers (CFRP) a modular microwave technology has been developed at the Karlsruhe Institute of Technology (KIT) [4]. This so called HEPHAISTOS concept is characterized by a hexagonal applicator geometry that results in excellent field homogeneity. In combination with microwave specific concepts for tooling and process control this technology meets the specific needs for avionic or automotive applications.

To enter new markets for microwave applications product specific systems and processes need to be developed. An important prerequisite for this is the precise knowledge for the material properties. Therefore many dielectric property measurement techniques have been developed [1, 3, 5]. According to the big variety of materials and properties there is no general method for dielectric measurements that can be used for all materials and purposes. Existing methods can be classified into resonant and non-resonant methods. In [2, 3, 5] the transmission-reflection method has been selected to characterize dielectric materials. Waveguides are used for the measurement of transmission and reflection coefficient of material under test that fully or partially cover the waveguide cross section. Since those measurements are motivated by microwave heating applications, the temperature dependence of the complex permittivity is very important for a successful design and optimization of new microwave processes and systems. A concept for temperature dependence measurement of thin materials is presented in [7]. The system uses a resonant method that is suitable for dielectric measurement of low loss materials.

II. THEORY

The non-resonant transmission and reflection method is appropriate to measure materials with moderate loss factor. The basic principle of permittivity measurement using the transmission-reflection method was published in [1, 6], where the analytical formulas for calculation of complex permittivity were described.

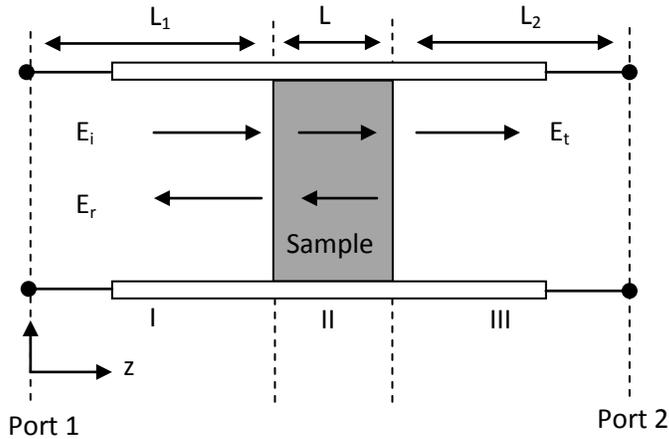


Figure 1. Material in transmission

The incident electromagnetic field E_i that enters the material under test is split into a transmitted wave E_t and a reflected wave E_r due to the characteristic of the material (see Fig. 1). The reflection and transmission coefficient can be measured and post processed to calculate the dielectric properties of the material. For a sample that fully covers the waveguide cross-section, the S-parameters at the reference planes, port 1 and port 2, can be estimated as:

$$S_{11} = R_1^2 \left[\frac{\Gamma(1-T^2)}{1-\Gamma^2 T^2} \right] \quad (1)$$

$$S_{22} = R_2^2 \left[\frac{\Gamma(1-T^2)}{1-\Gamma^2 T^2} \right] \quad (2)$$

$$S_{21} = R_1 R_2 \left[\frac{T(1-\Gamma^2)}{1-\Gamma^2 T^2} \right] \quad (3)$$

with R_1 and R_2 as described by the following formula:

$$R_1 = e^{-\gamma_0 L_1} \quad (4)$$

$$R_2 = e^{-\gamma_0 L_2} \quad (5)$$

The distances of the sample surfaces to reference plane 1 and 2 are L_1 and L_2 respectively. γ_0 is the propagation constant of the wave in the empty waveguide. The transmission coefficient is calculated from:

$$T = e^{-\gamma L} \quad (6)$$

The reflection coefficient can be calculated from:

$$\Gamma = \frac{\gamma_0 - \gamma}{\gamma_0 + \gamma} \quad (7)$$

Where γ is equal to the propagation constant in the wave guide filled by the material under test. The equation (6) and (7) can be solved to get an explicit formula for calculation of complex permittivity

