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Humidity and Temperature Effects on the Dielectric Properties of PET Film

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Abstract—The aim of the present manuscript is to contribute to the understanding of material performance of machine insulation film at high temperatures and changing relative humidity (RH). This represents a common stress under normal operation and may lower the material insulation performance. PET film is used as the example material and the complex dielectric permittivity is used as a marker to investigate changes in the dielectric properties. Measurements are carried out with a high precision dielectric spectrometer in a range of 10^{-2} .. 10^5 Hz at different temperatures (10..125 °C) and RH levels (0..80 %). In addition, evaluation of absorbed water at different RH levels (0..80 %) is done gravimetrically and exhibits a diffusion character according to Fick's law. Moreover, the proportionality of maximum absorption and RH level becomes apparent. Variation of temperature shows altered α and β loss mechanisms as well as changed DC conductivity proportions. The continuous permittivity increase during water absorption indicates the higher (moisture-induced) polar content in the polymer. However, a small portion of water seems to be bound to terminal –OH groups of PET, thus leading to an altered β relaxation. The observed increase of DC conductivity, not only with higher temperature but also with higher water content, points out the humidity-driven charge transport through the dielectric. A further, frequency-dependent loss mechanism is only visible above a certain RH level that indicates pure water formation within the polymer. This study shows that even though the level of absorbed water for PET is below 0.4 %, the dielectric spectra reveal significant changes.

Keywords— Machine Insulation, Dielectric Spectroscopy, Water Absorption, Temperature, Dielectric Losses

I. INTRODUCTION

Polyethylene terephthalate (PET) films have been widely used for many years as an electrical insulation material, e.g. for cables, transformers and rotating machines. However, the insulation performance is not only determined by the electrical and mechanical stress, but also by environmental conditions like relative humidity (RH) and temperature [1]. Because of their possible impacts on the polymer structure, broadband dielectric spectroscopy (BDS) is often used to observe such temperature- and RH-induced changes [2].

Various studies describe the existence of two relaxation processes in PET, which are named as α and β relaxations. Whereas the former is attributed to the glass transition (micro-

Brownian motion of polymer main chains) [3,4], different possible reasons for the local β relaxation have been proposed. These are namely the rotational motion of hydroxyl (-OH) groups at chain ends [5], the motion of the COO- group [6] as well as torsional vibrations of the main chain [7,8]. These dielectric properties depend strongly on the PET's degree of crystallinity [3,4]. Furthermore, in the low-frequency and/or high-temperature region, DC conductivity [9] and electrode polarization [10] were shown to dominate. While temperature effects in PET films have been extensively studied by means of BDS [3-5,9,11,12], comparatively little is known about the humidity effects. Miyairi found a minimum DC electric withstand strength of wet PET films around 0 °C which was explained by a conductivity increase due to adsorbed and/or absorbed water [13]. Further studies in the high-frequency region (> 0.1 kHz) showed an increase of dielectric permittivity and loss factor for wet PET films [5,14]. These authors attributed the loss factor increase to an enhanced β relaxation due to bound water molecules to terminal –OH groups of the PET chain ends.

Since the influence of RH on the dielectric properties of PET is not yet fully understood (especially in the lower frequency range < 0.1 kHz), the present contribution provides a detailed study on the humidity effects. This is complemented by temperature variations and BDS measurements in the same frequency range (10^{-2} .. 10^5 Hz) to compare their individual contributions to the changed dielectric properties. In order to quantify the amount of absorbed water, gravimetric analysis of the water content in the investigated PET films at different RH levels has been conducted by means of weight measurements.

