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Development of an open-ended microstrip stub apparatus and technique for the dielectric characterization of powders

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ABSTRACT

A new apparatus and method to characterize the complex dielectric permittivity of powders is described. The apparatus and technique are used to determine the dielectric properties of detergent powder agglomerates at different conditions. The technique is based on the measurement of scattering-parameters of an open circuit microstrip stub partly loaded with the test powder material. The scattering parameters relate the voltage waves incident on the ports of a microwave network to those reflected from the ports and can easily be measured with a vector network analyzer. A 3D finite element electromagnetic field simulation tool HFSS (High frequency structural simulator) is used to replicate the measured S-parameters and then extract the complex permittivity data from it. The method has been verified by measuring the dielectric properties of disks of known dielectric materials – specifically Duriod 5880 and Teflon. Results are in good agreement with manufacturer data sheets.
The complex permittivity of a range of detergent powder agglomerates with different moisture levels, at ambient and elevated temperatures, has been determined using this technique. Results are consistent with predictions of how the water interacts with the different components of the detergent particles at these different conditions.

1. Introduction

Microwave processing of materials is an area of on-going interest [1–6]. The dielectric properties of any material are obviously fundamental to its' suitability for any microwave/RF-based process [7,8]. Having the capability to easily characterize the dielectric properties of a material over a range of conditions – such as those experienced during processing – will help to better understand and predict material behaviours during processing.

1.1. Detergent powders – properties and characteristics

P&G (Procter & Gamble) makes many different types of detergent particles, such as agglomerates, blown powders, and extrudates. The most common manufacturing processes used are agglomeration and spray-drying. Agglomeration inherently makes higher density particles. Detergent particles will usually consist of surfactant(s) combined with organic polymers, such as polycarboxylates, soluble inorganic salts such as sodium carbonate, sodium sulphate and sodium silicate as well as insoluble materials such as silica and zeolite. Surfactants can have complex phase diagrams with a range of crystal, liquid crystalline and amorphous phases being present dependent on factors such as concentration, available water level, presence of electrolytes etc. Water can also be incorporated in inorganic hydrates formed from interactions with the non-surfactant components, such as sodium carbonate monohydrate.

Microwave radiation can be used to dry detergent powders using commercially available suitable equipment. However, microwave radiation can also be used to modify the properties of detergent particles, typically lowering the bulk density of the material by the formation of internal porosity. This is due to generation of steam inside the particles during heating. US Patent 6063751 [9] gives examples of this and the changes in the bulk density of the materials.

The water can be incorporated in multiple forms into a detergent particle – for instance, as non-associated free water, in surfactant liquid crystals or tightly bound in crystalline hydrates. The different strengths of these binding interactions mean that the impact of water on the dielectric properties of the particles is not straightforward. Water that is tightly bound in a hydrate crystal lattice will not interact significantly with microwaves/RF. However water in a surfactant liquid crystal will be affected much more strongly. What will complicate the characterization is that many of these hydrates have a temperature dependency – e.g., sodium sulphate decahydrate will decompose above 32 °C. Hence a priori knowledge of the composition is insufficient to predict dielectric properties during processing. Empirical determination over the range of conditions that will be experienced during processing is required.

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1.2 Equipment development

There have been several methods in the literature to measure the complex permittivity of the dielectric materials [10–16]. These methods could be classified mainly as either narrowband or wideband measurements. Each method has its advantages and disadvantages in terms of accuracy, ease of measurement, narrowband or wideband measurements, suitability for various liquids or solids, and frequency limitations etc. [17].

In this paper, a new sophisticated low cost technique is introduced to precisely characterize the complex permittivity of dielectric materials in powder form. The complex permittivity of various commercially used detergent particles is determined using this technique. The resonant technique is used to perturb the resonant frequency of the open circuit stub by the dielectric material placed at its open end. The shifts in resonance frequency and dielectric absorption are measured to determine the complex permittivity of the material. The complete measurement procedure is explained in Section 3 and the measured results of different detergent agglomerates are presented in Section 5. The complex permittivity of different commercially used detergent particles is determined at room temperature and later on at elevated temperatures. This is relevant because the water-binding mechanisms can change at different temperatures – e.g. some hydrates will not form at elevated temperatures. This complex permittivity data can be used for further microwave processing of the detergent agglomerates.