$$\Gamma = K \pm \sqrt{K^2 - 1} \quad (8)$$

$$K = \frac{(S_{11}^2 - S_{21}^2) + 1}{2S_{11}} \quad (9)$$

The positive or negative sign of equation (8) is selected based on the value of Γ that fulfills $|\Gamma| \leq 1$. The transmission coefficient can be defined as:

$$T = \frac{(S_{11}+S_{21})-\Gamma}{1-(S_{11}+S_{21})\Gamma} \quad (10)$$

For nonmagnetic materials the complex permittivity can be calculated from:

$$\epsilon_r = \frac{\lambda_0^2}{\left[\left(\frac{1}{\lambda_c^2}\right) - \left(\frac{1}{\lambda^2}\right)\right]} \quad (11)$$

where:

$$\frac{1}{\lambda^2} = - \left[\frac{1}{2\pi L} \ln \left(\frac{1}{T} \right) \right]^2 \quad (12)$$

where L corresponds to the sample thickness, λ_0 is the free-space wavelength, λ_c is cut-off wavelength of the used waveguide.

III. MEASUREMENT SETUP

The measurement setup for dielectric properties measurement is shown in the following figure:

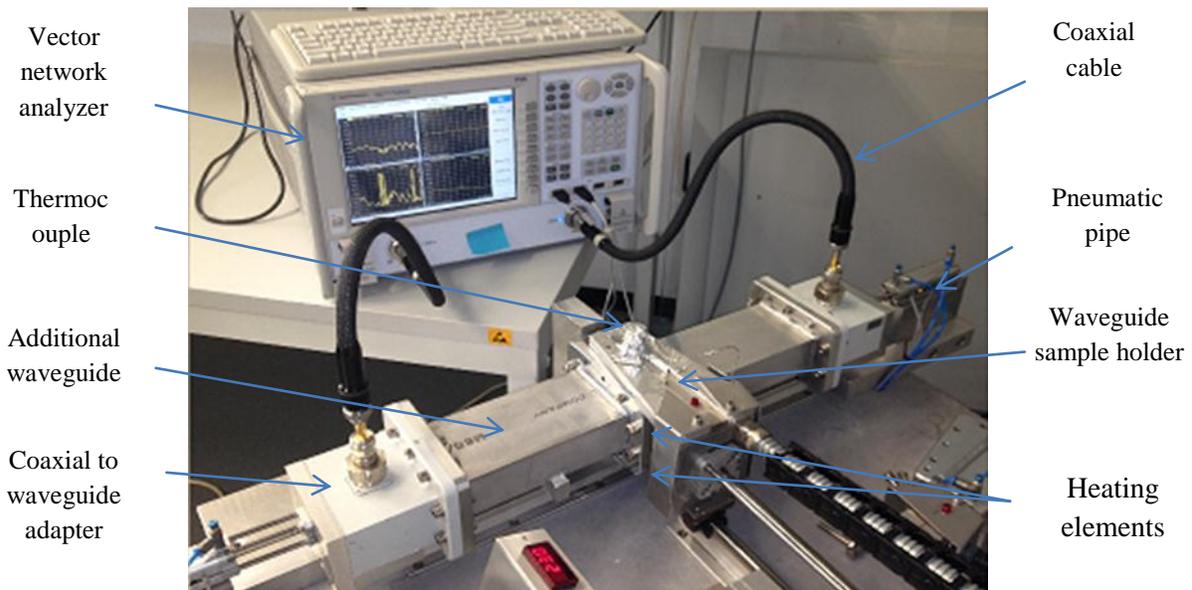


Figure 2. Measurement setup for temperature dependent dielectric properties at 2.45 GHz

