THE COMBINED DIELECTRIC SPECTROSCOPY INVESTIGATION IN A WIDER FREQUENCY RANGE

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ABSTRACT

The subject of this paper is testing of the improved time domain apparatus and combined dielectric relaxation spectroscopy measurement in time and frequency domain. The focus of interest is the experimental measurement of test dielectric samples and its interpretation with the objective to determine the best metering configuration for future applications in diagnostics and to analyze the physical origin of the dielectric spectra obtained.

1. INTRODUCTION

We know from the theory that an ideal dielectric or an insulator is made of substance, which contains charges bound only by electrostatic forces [3]. The basic electrical quantities describing the properties of dielectrics in electrical field over specific temperature, humidity and frequency are: relative permittivity (ε'), internal resistivity (ρ_V), surface resistivity (ρ_p), dissipation factor ($tg\delta$) and breakdown strength (E_P). The relative permittivity is constant of proportionality between capacity and geometric capacity of dielectric sample (in case dielectric replaced by vacuum). The dissipation factor is degree of dielectric losses of power in parasitic leaking resistance. The loss number (ε'') is defined as $\varepsilon'.tg\delta$, the complex permittivity by term $\hat{\varepsilon} = \varepsilon'\text{-j}\varepsilon''$. The dielectric polarization is an effect, where the bound electrical charges move due to external or internal field from their equilibrium positions to new ones across small finite distances. The measure of the polarization in a material is the polarization vector ($\vec{P}$) and relative permittivity (ε'). The polarization vector is defined as the density of dipole moment in dielectric; the time dependence of polarization is generally given by a response function ($\Phi(t)$):

$$P(t) = \varepsilon_0.\varepsilon_\infty.E + \varepsilon_0.(\varepsilon_S - \varepsilon_\infty).E.\Phi(t) \ , \qquad (1)$$

where E is electrical field strength, ε_∞ denotes optical permittivity at high frequencies ($f\to\infty$), ε_S is static permittivity at low frequencies ($f\to 0$).

2. DIELECTRIC RELAXATION SPECTROSCOPY THEORY

The subject of this paper is the combined measurement by time-domain dielectric relaxation spectroscopy (DRS) and frequency-domain DRS. The focus of DRS is to obtain the dependence of complex permittivity on frequency and other parameters. Frequency domain spectroscopy is based on alternating current measurements. Time domain DRS is used for frequency range starting at very low frequencies (about a few μHz) and is based on direct current measurement [1].

The current density of charging/discharging currents is given:

$$j_{POL}(t) = \frac{\partial P}{\partial t} + \gamma.E \ , \ j_{DEPOL}(t) = \frac{\partial P}{\partial t} \ , \tag{2, 3}$$

where the term $\gamma.E$ represents the part due to the conduction current through a dielectric. The time dependence of charge/discharge current densities is given by the decay function:

$$j_{DEPOL} = \varepsilon_0.(\varepsilon_s - \varepsilon_\infty).\varphi(t).E \ , \ \varphi(t) = \frac{d\,\Phi(t)}{dt} \ . \tag{4, 5}$$

Time-domain DRS is mostly done with discharge currents, because the depolarization current does not contain the conduction term (Fig. 1). This measurement starts to record the time response (current) by step changing voltage. After the time-domain measurements, it is necessary to convert the discharge current data into the frequency domain; the complex permittivity and the measured discharge characteristic are mutually related by the Fourier transformation:

$$\hat{\varepsilon} = \varepsilon_\infty + (\varepsilon_S - \varepsilon_\infty)\int_0^\infty \varphi(t) e^{(-j\omega t)} dt \ . \tag{6}$$

Like every measurement technique, the time-domain DRS exhibits also specific troubles brought about by measuring circuit parameters as e.g. very low working currents for long periods of time. The basic block diagram of the time-domain DRS set-up is shown in Fig.1.

3. MEASUREMENT SET-UP

The time-domain dielectric relaxation spectroscopy is on the instrument side implemented as the measurement of discharge currents. Programmable electrometer Keithley 617, available in our department, was used for the measurement, alongside with the required hardware equipment and software [2].

