

PART 2

## Phenomena Associated with Environmental Stress – Ageing

## Chapter 6

# Space Charges: Definition, History, Measurement

### 6.1. Introduction

Under the action of numerous factors (electric field, temperature, humidity, radiation, etc.), the properties of dielectric materials become degraded over time in an irreversible way. The mechanisms causing this phenomenon (called ageing), bring in all of the stresses which the insulator must obey, and are subjected to approaches described later in this chapter. Ageing generates unfavorable effects on electrical components, which can manifest themselves by the appearance of defects under much weaker stresses than those originally considered.

Due to economic and environmental factors, the past several years in the energy and electronic fields have witnessed a race for miniaturization of components and systems, and for the growth of the power densities they convey. This has led to an increase of electrical and thermal stresses to which the insulators are submitted. In order to guarantee the reliability of components and systems, it is of fundamental importance to know, in a more precise manner, the utilization limits of the insulators used and their long-term evolution.

An ideal insulator should not contain charges other than atomic cores, electrons in the internal layers and valence electrons. In reality, however, there is no such thing as an ideal insulator.

Thus, when an insulator is subjected to high stress (high electric field, irradiation, important mechanical efforts and friction, variations of temperature), an excess of electric charges, called *space charges*, appear in certain regions of the insulator. It seems more and more probable today that the issues of performance decrease and insulator breakdown are due to the presence and accumulation of space charges in bulk and on the surface. Indeed, the electric field caused by a space charge (known as a “residual field”) can locally increase the electrical stress, by over-soliciting the insulator. Moreover, during any massive detrapping of the accumulated charge, the energy released is considerable and can (either locally or completely) damage the material.

To illustrate this, remember that, when we apply an electric field to an insulator, any macroscopic discontinuity in the material (permittivity variation in a composite, for example) gives rise to classic interfacial (Maxwell–Wagner) polarization. This phenomenon also affects heterogenous materials at a microscopic scale. Moreover, following the internal order of the material, we notice the presence of traps (localized levels) at the molecular scale. These traps are likely to capture charges, producing polarization at a microscopic scale. Thus, one more or less electronic charge on a macromolecule of 100 Å of cross-linked polyethylene gives a density of  $10^{24}$  electrons/m<sup>3</sup> in three dimensions, i.e. a charge density  $\rho = 1.6 \cdot 10^5$  C/m<sup>3</sup>, which corresponds (integrated on a thickness of 0.1 μm) to a field of 1,000 kV/mm. Even at a lower degree, it is then very obvious that this type of trapping can cause an early dielectric breakdown or/and ageing of the material.

For the reasons described above, these electric charges which tend to accumulate in the insulators have been subjected to thorough studies for 20 years. In the following pages, we are going to tackle some fundamental and historical aspects related to the space charge, summarize the different methods developed to measure these charges and present current trends and perspectives in this area.

## 6.2. History

In the 1960s, notable advances were realized in the understanding of phenomena related to the application of solid-state physics: amongst others, we could mention the field of diodes and transistors. These important advances mostly touched on orderly materials, such as crystals, used in microelectronics. Theories – subsequently confirmed – showed the strong influence of charge gradients stored near interfaces, with outbreaks of local electric fields and the possible diffusion of carriers. Could these achievements – subsequently developed with the great success we now know – have a beneficial influence on knowledge about mineral insulating materials? And are these theories applicable to organic insulators?

These questions naturally cropped up because, at the same time that microelectronics emerged, there were also notable developments in the world of polymers, first in their mechanical properties (as light and cheap materials), then their electrical properties (as excellent insulators: the first polyethylene-insulated cables were put into service in 1962).

Also, during the 1960s and 1970s, important dielectric studies concerning mineral and organic insulators were undertaken, supported by knowledge of solid-state physics which had been so successful in electronics. Among the main works published on this subject, we can mention: Rose [ROS 63], Coelho [COE 66], Gutmann [GUT 67], Lampert and Mark [LAM 70], Mott [MOT 71], Many [MAN 71], O'Dwyer [ODW 73], Lewis [LEW 77]. However, the insulating nature of these materials on the one hand, and their internal defects due to disorder on the other, led researchers to develop new methods or to improve certain existing techniques.