The vector network analyzer (VNA) is connected to the waveguide sample holder with coaxial cables and waveguide to coaxial adapters. The waveguide sample holder is installed in-between two additional waveguides of adequate length to ensure sufficient suppression of higher order modes at the measurement port. The waveguide sample holder is equipped with a thermocouple to measure the temperature of the material. Four heating elements are installed in the waveguide sample holder to achieve a uniform heating of the material under test. Those heating

elements are PID-controlled to get the desired temperature in the waveguide sample holder. Therefore the difference between measured and preset temperature is used as feedback signal of the PID controller. The waveguide sample holder as well as the waveguide ports is equipped with pneumatic push rods. This enables remote controlled shift of the sample holder to allow for insertion of the test sample. Open and close movement of the waveguide adapter, before heating and before S-parameter measurement, respectively, will reduce the heat load to the waveguides. Any heating of the waveguides may influence the system calibration and therefore the measurement accuracy. Position sensors are installed to get a feedback about the waveguide position (open or close). All this equipment will be connected to the computer by use of a multifunctional input/output device to allow a fully remote controlled measurement procedure.

But before the measurement can be started the set-up needs to be calibrated to the waveguide sample holder ports. Since a waveguide calibration standard for the used WR 340 waveguide is not available, a calibration to the reference plane was achieved with a through-reflect-line (TRL) calibration. Parameter for through can be got by direct connection of both waveguide without the sample holder, while parameter for line can be received with the empty sample holder as line standard. Parameter for reflect can be obtained by closing each additional waveguide with a metal short.

Solid material is measured using a sample that fully covers the waveguide cross section (86.4mm x 43.2 mm x thickness of the sample). Liquid material is measured using a glass tube sample holder. Calculation of dielectric properties of liquid materials is performed using 3D full-wave electromagnetic simulation by means of CST microwave studio. Therefore the S-parameters of the waveguide sample holder including the liquid material and the glass tube were modeled. Using an optimization method the dielectric properties used were varied until the best fit of simulated and measured S-parameters was achieved. Besides such a numerical optimization method for more complex sample geometries, analytical approaches are also feasible if simple rectangular containers were used. The development of such analytical models is still in progress.

IV. RESULT AND DISCUSSION

The measurement of Polyvinylchloride (PVC) shows that the dielectric constant and the loss factor increase with increasing temperature. The dielectric constant and loss factor of PVC obtained at room temperature is close to values reported in literature [5] which is 2.85.

