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(F)LIMM deconvolution procedure for on-line measurements of space charge in thin polymeric insulations

Amanda Velazquez-Salazar, Laurent Berquez, Didier Marty-Dessus,
LAPLACE, Université de Toulouse, CNRS, INPT, UPS, France.

Abstract — On-line measurements are developed to monitor space charge evolution in a polymeric insulation film by the mean of a (Focused) Laser Intensity Modulation Method set-up. The purpose of this paper is to describe how, starting from experimental on-line (F)LIMM currents, one is able to develop a global procedure leading to extract space charge profiles in dedicated test samples. Objects of study are 25- μm films of PTFE submitted to a protocol of poling starting from 0V to 2.5 kV, alternating 6 volt-off and 6 volt-on measurements on each step of 500 V. Each measurement consists in recording a (F)LIMM pyro-current spectrum from 10Hz to 10 kHz hence a whole protocol is composed of 63 acquisitions of such spectra.

Once measurements performed on PTFE sample hereinabove described, a normalization process has allowed first to confirm that there was no charge evolution (injection) between two successive Volt-on to Volt-off measurements, and secondly to launch a thermal calibration of the bench that is needed in (F)LIMM experiment. Then a mathematical correction on Volt-on current was operated in order to extract the useful part linked to the internal field produced by the space charge. Finally, significant space charge representations and evolutions were obtained in our test samples by using a mathematical deconvolution procedure.

Keywords — (F)LIMM; insulations; polymers; space charge; polarization, on-line measurements

I. INTRODUCTION

On-line measurements are generally designating measurements of a specific physical quantity evolution on structures that are in a same time submitted to an external stress like for instance a DC electric field. In our case, the physical parameter to follow is the space charge evolution in a dielectric film by the mean of a (Focused) Laser Intensity Modulation Method ((F)LIMM) set-up. Indeed, space charge is usually known as an important role player on dielectric properties and more specifically on degradation and ageing [1].

That is why, many different techniques related to space charge investigation were developed these last 30 years [2], and the very first achievements of the LIMM technique were described by Lang in 1981 [3]. However, beyond all possibilities offered by these techniques, one question remains : what is the real impact of an external stress regarding to “live” space charge creation and behavior in a polymeric sample, and how could we answer to private companies, for which characterization of such materials should occur close to real industrial context?

To this aim, the (F)LIMM technique was adapted to on-line measurements. Objects of study are thin insulator films submitted to a protocol of an external DC poling, alternating volt-off (0 V) and volt-on (V_{ht}) steps. Each measure consists in recording a (F)LIMM pyro-current spectrum versus the frequency of modulation of the laser beam. From volt-on and volt-off measurements, a mathematical and complex treatment based on normalization, calibration, correction and deconvolution procedure is developed. It gives the possibility to show the space charge global evolution over the whole poling process, in particular during the volt-on steps. To sum-up, the whole process consists in 4 successive steps:

- LIMM on and off-line current measurements,
- normalization of currents,
- calibration and correction of the thermal model,
- deconvolution of the normalized currents.

II. ON-LINE PROTOCOL AND MEASUREMENTS

A. Experimental bench

The (F)LIMM traditional bench and the modified one dedicated to on-line measurements have been described in details in [4]. On Fig.1 are represented the main functional blocks developed to study the space charge evolution in thin films (typically a few tens on micrometers), coated by one or two metallic electrodes (typically gold of 100 nm-thickness). The heat source is provided by a laser diode switched periodically in an on/off mode at frequency f , producing a heat diffusion in the sample under study. Varying the frequency leads to a modification of the heat diffusion length, and then to probe more or less deeply within the structure.

By interaction of the local heat with the space charge, an AC pyro-current is produced at frequency f (LIMM current) as a consequence of the rebalancing of charges between the electrodes after heat perturbation. This one is pre-amplified, extracted from noise by using a lock-in amplifier and then recorded versus frequency (real and imaginary parts).

Regarding to on-line modifications, two more blocks were added (in grey in Fig. 1): a DC potential (V_{ht}) is applied to the top electrode of the sample, and an electrical protection in case of eventual breakdown of the sample. Effects of these

experimental modifications have been carefully studied and characterized in [4].