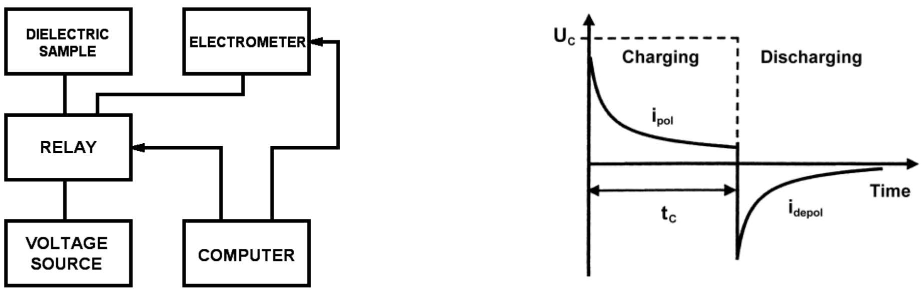

Fig. 1: Time domain DRS set-up and dielectric discharging process [4]

The above mentioned block scheme on Fig. 2 represents common solution of dielectric testing system, working in time-domain. We can use internal auxiliary power supply of Keithley 617 electrometer to charge the test sample in our case. But we need to control the contact switching system by some way. One easy method with some disadvantages, how to do it, is handy activation of the contact system. We have tested the modified automatic variant of this metering system with Keithley 617 electrometer on our department. We were trying two measuring shielding chambers with contact switching system based on the corred relays. Present system uses internal auxiliary supply of the electrometer for control the relays; the electrometer power source is easy full controllable from the computer. The electrometer supply controls 3 outputs for relays, across protection separation circuit and electronic logic unit. The system allows to automated measure after hand start of metering cycle with all needed procedures, as defined discharging and defined charging of the sample before testing. We need another one power supply for charging of the dielectric sample with using this system.

As it was mentioned above, we tested two types of contact switching systems for control of the sample circuit. The first common system used 3 corred relays, connected to support possible measuring of the charging current. We developed the second type of the contact switching system for maximum suppression of not-wanted noise signals in our conditions. The dielectric test sample is connected short to the switching (normal closed) terminals of only one corred relay. The input terminals of the electrometer are connected in parallel to this dielectric sample; crocodile clips are connected to the terminals of mentioned corred relay. We need another one relay yet, to full observe discharging/charging of the sample before the testing period; it can be used any universal low power relay. This solution provides that the surface and capacity of used conducting wires in input circuit of electrometer is low during the testing process, so the inducted noise to the sensitive input circuit is smaller in given ambient conditions.

4. TESTING

We were testing the sample of carboxymethylcellulose film (CMC) with trade description "blanosa" in time-domain. The test sample was tested with common three-relay apparatus at first, and then by improved two-relay apparatus. The original measured signals of discharge dependences are shown on Fig. 2, including already not useful end part; charging time 5 hours.

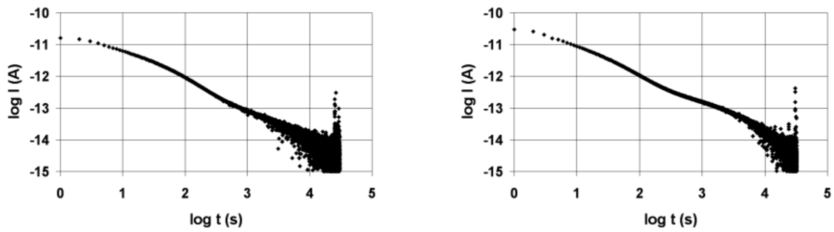

Fig. 2: The measured discharge characteristic with a) 3 relay b) 2 relay set-up

As we can see, the signal measured with the improved two-relay apparatus has a little higher value and has a less noise in comparison to common three-relay apparatus. The smoothed discharge dependence, suitable for next process, is shown on Fig. 3. The original signal was inspected for high errors, which can occur in some time point and are recorded. The next operation with this data was the locking out of the current samples, which was out of placed limit from the ambient samples average; this operation was automatically applied only for first 100 current samples. The rest part of the signal was averaged by useful statistic median function.