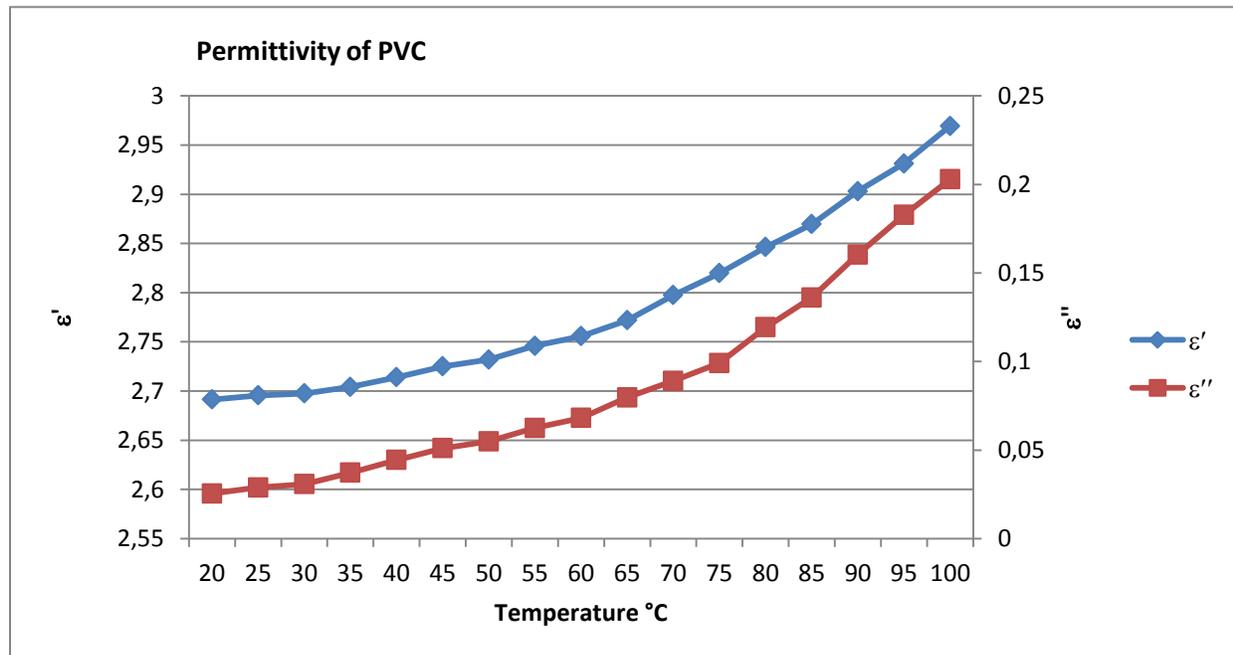


Figure 3. Complex permittivity of PVC

The increasing loss factor can be seen clearly since the loss factor of PVC is greater than the resolution limit of the system (0.01).

The dielectric properties obtained for Teflon show a temperature behavior similar to PVC. Nevertheless in case of dielectric loss factor the graph looks noisier than for PVC because the measured values are below the resolution limit of the system. However, the temperature trend still can be seen.

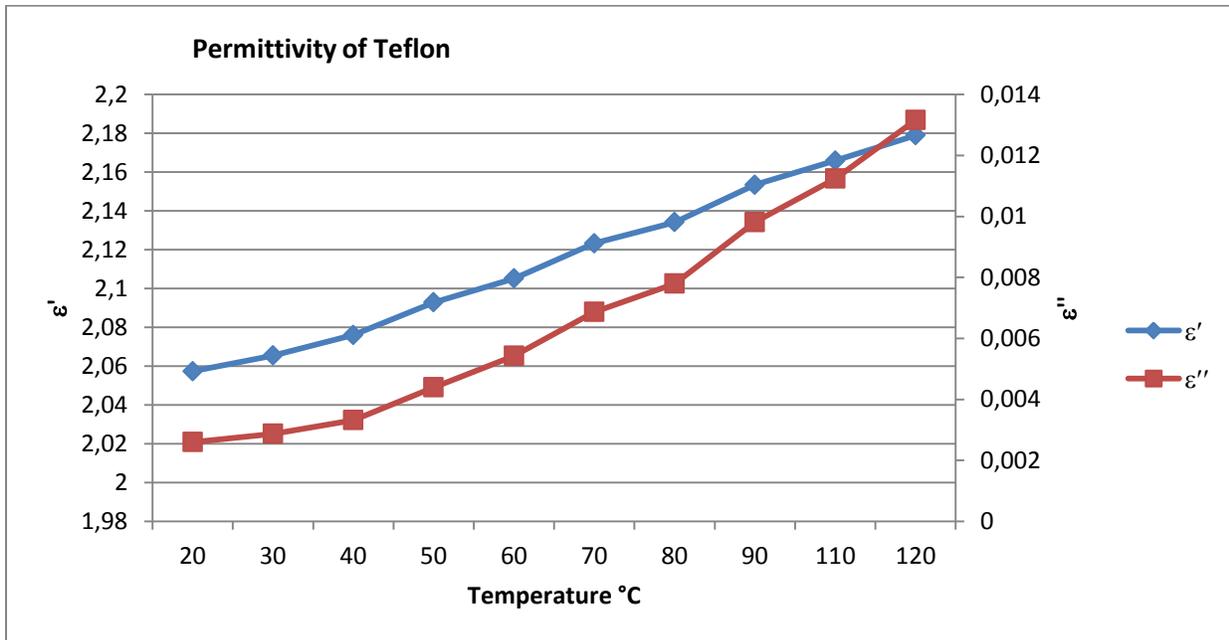


Figure 4. Complex permittivity of Teflon

Motivated by the activities on microwave curing of CRFP, the dielectric properties of the composite material, in particular of the thermoset polymer matrix is of special interest. Since the curing is going along with a chemical polymerization process, this will have significant influence on the temperature dependent behavior of the dielectric properties.

