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Reversible Dielectric Property Degradation in Moisture-Contaminated Fiber-Reinforced Laminates

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Abstract. The potential for recovery of dielectric properties of three water-contaminated fiber-reinforced laminates is investigated using a split-post dielectric resonant technique at X-band (10 GHz). The three material systems investigated are bismaleimide (BMI) reinforced with an eight-harness satin weave quartz fabric, an epoxy resin reinforced with an eight-harness satin weave glass fabric (style 7781), and the same epoxy reinforced with a four-harness woven glass fabric (style 4180). A direct correlation between moisture content, dielectric constant, and loss tangent was observed during moisture absorption by immersion in distilled water at 25 °C for five equivalent samples of each material system. This trend is observed through at least 0.72% water content by weight for all three systems. The absorption of water into the BMI, 7781 epoxy, and 4180 epoxy laminates resulted in a 4.66%, 3.35%, and 4.01% increase in dielectric constant for a 0.679%, 0.608%, and 0.719% increase in water content by weight, respectively. Likewise, a significant increase was noticed in loss tangent for each material. The same water content is responsible for a 228%, 71.4%, and 64.1% increase in loss tangent, respectively. Subsequent to full desorption through drying at elevated temperature, the dielectric constant and loss tangent of each laminate exhibited minimal change from the dry, pre-absorption state. The dielectric constant and loss tangent change after the absorption and desorption cycle, relative to the initial state, was 0.144 % and 2.63% in the BMI, 0.084% and 1.71% in the style 7781 epoxy, and 0.003% and 4.51% in the style 4180 epoxy at near-zero moisture content. The similarity of dielectric constant and loss tangent in samples prior to absorption and after desorption suggests that any chemical or morphological changes induced by the presence of water have not caused irreversible changes in the dielectric properties of the laminates.

Keywords: Dielectric constant, Loss Tangent
PACS: 77.84.Lf Composite materials

INTRODUCTION

Radar protecting structures or radar domes (radomes) form a critical component of the radar system by shielding it from hazardous environmental conditions. These conditions may include debris, precipitation, humidity, hail or bird strike, and exposure to aircraft-related fluids. Radomes are typically used in ground based, maritime, or aircraft-based radar installations. Ideally, in an aircraft, the radome structure should be transparent to electromagnetic energy and maintain its structural integrity while in service [1]. These structures are typically constructed of polymer fiber-reinforced composites due to the associated low weight, rigidity, impact protection and ability to maintain transparency to incoming radar waves. However, a significant concern throughout the service life of the polymer composite radome is its tendency to absorb moisture [2]. Assessment of the deleterious effects of moisture ingress on the mechanical properties of fiber-reinforced composites has garnered much focus throughout several decades of research [3-7]. In contrast, quantifying and assessing dielectric property degradation as a result of absorbed moisture has not received equivalent attention, though some studies have been performed [8-11].

Dielectric property variations can be precisely measured by means of a split-post dielectric resonator (SPDR). The SPDR is capable of measuring the dielectric constant (relative permittivity) and loss tangent of a material in the range of 1 to 20 GHz [12]. Accuracy of the method requires that samples should have a thickness that is uniform and less than the air gap in the SPDR (typically 1 mm) [13]. When a material sample is placed between the two dielectric resonators inside the metallic enclosure that comprises the SPDR fixture, an azimuthal electric field is generated [14]. The Rayleigh-Ritz method [12] is used to determine dielectric parameters of the inserted sample. The real part of permittivity, ε, in this device is calculated by the iterative function [14]:

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$$\varepsilon = 1 + \frac{f_0 - f_s}{hf_0 K_\varepsilon(\varepsilon, h)} \tag{1}$$

where f_0 is the resonant frequency of the empty SPDR, f_s is the resonant frequency with the sample inside the SPDR, h is thickness of the laminate, and K_s is a function of ε and h that is calculated for each resonator. Loss tangent can be determined from Equation (2):

$$\tan(\delta) = \frac{Q_U^{-1} - Q_{DR}^{-1} - Q_C^{-1}}{P_{es}} \tag{2}$$

where Q_U is the unloaded Q factor of the inner section of the resonator that contains the sample, Q_{DR} is Q factor due the dielectric losses of the resonator, Q_C is Q factor that depends on the metal losses of the inner section of resonator containing the sample, and P_{es} is the electric energy filling factor (ratio of electric energy stored in specimen to the electric energy stored in resonator) [14].