Among the noteworthy “key” innovations or publications were:

- thermally stimulated currents [CRE 70], which revealed the existence of deep traps in these materials;
- the role of these traps in conduction currents [WIN 73];
- electrode charge injections underscoring Schottky or Fowler–Nordheim laws [SIM 71];
- volume-limited currents (Poole–Frenkel effect [CAS 69] [HAL 71]).

However, certain authors revealed “anomalies” or current instabilities leading to oscillations [FAL 72], [TOU 74], [TOU 76]. These phenomena confirm the essential role of the “electrode-material” couple in injection dynamics and in charge flow.

All this work leans on the existence of both a forbidden gap (8 to 12 eV for an insulator) and trap levels due to structural defects in the material, or to impurities. There is a forbidden gap (a.k.a. a band gap) if we can consider that at least 12 atoms are orderly [AMB 62], [POH 67]. “Localized” levels or traps [LEN 66], [LIL 68] appear in the band gap. Their depths with respect to the conduction band may reach several eV, depending on the local disorder in the molecular arrangement. The most frequent mode of transport then seems to be from trap to trap, with or without passage through the conduction band.

Thus, we see in the early 1980s confirmation of all of these complex theories in thick insulators, where the same type of reasoning as for N or P structures can be used locally, with different N and P structures alternating at a nanometric scale. We therefore have a succession of interfaces; now, microelectronics shows that

everything is governed by the interface charges. Facing this evidence, the international community of dielectricians became aware of the necessity to create measurement systems for this local charge called a “space charge”.

We then see the appearance of several methods, using either pressure or temperature as a stimulus to reveal charges. The functioning of each of these techniques is different (see Chapters 11, 12, 13 & 14), but their basic principle is the same: to cause variation of influence charges at the electrodes, which results in a transient current or voltage which can be recorded and analyzed.

The LIPP (*Laser Induced Pressure Pulse*, Chapter 13) technique, developed in Paris by Alquié [ALQ 81], then resumed in Darmstadt by Sessler [SES 81], uses a laser to send a pressure pulse to a sample. This technique was used in Paris by Lewiner and his team [ALQ 81], who applied it to polymer samples of the order of a millimeter of thickness. The thermal pulse technique, developed in Washington by Collins [COL 80], and then by De Reggi [DER 82], uses a laser to create a thermal pulse. The LIMM (*Laser Impulse Modulation Method*), developed by Lang, uses a laser to create a thermal modulation [LAN 86]. The *Thermal Step Method* (TSM), developed in Montpellier by Toureille and his team (Chapter 14) [TOU 87], [TOU 91], uses a constant temperature front, thus allowing an analysis of large thicknesses such as those of insulated power cables.

The 1980s then saw the presence of two distinct schools:

- that using thermal stimuli: these are high sensitivity methods, of slow dynamics (the temperature moves at the speed of several hundred microns per second in the insulators studied);
- that using pressure: these are fast methods, requiring sophisticated equipment (the speed of sound is of several kilometres per second in the studied insulators).

In the 1990s, numerous results given by all these methods gave rise to new developments or extensions. With regard to the thermal methods, Franceschi and his team [FRA 97], [FRA 00] developed the FLIMM (*Focused Laser Intensity Modulation Method*, see Chapter 12) by focusing a laser beam in order to obtain space charge distributions in three dimensions on samples of several dozen microns. Reboul [REB 00] uses the ATWM (alternative thermal wave method) on thicknesses of 100 microns. The TSM is industrially applied on cables [CHE 92], [ABO 97], [AGN 00], [NOT 01].

As for methods using pressure, there has been a Japanese innovation; Maeno [MAE 88] developed the PEA (Pulsed Electro Acoustic) method (Chapter 11). This technique uses the pressure pulse created by an electric pulse applied to a sample.