A widespread used thermoset polymer is based on epoxy resin, a molecule containing epoxide groups. These thermoset polymers are used as adhesives, high performance coating materials or for production of fiber composite materials in avionic and automobile industries. One type of epoxide resin is glycidyl-ether epoxies such as, diglycidyl ether of bisphenol-A (DGEBA).

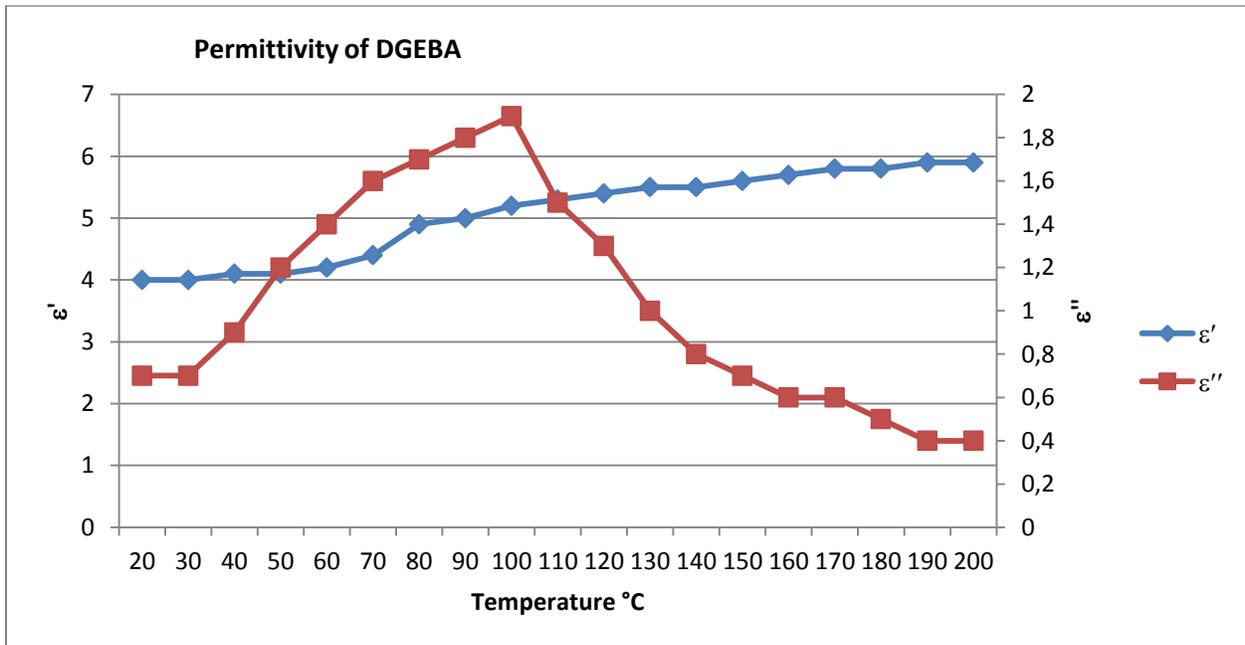


Figure 5. Complex permittivity of DGEBA

DGEBA is a typical commercial epoxy resin and is synthesized by reacting bisphenol-A with epichlorohydrin in presence of a basic catalyst. The properties of the DGEBA resins depend on the degree of polymerization. In order to convert epoxy resins into a hard and rigid material, it is necessary to cure the resin with hardener. Depending on the choice of curing agent epoxy resins cure quickly and easily at practically any temperature from 5-150°C. The dielectric properties obtained for DGEBA shows a monotonic increase of dielectric constant in the investigated the temperature change. However, the loss factor has a different characteristic. The loss factor increases until a certain temperature and then decreases again at temperature above.

