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## A time-resolved current method and TSC under vacuum conditions of SEM: Trapping and detrapping processes in thermal aged XLPE insulation cables



BEAM INTERACTIONS WITH MATERIALS AND ATOMS



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#### ABSTRACT

Thermal aging of cross-linked polyethylene (XLPE) can cause serious concerns in the safety operation in high voltage system. To get a more detailed picture on the effect of thermal aging on the trapping and detrapping process of XLPE in the melting temperature range, Thermal Stimulated Current (TSC) have been implemented in a Scanning Electron Microscope (SEM) with a specific arrangement. The XLPE specimens are molded and aged at two temperatures (120 °C and 140 °C) situated close to the melting temperature of the material. The use of SEM allows us to measure both leakage and displacement currents induced in samples under electron irradiation. The first represents the conduction process of XLPE and the second gives information on the trapping of charges in the bulk of the material. TSC associated to the SEM leads to show spectra of XLPE discharge under thermal stimulation using both currents measured after electron irradiation. It was found that leakage current in the charging process may be related to the physical defects resulting in crystallinity variation under thermal aging. However the trapped charge can be affected by the carbonyl groups resulting from the thermo-oxidation degradation and the disorder in the material. It is evidenced from the TSC spectra of unaged XLPE that there is no detrapping charge under heat stimulation. Whereas the presence of peaks in the TSC spectra of thermally aged samples indicates that there is some amount of trapped charge released by heating. The detrapping behavior of aged XLPE is supported by the supposition of the existence of two trap levels: shallow traps and deep traps. Overall, physico-chemical reactions under thermal aging at high temperatures leads to the enhancement of shallow traps density and changes in range of traps depth. These changes induce degradation of electrical properties of XLPE.

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### 1. Introduction

Cross-linked polyethylene (XLPE) is promoted to be the selected material which can be used in high voltage cables insulation because it exhibits a very interesting combination of properties such as the non-polar chemical formulation, very low dielectric losses factor, easy processing, flexibility and low cost [1]. Nevertheless, a consequence of its limited conductivity is that this material readily become electrostatically charged which limits its advantages in the high voltage direct current power transportation use [2]. Moreover, the long term dielectric strength of polyethylene

\* Corresponding author. *E-mail address:* sebastien.rondot@univ-reims.fr (S. Rondot). is strongly dependent on the space charge characteristics which have not yet been very well understood and evaluated [3–5].

Many factors can cause the cable failure: production faults (voids, impurities), incorrect handling during installation, inappropriate mechanical and electrical use and aging of polymeric insulation under service conditions. This latter presents the most important cause of the cable failure. Under service conditions, the cable is permanently subjected to electrical, thermal, mechanical and environmental stresses [6]. Under heat conditions, thermal aging occurs and causes an irremediable failure of the cable insulation. When high temperatures were applied on the insulating materials for long time, its chemical composition may change [7] and its physical morphology alters [8,9]. In consequence, almost of its characteristics can change. The main effects of thermal aging are highlighted by decreasing in volume resistivity, increasing of

dielectric losses [10] and finally mechanical properties drop down [11]. For this reason, the development of diagnostic techniques which give information on the aging process of the cable insulation is necessary [12]. It is known that additives and cross-linking by-products generate residual charge that plays an important role in the degradation processes. A wide variety of work was conducted worldwide with focus on the existence of correlation between dielectric breakdown and trapping-detrapping process of space charge, and its relation with polyethylene aging process [3,4,13]. Most of works try to correlate macroscopic properties (mechanical, electrical and optical...etc.) with microstructure, defects and impurities which act as traps of charges in the bulk and at the surface of insulators.

Many characterization techniques, such as acoustic and thermal techniques, have been used in the field of trapping and detrapping measurements of space charges in insulating materials [14]. In these methods, propagation of thermal or acoustic waves allows distribution of the space charges through the sample dimension to be determined. Usually these techniques use two steps of measurements; the sample is irradiated first, and only after the measurement of trapped charge is made. This way to measure the trapped charge does not lead to investigate the dynamic charging and discharging process of the material.