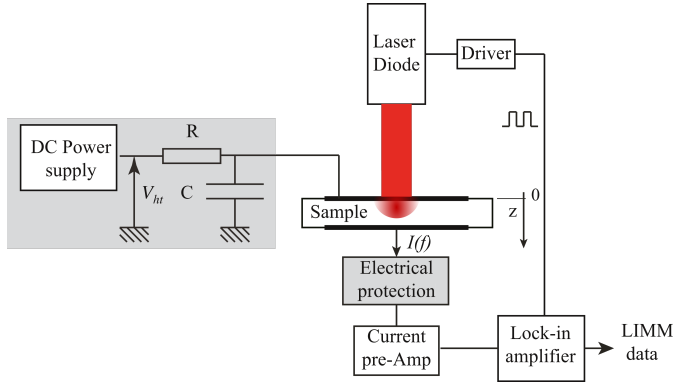


Fig. 1. (F)LIMM "on-line" set-up

B. On/Off-line LIMM current measurements

Sample of study are thin films of PTFE (25 μm -thickness Polytetrafluoroethylene). A cumulative protocol of measurement is depicted in Fig. 2: a DC electric field ranging from 10 to 100 kV/mm (corresponding to external potential from 250 V up to 2.5 kV) is applied with different steps. After each polarization step, a depolarization process is performed by short-circuiting the sample. The full protocol consists in 63 LIMM measurements over approximately 10 hours. Each LIMM data acquisition lasts 10 minutes, and 6 of them are successively conducted in volt-off (0 V) and volt-on (V_{ht}) modes.

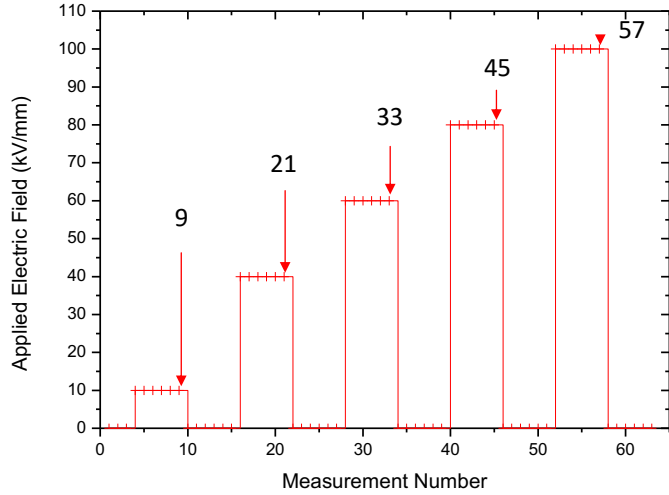


Fig. 2. Protocol of measurement

Considering a laser beam heating a surface S at the top sputtered electrode of the sample of thickness L , the volt-off and volt-on current may be expressed as [4]:

$$I_{volt\ off}(f) = -j \frac{2\pi f}{L} (\alpha_z - \alpha_\epsilon) \epsilon S \int_0^L E_{SC}(z) T(z, f) dz \quad (1)$$

$$I_{volt\ on}(f) = -j \frac{2\pi f}{L} (\alpha_z - \alpha_\epsilon) \epsilon S \left(\int_0^L E_{SC}(z) T(z, f) dz + \frac{V_{ht}}{L} \int_0^L T(z, f) dz \right) \quad (2)$$

where z is the direction normal to the sample, α_z the coefficient of thermal expansion (K^{-1}), α_ϵ the coefficient of thermal dependence of the dielectric permittivity (K^{-1}), ϵ the permittivity, E_{SC} the internal electric field resulting from inner space charge along the z -axis ($\text{V}\cdot\text{m}^{-1}$), V_{ht} the external electrical constraint (V) and $T(z, f)$ the simulated spatial variations of the temperature versus frequency.

Fig.3 represents all the LIMM currents recorded in volt-on (top) and volt-off (bottom) mode.