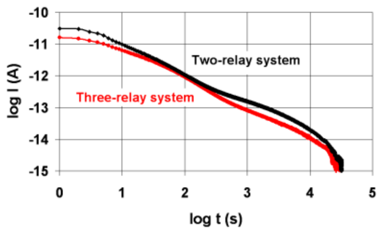

Fig. 3: Smoothed discharge characteristic of the both set-ups

The better signal from improved apparatus was transformed to the frequency-domain after the smooth process. The dependent of relative permittivity was valuated using the Fourier transformation (FT); the frequency dependents of loss number were calculated by FT and Harmon's approximation for comparing of these methods (Fig. 4). The straight FT from the real signal known only in finite time window generates periodical noise to the transformed signal.

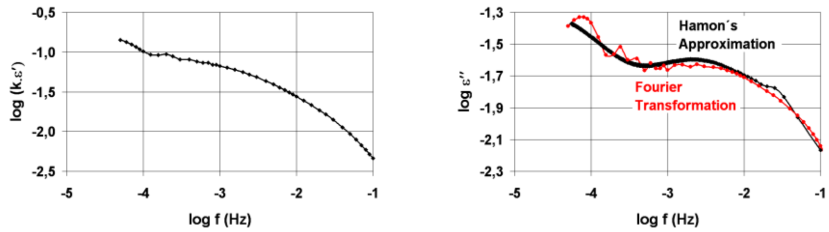

Fig. 4: Relative permittivity and loss number dependence

The next parts of measurements was proceed in frequency-domain. The parameters of dielectric sample measured with impedance analyzer HP 4284 A at room temperature are shown on Fig. 5.

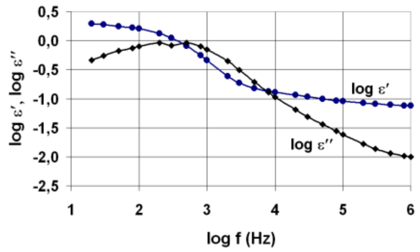

Fig. 5: Complex permittivity characteristics by HP 4284A

The test sample was also measured at the University of Augsburg by high-frequency analyzer HP 4291A in a wider range of temperatures; these data are shown in Fig. 6.

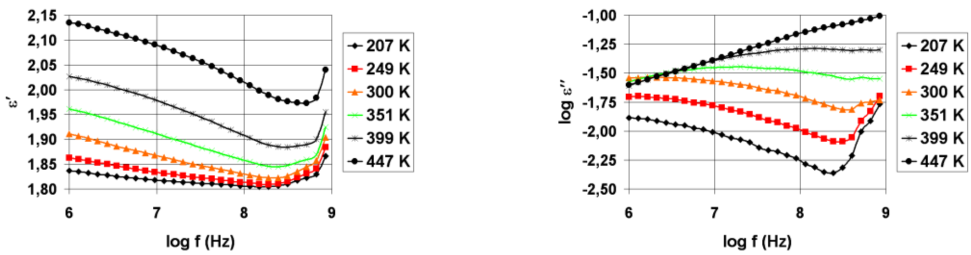

Fig. 6: Complex permittivity dependence by HP 4291A analyzer

The final picture shows the comparison of all presented outputs, which features the given test sample in wider frequency range.

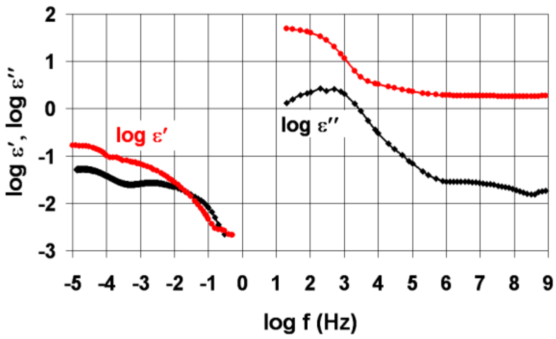

Fig. 7: Plot of relative permittivity and loss number vs. frequency in the full available frequency range (10^{-5} – 10^{9} Hz) at room temperature, with sectional application of scaling factors

5. CONCLUSION

The basic set-up and improved apparatus for dielectric current metering were described and tested. The paper shows the basic options, how to process the products of discharge current metering not only for purpose of dielectric relaxation spectroscopy. The results of time-domain measurements demonstrated, that it will be necessary to integrate the full digital filtering into the existing system in future. These results were also completed with frequency-domain measurements; available apparatus doesn't enable to cover the all useful frequencies.

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