A different method to the previous ones (collectively known as stimulus methods) has also made its appearance: it is a mirror method, which uses a scanning electron microscope (SEM). It has been developed at the CEA by Le Gressus, who uses a reflected beam to measure charges implanted in an insulator [LEG 95], [VAL 95].

Thus, the multiplication of space charge measurement methods has created an increasing number of publications, congresses and international networks on the subject, especially from the 1990s onwards. First, this increase gave rise to more tools and a better scientific knowledge of insulators, and, second, in the industrial sector, it led to the development of smaller and smaller electric and electronic components with improved performances.

If we now consider the scientific results obtained over the last 20 years, we must take for granted (by trying to put the qualitative aspects into focus first) that:

- there are space charges in all insulators, even after manufacture [TOU 98];
- we can inject and accumulate space charges in all insulators, even under alternative current (AC) [AGN 99], [NOT 99];
- these space charges evolve with stress (mechanical, thermal, electrical, chemical, radiative, temporal, etc.) [MAL 97];
- these space charges evolve with ageing [BER 99];
- these space charges are related to lifetime [MAZ 01];
- these space charges reflect the state of the microstructure being considered [RAM 01], and therefore represent one of the essential parameters of the material.

### 6.3. Space charge measurement methods in solid insulators

After presenting the history of space charges, we will now describe the methods used for their measurement in a more detailed way.

The techniques developed can be classified as “destructive” and “non-destructive” methods. “Destructive” techniques are so called because they “destruct” the charge state of the sample by emptying the charges stored. These methods do not provide information on the localization of carriers, but allow the total charge and the energies required to evacuate the different types of carriers (“activation energies”) to be estimated. Since the sample is discharged at the end of the measurement, destructive techniques do not permit the evolution of charges to be studied. The “non destructives” methods do not remove the accumulated charges and allow their

distribution throughout the material to be determined. These techniques have the advantage of allowing the charge evolution to be monitored over time.

Finally, the coupling of “destructive” and “non-destructive” methods allows the complete characterization of the processes of charge trapping and/or polarization.

### 6.3.1. Destructive methods

#### 6.3.1.1. The thermally stimulated current method

The most widespread method is that of thermally stimulated depolarization currents, or *thermostimulated currents (TSC)*. Developed in the 1970s [BUC 64], [CRE 70], [VAN 75], [VAN 79], it is one of the oldest methods and has numerous variants. The principle of TSC consists of warming up a short-circuited sample by applying a temperature ramp to it and, at the same time, measuring the “thermostimulation” current, which appears in the external circuit because of the liberation of charges and/or the disorientation of dipoles (see Figure 6.1).