II. THEORY

A. Water Diffusion in Solids

One-dimensional diffusion of a mass density m with time t and position x in a solid can be described by Fick's second law

$$\frac{\partial m}{\partial t} = D \frac{\partial^2 m}{\partial x^2}, \quad (1)$$

where D is the diffusion constant [15]. The time-dependent amount of a diffusing substance Δm (e.g. water) in relation to the saturation value Δm_{sat} (at infinite time) across the polymer sample thickness h is given by [16]

$$\frac{\Delta m}{\Delta m_{\text{sat}}} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{\exp[-D(2n+1)^2 \pi^2 t / h^2]}{(2n+1)^2}. \quad (2)$$

For values of $\Delta m / \Delta m_{\text{sat}} < 0.6$, (2) can be simplified to calculate D , using the initial slope $k = \Delta m / \sqrt{t}$ [17]:

$$D = \frac{\pi}{16} \left(\frac{h}{\Delta m_{\text{sat}}} \right)^2 \cdot k^2 \quad (3)$$

B. Broadband Dielectric Spectroscopy (BDS)

By excitation of a dielectric material with an (alternating) electric field, local dipoles (“bound charges”) are oriented and/or charge carriers (“free charges”) are able to migrate over macroscopic distances. These effects can be described by the frequency- and temperature-dependent complex permittivity

$$\varepsilon^* = \varepsilon_0 \varepsilon_r^* = \varepsilon_0 (\varepsilon_r' - j \varepsilon_r''). \quad (4)$$

In order to quantify dielectric losses, the loss factor

$$\tan \delta = \frac{\sigma_{\text{DC}} + \omega \varepsilon_0 \varepsilon_r''_{\text{pol}}}{\omega \varepsilon_0 \varepsilon_r'} = \frac{\varepsilon_r''}{\varepsilon_r'} \quad (5)$$

is often used. It is divided into a DC conductivity loss component and a polarization loss component $\varepsilon_r''_{\text{pol}} / \varepsilon_r'$. The real part of the complex conductivity σ^* (consisting of DC and AC components) can be calculated with the angular frequency $\omega = 2\pi f$ as follows [2]:

$$\sigma' = \omega \varepsilon_0 \varepsilon_r'' = \sigma_{\text{DC}} + \sigma_{\text{AC}}, \quad \text{with } \sigma_{\text{AC}} = \omega \varepsilon_0 \varepsilon_r''_{\text{pol}}. \quad (6)$$

The temperature dependence of the DC conductivity σ_{DC} can be expressed with the activation energy E_a , the Boltzmann constant k_B and a pre-factor σ_0 in the following form [9]:

$$\sigma_{\text{DC}}(T) = \sigma_0 \cdot \exp(-E_a / k_B T) \quad (7)$$

III. EXPERIMENTAL

A. Materials

Measurements were conducted on semicrystalline, biaxially oriented PET film (*DuPont Melinex[®] S*) of 23 μm thickness.

B. Water Absorption Measurements

The water absorption in the cut PET samples was measured by means of gravimetric analysis with a high precision scale (*KERN ABT 100-5NM*, accuracy down to 0.01 mg). The sample weight at dry conditions was 532.21 mg. Before exposure to a specific RH level at room temperature (23 ± 1 °C), the film samples were dried in an oven for > 1 day at 50 °C according to EN ISO 62. Additionally, a desiccant was used during drying to remove remaining humidity from the atmosphere. After cooling down to room temperature under a constant flow of dry air, the samples were exposed to the desired RH level in an experimental setup as shown in Fig. 1 on the left side. A dry air flow is separated into two branches, of which one passes through a distilled water bath. By changing the volume flow of each branch, the RH level of the test cell (connected in flow direction behind the mixing chamber with RH sensor) can be controlled.

Due to the low sample thickness of the PET film, a fast diffusion process was expected. Accordingly, care needed to be

taken that weight measurements at each time step were carried out below 10 s to not influence water absorption by the ambient RH level (instead of the RH level in the test cell). A steady-state value Δm_{sat} was assumed when the measured weight change Δm did not vary more than 3 % over a period of 10 min.