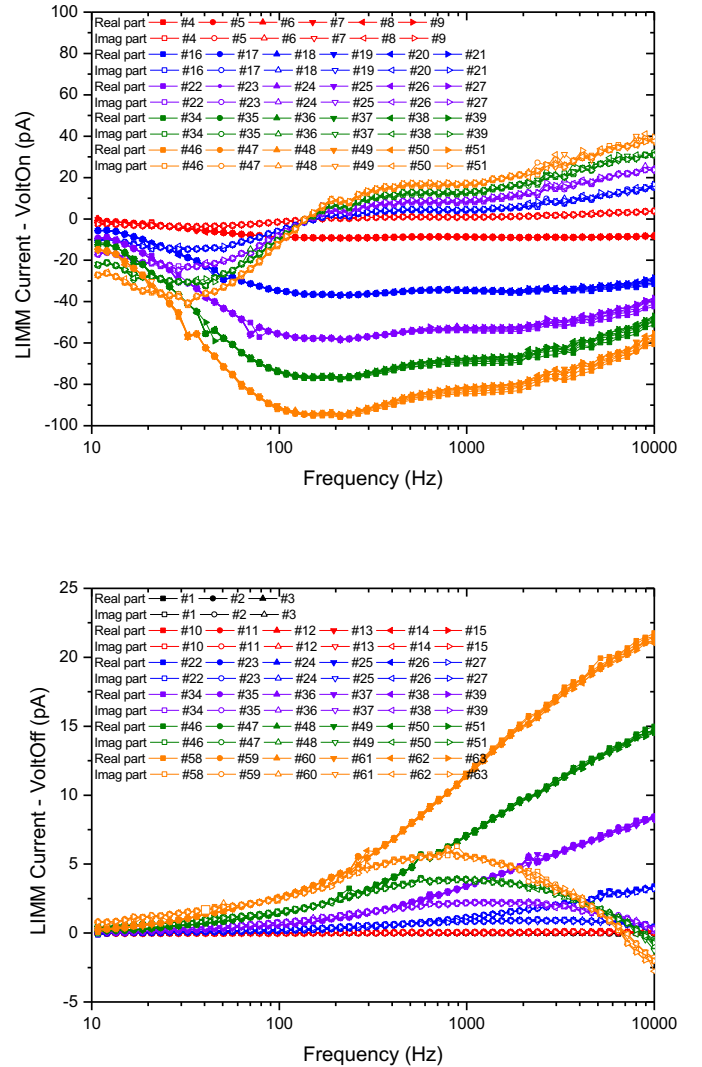


Fig. 3. LIMM Volt-on (Top) /off (Bottom) currents on PTFE

C. Calculation of a (F)LIMM normalized current

The normalization process has two objectives: firstly, to prove that there is no charge injection between two successive measurements and secondly, to allow a thermal calibration of the bench.

The calculation of a normalized current should be the result of the measurement of a LIMM current if 1 V is applied to the sample, then:

$$I_{1V}(f) = \frac{I_{voltOn}(f) - I_{voltOff}(f)}{V_{ht}} = -j \frac{2\pi f}{L^2} (\alpha_z - \alpha_\varepsilon) \varepsilon S \int_0^L T(z, f) dz \quad (3)$$

At this stage, the assumption is that E_{SC} remains constant between two successive measurements. This can be proven if one represents the calculates 5 normalized currents at different measurement steps: #9, #21, #33, #45 and #57 (they correspond to the 6th data acquisition in the volt-on mode). All correspond to the last measure in volt-on mode for different external applied fields (Fig. 4).

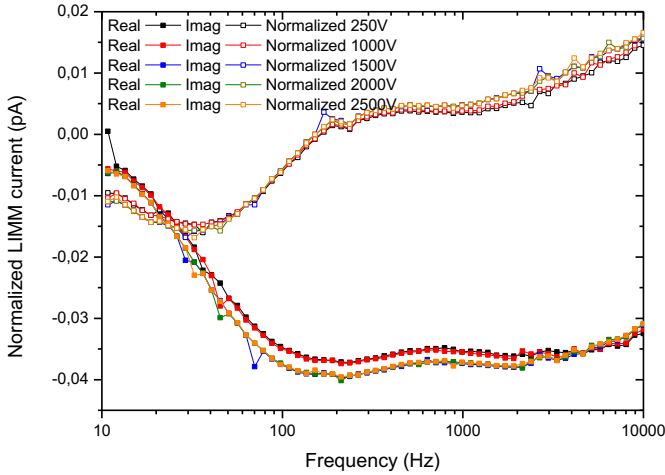


Fig. 4. Volt-on normalized currents - Real part (filled) and Imag. part (open) circles

Considering that if one applies 1 V to the sample, no charge injection should occur, then all normalized current should be identical, and our assumption remains valid. It is then possible to envisage calibrating the thermal model, considering that a direct link exists between $I_{1V}(f)$ and the thermal gradient according to (3).

D. Calibration and correction of the thermal model

In order to proceed to the thermal calibration and correction (see details in [5]), $I_{1V ave}(f)$ the mean of the 5 normalized currents is calculated, then injected in the thermal model to compare experimental and simulated currents (Fig. 5).