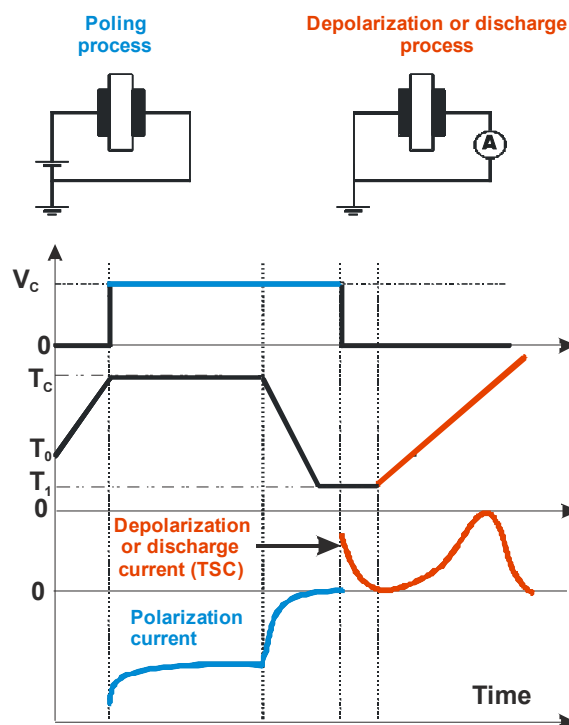


Figure 6.1. The thermally stimulated currents (TSC) method

Thus, a sample polarized under a voltage  $V_C$  at a temperature  $T_C$  is at first cooled down to  $T_I$  (often under voltage) in order to “freeze” the charges. The sample is then short circuited and its temperature, considered constant over the whole sample, is linearly increased (by a few degrees/minutes), whilst the current which appears between the electrodes is measured at the same time. This current gives rise to a spectrum composed of one or several peaks, representative of the different discharge or depolarization phenomena.

The spectra obtained in practice are often complex and poorly separated. The analysis of thermostimulated currents allows, especially in the case of dipolar mechanisms, relaxation times, total equivalent charge and activation energies to be measured. On the other hand, this technique is not so suitable for the measurement of other types of charges, notably because of the possibility that the carriers will recombine in the material without giving rise to a current measurable in the external circuit.

#### 6.3.1.2. *The mirror method*

This method [LEG 95], [VAL 95] permits the analysis of the amount of charges likely to be stored in an insulator, as well as their evacuation dynamics (“detrapping”) following the application of different stresses (thermal, electrical, mechanical, etc.) Its principle lies in the implantation of charges in the material with the aid of a high energy electron beam. The dynamics of the implanted charge is then studied with the help of a lower energy electron beam.

The mirror method is not really classifiable in one of the two large categories mentioned above. It has been placed in the destructive techniques section since the implantation of charges modifies the material.

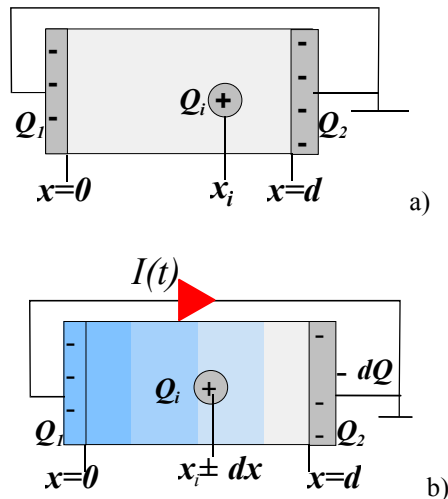
#### 6.3.2. *Non-destructive methods*

As previously described, these methods have been developed since the 1980s and are based on the application of a mechanical or thermal stimulus to the material, non-homogeneous in time and in space. The stimulus locally perturbs the electrostatic equilibrium of the sample by giving rise to an electrical response measured in an external circuit (voltage or current). The analysis of this signal permits the electric field distribution and space charge density in the insulator to be calculated.

Most of these techniques lie in the relative displacement of charges with respect to the electrodes, and in the local variation of the permittivity with temperature. Indeed, in the absence of the stimulus, in both electrodes in the sample containing a space charge  $Q_s$ , influence charges (called  $Q_1$  and  $Q_2$  in Figure 6.2) appear with



respect to the total influence principle. During the application of the stimulus, these charges are modified by  $dQ$ , resulting (if the sample is in short circuit, as in Figure 6.2) in a transient current in the external circuit, or in a transient modification of the voltage across the sample if the latter is in open circuit.



**Figure 6.2.** Principle of a stimulus method (example for a sample in short circuit): a) sample in the absence of stimulus; b) sample during the application of the stimulus

Most of the techniques used today call on thermal or mechanical stimuli. We also need to mention here the *Kerr effect technique* [COE 93], based on the birefringence induced in the insulators by an electric field, but which is therefore exclusively applicable to transparent insulators, and particularly those presenting a high electro-optical birefringence. The sensitivities of the techniques used today range between  $0.001 \text{ C/m}^3$  and  $1 \text{ C/m}^3$ , and their resolutions between 100 nm and 25 mm, according to the characteristics of each technique, and on the nature and thickness of the samples studied.