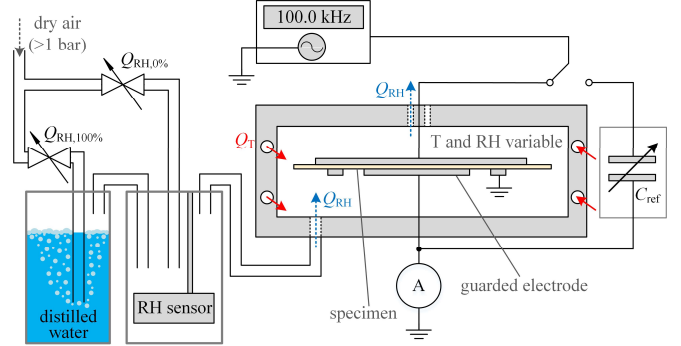


Fig. 1: Schematic of the broadband dielectric spectroscopy setup, using the reference air capacitor measurement principle (right) [19]. The three-electrode test cell (middle) includes both a temperature and humidity control circuit (left).

C. Broadband Dielectric Spectroscopy (BDS) Measurements

All BDS measurements have been carried out with the three-electrode test setup shown in Fig. 1. The principle is based on measuring the current flow through the sample as well as through a low-loss reference capacitor with air between the electrodes ($\varepsilon_{r,\text{air}} \approx 1$). This capacitor is adjusted automatically to eliminate influences of system nonlinearities. The sinusoidal excitation voltage was generated by a *Keysight 33522B* function generator and amplified with a linear amplifier *WMA-300 (Falco Systems)* afterwards. The current was measured via transimpedance amplifier (*FEMTO DLPCA-200/DDPCA-S*) and recorded (like the excitation voltage) by means of a *TiePie HS5*. More detailed information about the principle and precision of the measurement setup is given in [18,19]. Temperature changes are realized by active heating/cooling of a circulating oil flow through the test cell walls. The humidity control circuit from Fig. 1 was built within this work and described in the previous chapter.

The samples were prepared with silver painted electrodes (*Electrolube SCP*) to avoid measurement errors by an air gap occurring with pressed electrodes. A three-electrode design was used, even if the guard electrode (used to prevent measurements from parasitic surface currents) was found to have no significant influence. Drying of samples at 50 °C (well below the expected glass transition temperature of 80 °C [12]) and RH = 0 % were carried out for > 1 day prior to each BDS measurement. For investigated RH variations, the samples were stored at the desired RH atmosphere for > 1 day to reach steady-state absorption. This assumption could be validated by water absorption measurements, see Fig. 2. The test conditions during the BDS measurements in this work are given in Tab. 1.

Tab. 1: Test parameter values throughout the presented BDS measurements

Excitation voltage	Frequency range	Temp. in test cell	RH in test cell	Ambient conditions
100 V (peak-peak)	$10^2 \dots 10^5$ Hz	10...125 °C	0.80 %	$T = 23 \pm 1$ °C RH = 35 ± 10 %

IV. RESULTS

A. Water Absorption Measurements

The relative mass gain Δm was measured for different RH levels over time and plotted as a function of \sqrt{t} in Fig. 2. This allows for calculating the diffusion constant from (3) by means of the initial slope $k = \Delta m/\sqrt{t}$. The average value $D \approx 3.9 \text{ cm}^2/\text{s}$ (from the evaluated RH levels with a minimum value of $3.54 \text{ cm}^2/\text{s}$ and a maximum value of $4.99 \text{ cm}^2/\text{s}$) leads to a saturation time $t_{\text{sat}} \approx 11.5 \text{ min}$, which was used to approximate Fick's law (2) in Fig. 2. Furthermore, the results clearly show a linear increase of the saturated mass gain Δm_{sat} with RH level.