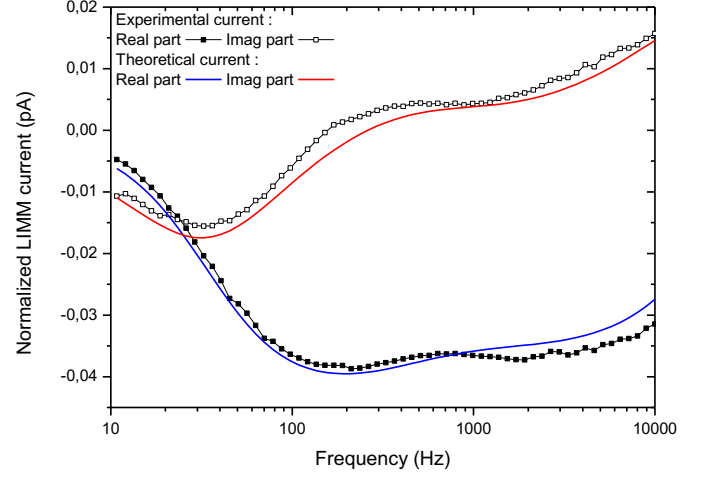


Fig. 5. Thermal calibration ($V_{ht}=1V$)

The good agreement between the experimental current data and the simulated one depicted in Fig.5 allows to conclude that our mathematical thermal model is calibrated to fit correctly the real heat diffusion process in the sample.

E. Deconvolution of the normalized current

The last step is to now calculate the correction that one has to conduct on $I_{voltOn}(f)$ in order to extract from it the sole part linked to the presence of the inner space charge in the sample, then:

$$I_{voltOn Corrected}(f) = I_{voltOn}(f) - V_{ht} \cdot I_{1V ave}(f) \quad (4)$$

At this stage, $I_{voltOn Corrected}(f)$ (4) will be used as the entry data in the mathematical process of deconvolution, leading to space charge cartographies (Fig. 6).

The deconvolution process consists in the inversion of the (F)LIMM current equation (3). However, the inversion process is a complex one because this equation corresponds to a Fredholm equation of first kind which particularity is a non-unique solution. Hence, to find a mathematical solution nearest to the experimental (F)LIMM current the Tikhonov regularisation method is adapted and applied [6]. Thus, the optimal solution is found and the (F)LIMM current equation can be then inversed. Thanks to this mathematical treatment, it is possible to locate, distinguish and treat the influence of the experimental noise from the numerical one. Then, it is possible

to correctly inverse the (F)LIMM current equation. Finally, once the inversion is carried out, it is easy to have access to the space charge distribution.

Then it is observed that the thin film insulator sample submitted to a total poling protocol, presented a displacement of space charges towards the anode (up). The negative charges are closer to the anode than the positive ones. These last have an important concentration relative to the intensity of the external electrical constraint applied. These results confirm the validity of this (F)LIMM on-line method to follow up the evolution of space charges on thin polymeric layers.

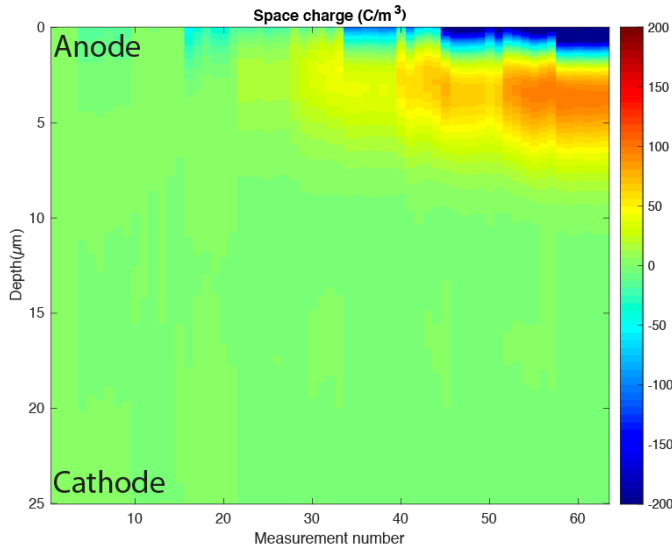


Fig. 6. Space charge evolution

III. CONCLUSION

A new step was reached with the (F)LIMM technique: beyond being able to perform on-line measurement under DC stress, the procedure used has led to a real tracking of space charge evolution during a classical volt-on / volt-off poling procedure. The envisaged next study will consist in following space charge evolution by (F)LIMM under DC stress and controlled thermal environment of the sample in order to be closer to real conditions of utilization in industrial systems.

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