#### 6.3.2.1. Thermal methods

In this case, the sample is subjected to an abrupt temperature rise on one of its sides, which leads to a non-uniform dilation of the material, and to a local variation of its permittivity. Note that the variation factors are very weak (of the order of  $10^{-4}$  to  $10^{-6}$ ), which does not prevent these techniques displaying better sensitivities ( $1 \text{ mC/m}^3$ ) and resolutions ( $< 0.1 \text{ }\mu\text{m}$ ), the reason being a much better thermal transfer efficiency than those of energy transfers of mechanical origins. They are generally “slow” methods, the duration of the measured signals being longer than

one second. As a result, the charge monitoring for durations less than a minute is limited; on the other hand, they provide significant signal-to-noise ratios and an excellent reproducibility.

In general, a “thermodilation” signal is measured, whose expression is in the form (for a measurement in short circuit):

$$I(t) = \pm \frac{S}{d} \int_0^d E(x) \alpha(x,t) \frac{\partial \Delta T(x,t)}{\partial t} dx \quad [6.1]$$

with  $S$  surface of the sample,  $E(x)$  distribution of the electric field throughout the sample,  $\alpha(x,t)$  function containing the variation laws and the permittivity thickness of the material with the temperature, and  $\partial \Delta T(x,t)/\partial t$  variation law of the temperature over time on the  $x$ -axis in the material. Knowledge of the measured current, temperature distribution and thermal parameters of the material allow  $E(x)$  to be calculated and, subsequently, the charge distribution. The resolution of the integral equation can be difficult, but mathematical techniques are now much improved and today allow remarkable results to be obtained.

The difference between the thermal methods is most notable at the level of the means of achieving localized heating, which has a direct influence on the amplitude of thermal perturbation (and so on the measurable thickness) and on the equipment to bring into play. According to the technique used to produce a “heat wave”, we distinguish:

- the *Thermal Pulse Method* [COL 80] [DER 82]), which leans on the application of a heat pulse (realized by a laser pulse) on a metallic sample whose electrodes are in open circuit. The pulse propagates by moving the charges by local dilation of the material. This method ensures an excellent resolution ( $< 1 \mu\text{m}$ ) and is notably suitable for the study of thin samples ( $< 100 \mu\text{m}$ ), because of heat wave damping in thicker materials;

- the *Laser Intensity Modulation Method* [LAN 86] consists of heating a side of the sample with a laser beam, modulated in intensity, at a variable frequency, and in measuring the resulting pyroelectric current. As for the thermal impulse method, the LIMM suits thin samples ( $< 100 \mu\text{m}$ ), its resolution being less than  $10 \mu\text{m}$ ;

- the *FLIMM* (Focused Laser Intensity Modulation Method) and the *FLAMM* (Focused Laser Acoustic Modulation Method [FRA 97], [FRA 00]) use the same principle as the LIMM, but the laser beam is focused and its displacement allows a 3D mapping of the sample. The resolutions of these techniques are of the same order as the LIMM. The insulating thicknesses that can be characterized by FLIMM and FLAMM are also in the same gap as the LIMM;

– the *Thermal Step Method* [TOU 91] [NOT 01] consists of the measurement of the thermomodulation current of the sample after application of a *temperature step* on one of its sides. Because of the significant quantity of heat transferred to the material, this latter method is as suitable for thin as for thick samples (between 100 nm and 25 mm), whether the structures are plane or cylindrical. As for other techniques, this method's resolution depends on the thickness of the sample and can get down, in the case of very thin samples such as those used in microelectronics, to 100 nm, or even less. A version of this technique (called the alternative thermal wave method (ATWM [REB 00]) was recently developed, and uses a sinusoidal thermal perturbation to analyze samples whose thickness is of the order of 100  $\mu\text{m}$ .