The goal of this study is to contribute to a more thorough understanding of the magnitude and reversibility of dielectric property changes as a result of water absorption within the fiber reinforced polymer networks that are typical of radome structures. In order to ensure relevance, characterization is performed at typical radar frequencies (X-band).

EXPERIMENTAL

The polymeric composites used in this study are a three-ply BMI resin reinforced with an eight-harness satin weave quartz fabric, a four-ply epoxy resin reinforced with a four-harness glass satin weave, and a similar four-ply epoxy resin reinforced with an eight-harness glass satin weave. The trade names for the BMI/quartz and the two epoxy systems are HexPly® F650 and Cycom 919®, respectively. The BMI composite was cured in an autoclave at 190 °C and 22 inHg vacuum for four hours and followed by an eight-hour post-cure at 232 °C. Epoxy composites were autoclave-cured at 121 °C and 23 inHg vacuum for two hours. Five laminates each for BMI/quartz, 7781 style epoxy, and 4180 style epoxy with dimensions of 63.5 mm^2 and thicknesses of 0.755, 0.866, 0.373 mm, respectively, were cut with a wet diamond saw, which is a typical machining method for this type of composite material. Laminates were dried at 80 °C and stored in a desiccator in order to remove all residual moisture. Samples were then weighed and the dry weight was recorded. Laminates were fully immersed in water and maintained in a constant temperature water bath at 25 °C for approximately four months. Upon removal for periodic weighing, samples were dried with a lint-free cloth to remove all surface moisture. Gravimetric weight gain data during moisture uptake was periodically recorded using a high precision analytical balance according to ASTM D5229 [15]. Immediately following weighing, samples were inserted into an SPDR connected to an Agilent Programmable Network Analyzer (PNA). The resultant changes in Q-factor and resonant frequency of the cavity due to the presence of the sample was used to calculate the dielectric constant and loss tangent of the material at 10 GHz. Following the monitoring of dielectric property loss during the absorption phase, desorption was performed in an oven at 50 °C and gravimetric data was recorded periodically until the initial sample weight stabilized, indicating the absence of absorbed water. Dielectric measurements during desorption were performed in the same manner as the absorption process. The dielectric data recorded is an average of all five samples for each material type, with an uncertainty level calculated using a 95% confidence interval.

RESULTS AND DISCUSSION

Dielectric constant as a function of moisture content is shown in Figures 1-3 for BMI/quartz, 7781 epoxy, and 4180 epoxy, accordingly. Moisture uptake in the aforementioned laminates resulted in a 4.66%, 3.35%, and 4.01% increase in dielectric constant for a 0.68%, 0.61%, and 0.72% increase in water content by weight, respectively. In order to simulate practical conditions, in which the time allowed for moisture desorption via moderate heating or vacuum conditions would be limited, desorption was terminated when the rate of weight loss for the samples decreased dramatically. As a result, the moisture content before absorption and after desorption are very slightly different. This is indicative of a small amount of firmly bound water molecules still existing within the polymer network. Given enough time, it is expected that this moisture could also be removed. As such, dielectric constant and loss tangent is reported at the projection of the best fit line of the desorption data points. By this method, the difference in dielectric constant after desorption with respect to the initial absorption dielectric constant measurements was 0.144 % in the BMI/quartz laminates, 0.084% in the style 7781 epoxy, and 0.003% in the style 4180. Figures 4-6 display loss tangent as a function of moisture content. The same water content responsible for the increase in dielectric constant is responsible for a 228%, 71.4%, and 64.1% increase in loss tangent for BMI/quartz, epoxy 7781 style, and epoxy 4180 style laminates, respectively. The much larger relative increase in loss tangent for the BMI laminates is likely a result of the higher void content in these laminates, leading to more water in the liquid, bulk phase. In the preceding composites, the changes in loss tangent after desorption were 2.63%, 1.71%, and 4.51%, respectively. The dielectric constant and loss tangent as a function of water content for all three laminate types during the desorption phase were approximately equivalent to those measured during the absorption phase. An analogous linear relationship of dielectric properties as a function of moisture content is observed for each composite system during both periods of moisture absorption and desorption. Though not relevant at radar frequencies, Cotinaud et al. performed a similar investigation on glass-fiber reinforced epoxy at a much lower frequency of 50 Hz and concluded that although mechanical properties after 0.6 to 0.7 % of water concentration are irreversible, dielectric properties are reversible and are a function of moisture content [16]. This indicates that the dielectric property loss associated with low-levels of moisture within the laminates can be reversed by a simple desorption method.