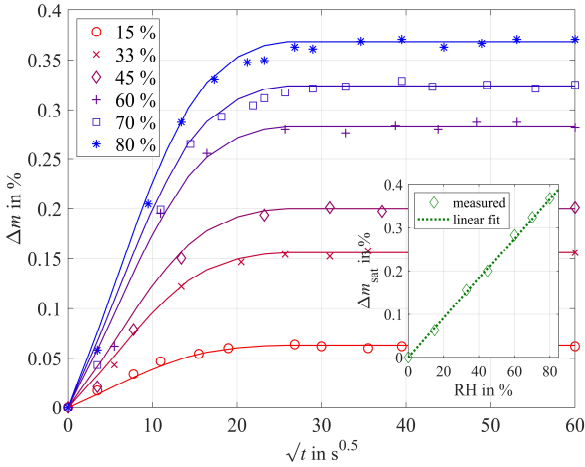


Fig. 2: Relative mass gain of PET samples ($23 \mu\text{m}$) as a function of square root of time at $23 \pm 1 \text{ }^\circ\text{C}$ for different RH levels. The solid lines are calculated curves according to Fick's law of diffusion. The small inset plot shows the relation between the relative saturated weight gain and corresponding RH level.

B. BDS: Temperature Variation Results

In Fig. 3, the BDS results for dry PET foils at varying temperatures are subdivided into the frequency spectra for the magnitude of the complex relative permittivity $|\epsilon_r^*|$ (top) and the loss factor $\tan\delta$ (bottom). In the high-frequency range, a loss peak emerges, which is shifted to higher frequencies (outside of the measured window) with increasing temperatures. For lower frequencies, a second peak occurs, which is located in the shoulder of the loss factor increase at low frequencies and high temperatures. This peak becomes visible only for $T \geq 85 \text{ }^\circ\text{C}$. In the same temperature range, $|\epsilon_r^*|$ increases more strongly.

C. BDS: RH Variation Results

Fig. 4 shows similar spectra as in Fig. 3, but for varying RH levels at $10 \text{ }^\circ\text{C}$. An approximately linear increase with humidity is observed for $|\epsilon_r^*|$, see also Fig. 5. The high-frequency relaxation mechanism in Fig. 4 increases slightly with rising RH level. Significantly higher (RH-dependent) dielectric loss changes are observed at lower frequencies. In the mid-frequency range a new peak appears only above a certain humidity level (here for $\text{RH} = 33 \%$ and above). Its magnitude could not be found to be humidity-dependent, but its position is clearly shifted to higher frequencies with increasing moisture content (compare with Fig. 5). It should be mentioned that the reported changes are reversible. After cooling and drying of the samples, the original dry weight was reached again and also the dielectric

spectrum returned to its original values (measurements not shown in this work).

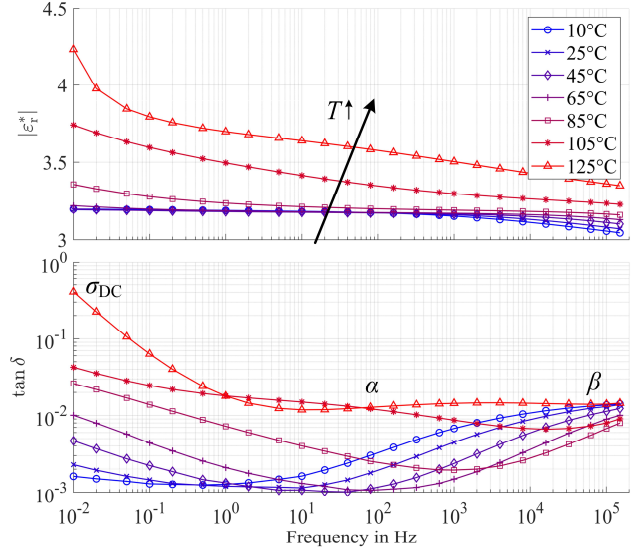


Fig. 3: Variation of the magnitude of the complex relative permittivity (top) and the loss factor (bottom) with frequency at different temperatures for dry PET foils ($\text{RH} = 0 \%$). The identified loss mechanisms are indicated in the picture.