Here we used a time-resolved current method that was developed a few years ago [15] in a scanning electron microscopy (SEM). To study the charge trapping and detrapping of insulators, electrons are injected directly in the insulator using a relatively high energy electron beam. This SEM method is based on the electrostatic induction phenomena. The simultaneous measurement of temporal change of displacement and leakage currents can be carried out during charging and discharging periods. This technique proved its effectiveness especially in the characterization of materials for which part of trapped charge may be released [16,17] in order to study also the discharging step. To remedy the lack of discharge in the materials with a big capacity to trap and maintain charges for a long time [18-20], the so-called Thermally Stimulated Current method (TSC) is associated with the present SEM technique. The TSC measurements have been performed on already e-beam charged samples stimulated with heating from room temperature to temperatures close to the melting temperature of the material allowing the possibility of all the storage charge to be released. Unlike conventional TSC, our adapted arrangement in the SEM, allows the displacement current and the leakage current to be measured separately during electron irradiation. Moreover, the displacement current represents the dynamic aspect of the trapped and detrapped charge in the material whereas use of the leakage current, usually measured in conventional TSC, overestimates the trapped charge.

It should be emphasized that the manufacturers of HV cables usually recommend a service temperature of 90 °C for crosslinked polyethylene. This temperature is often near the melting temperature [21] which reinforces the thermal aging process. Moreover the behavior of XLPE in the temperature range of 110– 150 °C is of practical importance especially close to emergency operating temperatures for the material when used as insulation in power cables [22]. These considerations motivate us to use both proposed techniques (time-resolved current and TSC) together to study the thermal aging effect on the trapping and detrapping behavior of XLPE close to its melting temperatures range without electrode interfaces as for thermal and acoustic techniques.

### 2. Basic principles

The irradiation of thermally aged XLPE by mean of a penetrating electron beam with a primary current  $I_0$  induces two currents in

the sample and electron emission in the vacuum. A leakage current  $I_L$  which is the result of the evacuation of electrons through the volume or along the side surface of the sample towards ground. A displacement current  $I_d$  resulting from the change of trapped charges amount in the sample. The electron emission results from secondary and backscattered electrons escaping from the sample. The conservation law that links these different signals leads to:

$$I_0 = \sigma I_0 + \frac{dQ_t}{dt} + I_L \tag{1}$$

where  $\sigma$  represents the total electron emission yield (*TEEY*), and is defined by  $\sigma = \delta + \eta$ .  $\delta$  is the secondary electron (SE) yield.  $\eta$  is the backscattered electron (BSE) coefficient.

 $dQ_t/dt$  is the charging rate of the sample (trapping rate) and  $Q_t(t)$  is the net trapped charges in the sample due to the formation of holes left behind by secondary electron emission (positive charges), and trapped injected electrons (negative charges):

$$Q_t = \int (I_o - \sigma I_o - I_L) dt \tag{2}$$

In our case, the primary beam energy is in the energy range that charges sample only negatively.

The trapped negative charge in the sample produces a positive image charge  $Q_{im}$  in all conductor parts of scanning electronic microscope chamber. The created image charge in a grounded metallic electrode, which is not in electrical contact with the sample (see Fig. 1), is related to trapped charge with the electrostatic displacement factor *K* by the following equation:

$$Q_{im} = KQ_t \tag{3}$$

The factor K is deduced from the discharging step when the beam is blanked [23]. Considering the device Fig. 1, this image charge is related to the measured displacement current as follow:

$$I_d = \frac{dQ_{im}}{dt} = K \frac{dQ_t}{dt} \tag{4}$$

Then, after time integration, the time evolution of the total charge into the specimen is deduced knowing the factor K. This factor is less than the unity because the induction effect is not total here (see Fig. 1).



Fig. 1. Sectional view of the measurement device used for induced small currents acquisition.

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