2. Measurement fixture

A quarter wavelength open circuit stub is designed on a microstrip line. The Duriod material used has $\varepsilon_r = 2.33$, $\tan\delta = 0.0009$ and substrate thickness of 3.18 mm. The width of the microstrip line can be calculated from [18].

$$Z_0 = \frac{120\pi}{\sqrt{\varepsilon_{eff}}\left[\frac{W}{d} + 1.393 + 0.667\ \ln\left(\frac{W}{d} + 1.444\right)\right]} \quad (1)$$

for $\frac{W}{d} \geq 1$

Where

$$\varepsilon_{eff} = \frac{\varepsilon_r + 1}{2} + \frac{\varepsilon_r - 1}{2}\frac{1}{\sqrt{1 + \frac{12d}{W}}} \quad (2)$$

where,
ε_r is the dielectric constant of the substrate material, W is the width of the conductor, d is the height of the substrate and ε_{eff} is the effective dielectric constant of a homogenous medium which replaces the dielectric and air region of the microstrip. The width W of the copper trace is selected to match the 50 Ω input/output connectors and vector network analyzer, this gives W = 9.18 mm.

The length of the open ended stub is selected as a quarter wavelength at the frequency of interest (2.45 GHz). A small adjustment in the length of the transmission line is required to account for fringing capacitance at the open end of the transmission line stub. The capacitive reactance due to fringing capacitance can be calculated as [19].

$$X_f = \frac{1}{j\omega C_f} \quad (3)$$

where, C_f is the capacitance due to fringing fields.

When the sample holder is loaded with a dielectric material, the fringing capacitance increases and a down shift in resonance frequency of the quarter wavelength stub is noticed due to dielectric loading of the end of microstrip line. Fig. 1 represents the fabricated microstrip stub along with the Teflon holder.

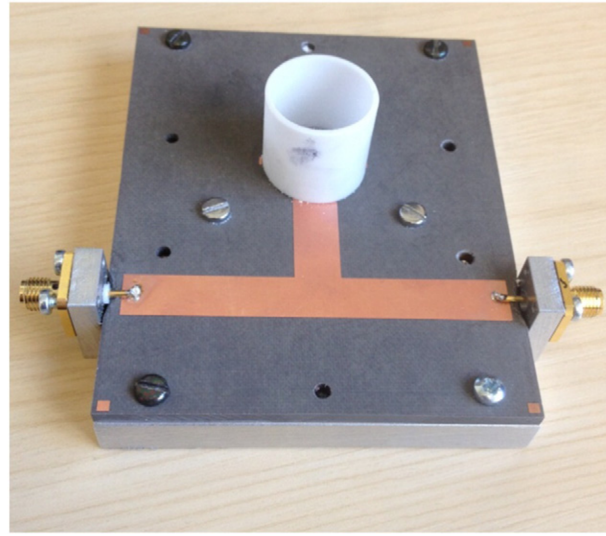

Fig. 1. Microstrip dielectric measurement circuit.

The substrate is placed on a 10 mm thick aluminium block for proper grounding. The sample holder is made up of Teflon material with dielectric properties of $\varepsilon_r = 2.1$, $\tan\delta = 0.001$. The sample holder has a wall thickness of 1 mm, an internal diameter of 20 mm, and a height of 20 mm.

3. Measurement procedure

The process of determining the complex permittivity of a detergent powder agglomerates consists of two steps. First - measure the S-parameters of the transmission line with the test powder loaded in the sample holder. Second- replicate these results using the Ansys HFSS electromagnetic simulation tool. This software is widely used for very reliable electromagnetic field simulations of high frequency and high speed components. In the 3D EM simulation modelling step, the dielectric powder sample being measured is replaced with a homogeneous dielectric material having an arbitrary value dielectric constant and loss tangent. A series of iterative simulations is then carried out until the simulation shows a close agreement with the measured results. Fig. 2

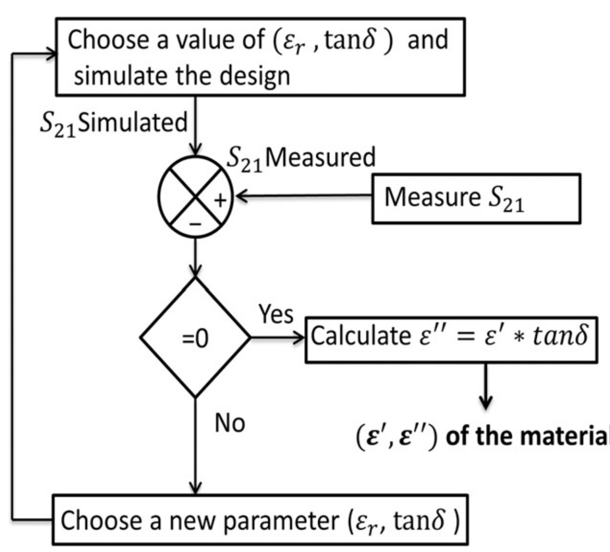

Fig. 2. Dielectric characterization flow diagram.