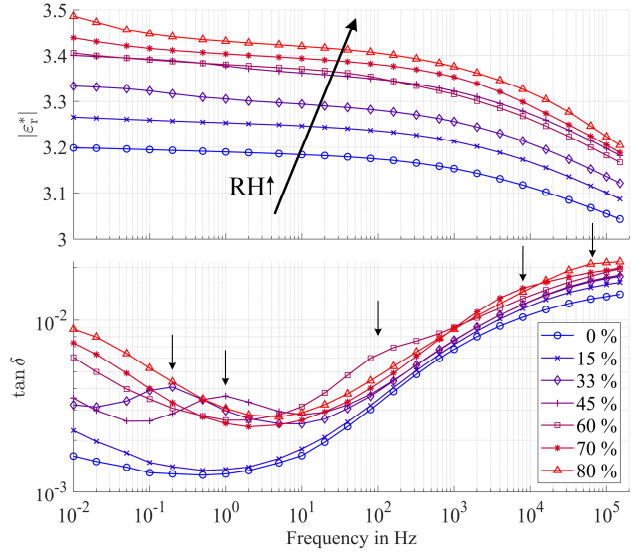


Fig. 4: Variation of the magnitude of the complex relative permittivity (top) and the loss factor (bottom) with frequency at $10 \text{ }^\circ\text{C}$ for PET at different relative humidity (RH) levels (saturated water absorption). The mid-frequency loss peaks are indicated by arrows in the picture.

V. DISCUSSION

A. Water Absorption Measurements

The observed relative amount of absorbed water (0.37% at $\text{RH} = 80 \%$) during our measurements is in accordance with earlier findings [12,20]. According to Fig. 2, the good agreement of measured data and calculated absorption curves demonstrates that the simplified relation (3) can be applied to describe the water absorption behavior in PET foils according to Fick's law of diffusion. The average value $D \approx 3.9 \text{ cm}^2/\text{s}$, determined in the present contribution, is similar to the values found by Yasuda et

al. [21] ($D \approx 3.99 \text{ cm}^2/\text{s}$ at $25 \text{ }^\circ\text{C}$) and Langevin et al. [22] ($D \approx 4.5 \text{ cm}^2/\text{s}$ at $20 \text{ }^\circ\text{C}$). The linear relationship between the saturated mass gain and the corresponding RH level, measured in this work, has also been observed by other authors [20,21].

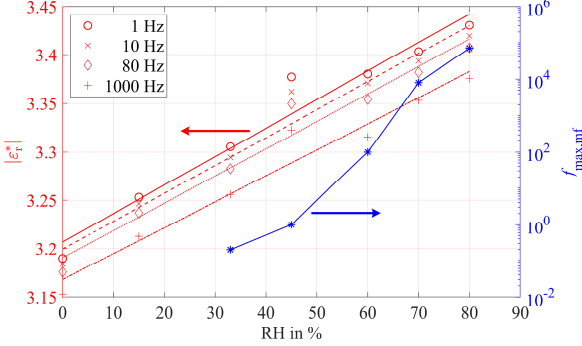


Fig. 5: Variation of the magnitude of the complex relative permittivity versus relative humidity (RH) level at different frequencies with linear fits (left axis) and frequency shift of the mid-frequency loss peak with RH level (right axis).