A second type of epoxy resin that was investigated is tetraglycidyl methylene dianiline (TGMDA). Beside the epoxide groups TGMDA the aniline part consists of phenyl and amino groups. TGMDA epoxies are characterized by high cross-link densities, which results in a high modulus of elasticity and a high glass transition temperature. For TGMDA a temperature behavior of loss factor similar to DGEBA was found

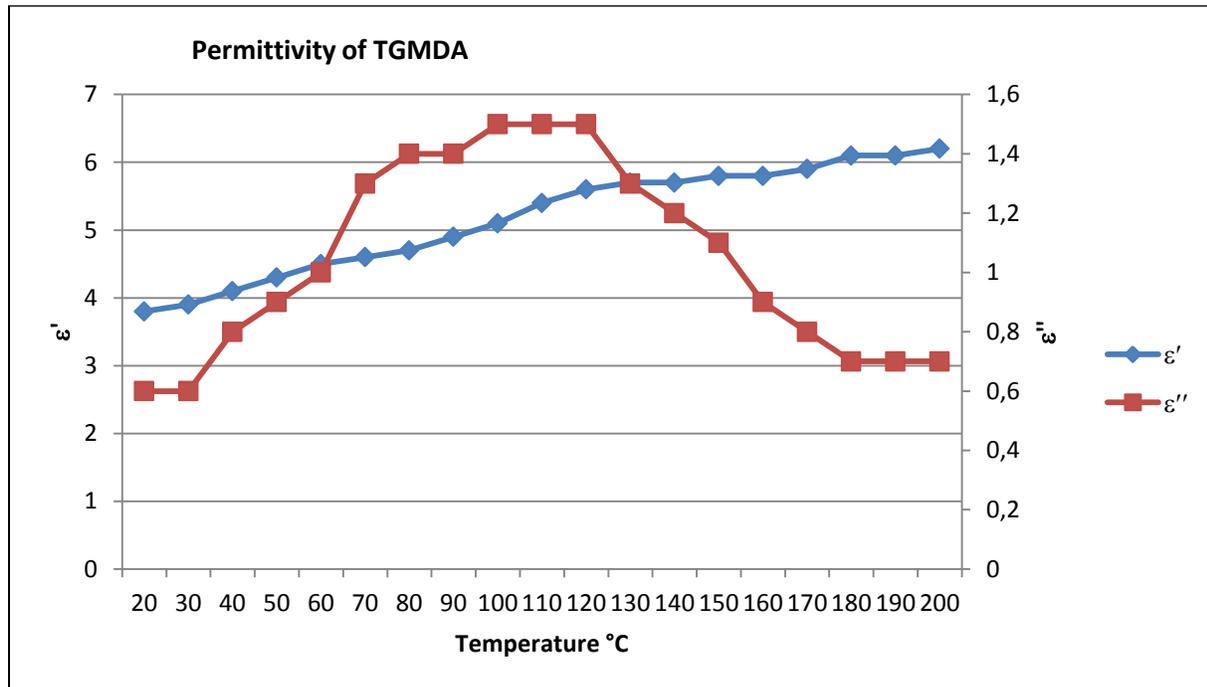


Figure 6. Complex permittivity of TGMDA

V. CONCLUSION

An experimental setup for temperature dependent measurement of dielectric properties at 2.45 GHz has been developed. The setup can measure both solid and liquid materials with moderate dielectric loss factor. Experimental results obtained for PVC and Teflon show that complex permittivity increases monolithically with increasing temperature. The results obtained for two different epoxy resins as they were DGEBA and TGMDA show that the loss factor increases until certain temperature and then decrease.

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