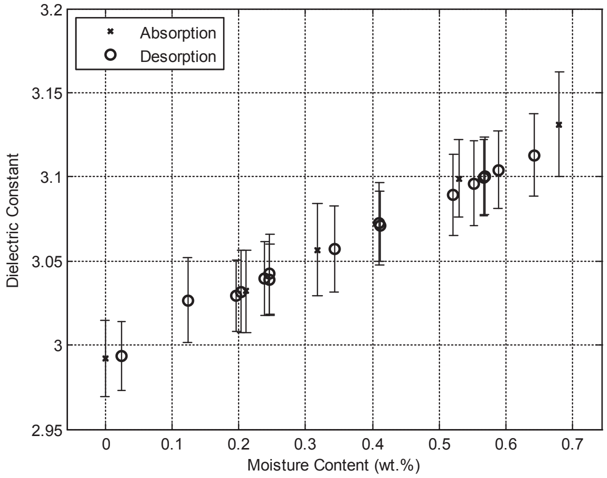

FIGURE 1. BMI/Quartz dielectric constant as a function of moisture content

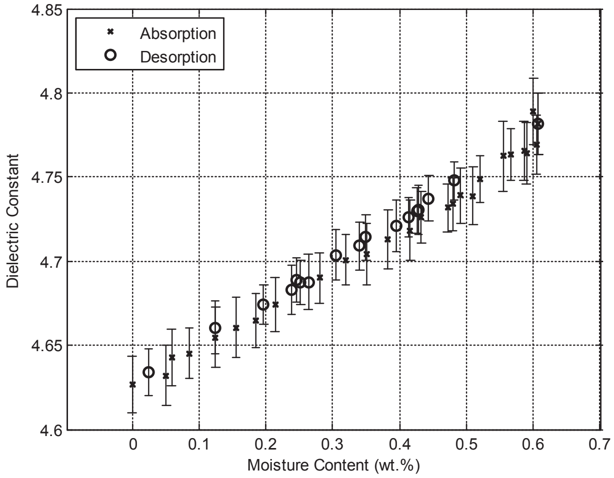

FIGURE 2. Style 7781 epoxy dielectric constant as a function of moisture content

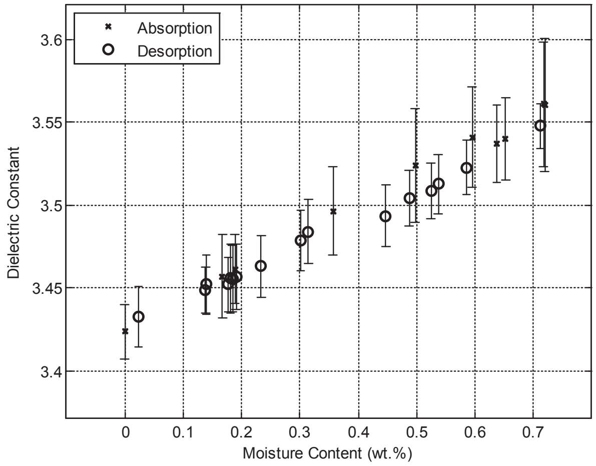

FIGURE 3. Style 4180 epoxy dielectric constant as a function of moisture content

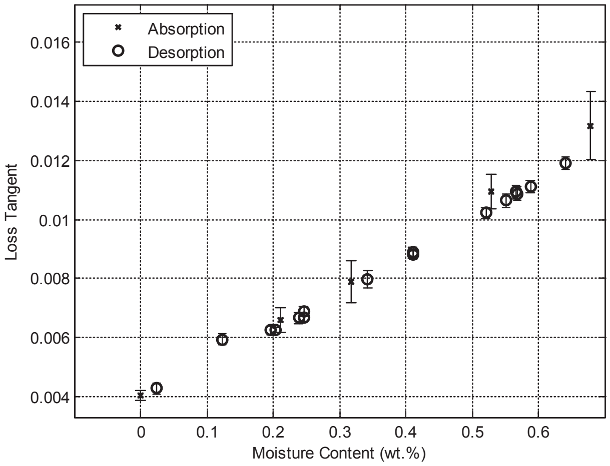

FIGURE 4. BMI/Quartz loss tangent as a function of moisture content

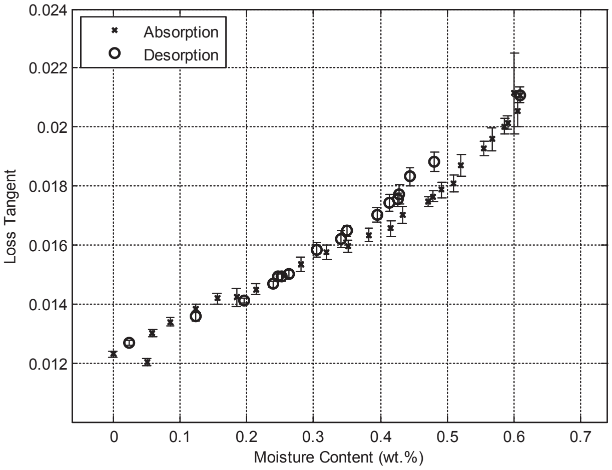

FIGURE 5. Style 7781 epoxy loss tangent as a function of moisture content

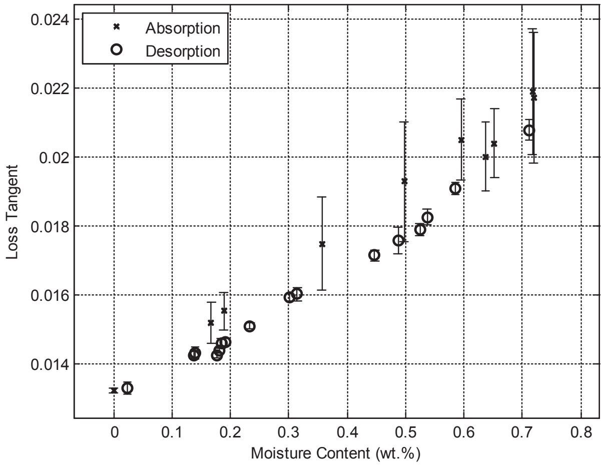

FIGURE 6. Style 4180 epoxy loss tangent as a function of moisture content

CONCLUSION

A significant increase in dielectric constant and loss tangent was observed for water-contaminated fiber-reinforced polymer radome laminates at X-band. Dielectric properties are directly related to total absorbed moisture content in each case. Dielectric property degradation is likely related to the energy loss associated with the reorientation of the dipole water molecule to align with an oscillating electric field. The similarity of dielectric constant and loss tangent in samples prior to absorption and after desorption suggests that any chemical or morphological changes induced by the presence of water have not caused irreversible changes in the dielectric properties of the laminates. Therefore, compromised dielectric properties and the associated loss of radar performance due to moisture contamination of a polymer composite radome may be mitigated by a simple desorption method such as moderate heating or vacuum conditions.

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