B. Temperature Variation: Loss Mechanisms

By comparison of our BDS results with former studies [3,7,11], it becomes clear that the high-frequency loss mechanism can be assigned to the β relaxation, see Fig. 3. It is visible at all elevated temperatures and shifted to higher frequencies with increasing temperature. The additional loss peak that appears only for $T \geq 85 \text{ }^\circ\text{C}$ is an indication of the α relaxation, which is activated above the glass transition temperature ($T_g \approx 80 \text{ }^\circ\text{C}$ for PET [12]). This coincides with a stronger $|\epsilon_r^*|$ increase with temperature over the whole frequency range. Above a certain temperature the β peak seems to coalesce with the α peak to form one single ($\alpha\beta$) process. This is characteristic for polymers with a local β relaxation above its glass transition temperature [23]. For very low frequencies and high temperatures in Fig. 3, the strong loss factor increase can be explained by the growing contribution of DC conductivity according to (7). This is supported by Fig. 6, where at $125 \text{ }^\circ\text{C}$ DC conductivity becomes visible by the emerging plateau of σ' in the low-frequency range. For higher temperatures than investigated in our experiments, additional electrode polarization could be observed at very low frequencies [9].

C. RH Variation: $|\epsilon_r^*|$ Increase and β Relaxation Enhancement

Despite the low water absorption saturation level of PET, significant changes in the dielectric properties could be detected with varying RH level. The linear permittivity increase in Fig. 4 is most likely due to the higher polar content (i.e. number of water dipoles that can readily respond to the applied field), introduced by the absorbed moisture [16]. This linearity is shown in Fig. 7 for different frequencies. The linear relation between $|\epsilon_r^*|$ and the RH level can be expressed as follows:

$$|\epsilon_r^*| = |\epsilon_{r,0}^*| + (A - |\epsilon_{r,0}^*|) \cdot \text{RH}/100\% \quad (8)$$

At dry conditions, a value $|\epsilon_{r,0}^*| = 3.19$ was found. The variable A in (8) represents the permittivity value at $\text{RH} = 100\%$ and has a value of $A = |\epsilon_{r,\text{water}}^*| = 80.13$ [16,24] for pure water. The results in Fig. 7 on the one hand support the hypothesis that mainly pure water leads to the permittivity increase, but on the other hand a small portion of water seems to not contribute to

this behavior. This can be seen by the better conformance of our data compared with (8) for a lower value $A = 70$ in Fig. 7.

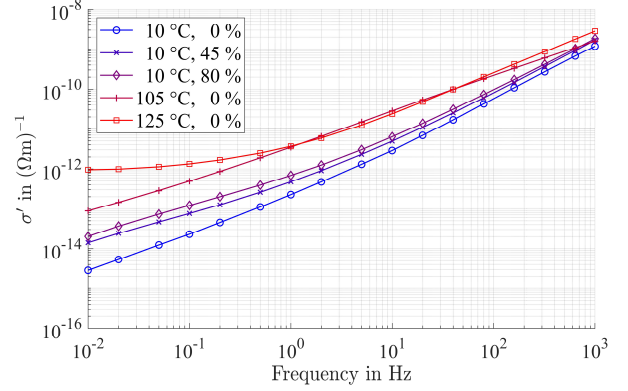


Fig. 6: Frequency variation of the (real part of the) conductivity at different combinations of temperature and relative humidity level.

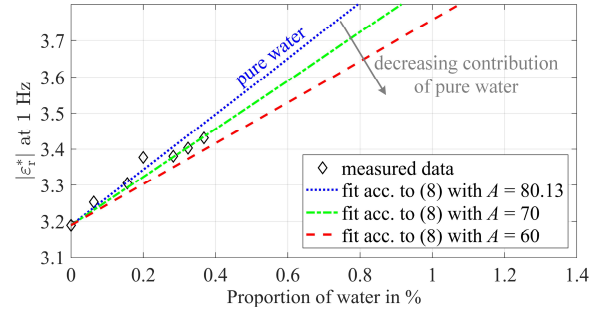


Fig. 7: Relationship between the magnitude of the complex relative permittivity (at 1 Hz) and the (weight) proportion of water in the PET sample. The data is fitted linearly according to (8) with values of A in a range from $A = |\epsilon_r^*| = 80.13$ (relative permittivity of water [16,24]) down to $A = 60$ at assumed 100 % water.

An explanation for the better approximation of our data by (8) for $A \approx 70$ (instead of 80.13 for pure water) in Fig. 7 is given by the frequency behavior of the loss factor. From Fig. 4 it can be seen that the β relaxation is enhanced by increasing RH level. This means that the material-inherent β relaxation is altered by water ingress. Lightfoot et al. [14] found that water molecules are likely to form van der Waals bonds to terminal $-\text{OH}$ groups of the PET chain. Since motion of these $-\text{OH}$ groups can be an origin for the β process [5], it is assumed that water does not only exist purely in the polymer, but also bonds to $-\text{OH}$ groups.

D. RH Variation: Mid-frequency Relaxation Process

In the present contribution, a new relaxation peak was found which becomes visible only for $\text{RH} \geq 33\%$ in Fig. 4. Its magnitude does not change much, but the frequency of the local loss maximum $f_{\text{max,mf}}$ is shifted to higher frequencies when increasing RH, see also Fig. 5. In principle, there can be two reasons for this. One explanation is based on the fact that water can act as a plasticizer, thus lowering T_g [24,25]. This facilitates easier motion of some polymer groups, which therefore shifts the relaxation peak to higher frequencies [26,27]. Jabarin et al. found the lowering effect of T_g at $\text{RH} = 80\%$ to be $\Delta T_g \approx 16 \text{ }^\circ\text{C}$ (compared to dry conditions) [20]. Since our measurements were performed at $10 \text{ }^\circ\text{C}$ (well below $T_g \approx 80 \text{ }^\circ\text{C}$ [12]), a plasticization effect as explanation for the observed peak shift is unlikely. The other possible reason is based on the presence of

(loosely bound) water layers with increasing conductivity which is related to higher water content [28]. Thus, it is assumed that water existing (e.g. in colloidal form [29]) in the free volume of the polymer is the driving factor behind this newly found peak.

E. RH Variation: DC Conductivity Increase

Below 1 Hz in Fig. 4, $\tan\delta$ increases with lower frequency. By using (6) to calculate the frequency-dependent conductivity σ' for both temperature and RH variation, Fig. 6 is obtained. It is evident that not only higher temperatures lead to a σ' increase, but also higher RH levels. The former can be explained with the exponential temperature dependence of the DC conductivity in (7). A similar DC conductivity enhancement is observed for increasing RH level in Fig. 6, indicating a higher number of mobile charge carriers. These can be due to the water molecules themselves or because of ionic molecules in the bulk dissociated into ionic charge carriers (due to the polar nature of water) [2].

VI. CONCLUSION

The key findings of the present investigation on PET foil are as follows (and in accordance with literature where applicable):

- The level of water absorption (measured gravimetrically) increases linearly with increasing RH level up to 0.37 % relative mass gain at RH = 80 %. Because of the good accordance with Fick's law of diffusion the diffusion constant can be calculated as $D \approx 3.9 \text{ cm}^2/\text{s}$.
- BDS measurements revealed the temperature dependent α and β relaxations as well as DC conductivity effects.
- RH variation leads to a linear permittivity increase that was found to be mainly due to pure water (additional polar content). A small amount of water seems to be bound via van der Waals bonds to terminal -OH groups, thus contributing to the β relaxation in PET.
- A new loss peak was found to occur only above a certain RH level, which exhibits a shift of its peak frequency to higher frequency values with increasing humidity. It can most probably be attributed to the water molecules existing within the free volume of the polymer.
- It was shown that DC conductivity increases not only with higher insulation temperature, but also with higher RH level and related higher amount of absorbed water.

Consequently, significant changes in dielectric properties could be identified despite the low maximum water absorption. In the future, similar investigations will be performed on further materials used for similar applications (e.g. motor insulation). In a next step, (destructive) lifetime testing of the studied materials will be performed, which enables a better understanding of possible failure mechanisms of polymeric insulation materials.

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