### 6.3.2.2. Pressure methods

These techniques use an elastic wave to quantify the charges trapped in materials. Because of the high speed of sound in insulators, these are “fast” methods: the duration of signals does not exceed a few  $\mu\text{s}$ . This makes them particularly appealing for the monitoring of charge dynamics, notably under sinusoidal electrical stress, and also under thermal stress. On the other hand, the energetic efficiency of the stimulus being more reduced than for thermal methods, the signal-to-noise ratio is generally weak. Consequently, achieving reproducible results fairly often requires the need for an average of several dozen (even hundreds) of successive measurements to be made. The spatial resolution of these techniques can get down to 1  $\mu\text{m}$ , their sensitivity being in the order of 10  $\text{mC}/\text{m}^3$ .

Today, we mainly use:

– methods calling on a *pressure wave* [ALQ 81], [SES 81], whose principle is the compression of the material by a transient wave with a stiff front. Different means are used to generate this pressure wave (shockwave tube, piezoelectric transducer, laser pulse), and hence the existence of several versions. The most developed method is the *LIPP (Laser Induced Pressure Pulse* [SES 81]), which uses a laser impulse of a duration of less than ten nanoseconds. The equations relating the signal measured to the internal electric field in the sample are analogous to those of thermal methods. For example, for a sample in short circuit, the measured current is expressed as:

$$I(t) = \lambda CG(\epsilon) \int_0^d E(x) \frac{\partial P(x,t)}{\partial t} dx \quad [6.2]$$

where  $d$  is the thickness of the sample,  $\chi$  the compression factor of the material,  $C$  the capacitance of the sample before compression,  $G(\epsilon)$  the coefficient related to the permittivity variation of the material with pressure,  $E(x)$  the distribution of the electric field throughout the sample, and  $\partial P(x,t)/\partial t$  the variation law of the pressure with time on the  $x$ -axis in the material. Knowledge of the measured current,

pressure distribution and parameters of the material allows the calculation of  $E(x)$ , and, subsequently, the charge distribution. If the attenuation of the pressure wave is negligible (in the case of thin samples), we can obtain a reading of the charge distribution without mathematical processing, which constitutes an important advantage. However, consideration of issues related to pressure wave reflection at the interfaces often requires a complex analysis of signals and the need to resort to deconvolution;

– the *Pulsed Electroacoustic Method* [MAE 88] originates in a slightly different principle, and consists of placing a piezoelectric transducer on one side of the sample and exciting the test-tube by a brief high voltage impulse. The Coulomb force produced by the charges under the effect of this stimulus generates a pressure wave, collected by the transducer after a time  $t_A$ . This time allows the charges to be located. The PEA method is today very widely adopted around the world and seems well suited for samples of thicknesses greater than 100  $\mu\text{m}$ .

It is quite difficult to compare the different methods. However, thermal methods could be compared, and the mirror method could be positively correlated to the TSM. Comparisons have also been made between the LIPP and the PEA method. On the other hand, genuine comparisons between thermal and pressure techniques cannot be made for now, mostly because the two classes of methods have different sensitivities and because their dynamics are different.

#### 6.4. Trends and perspectives

It seems undeniable that these methods will play an important role in scientific as well as industrial aspects in the future. The “low energy” thermal methods (LIMM, FLIMM, ATWM) can claim thin 3D type measurements on thicknesses from 10 to 100 microns, with a “material” or component orientation. The “high voltage” TSM, with industrial applications, contributes to the choice of material for polymer-insulated cables for direct current power transport, and characterizes the cables aged on site under alternative stress (50 Hz). Further, the TSM recently showed its possibilities for new characterization methods for micro or nanoelectronics. Considering their fast response time, basic pressure methods can directly be used under alternative current.

In conclusion, because the space charge reflects the microstructural state of a material, it has been important, for 30 years now, to develop non-destructive methods for measurement of this fundamental parameter which represents the history of the material or of the component. These methods – in the form of two distinct approaches – have each progressed very far in their respective fields, to

arrive today at a protocol [TAK 06] which can act as a base for more and more numerous scientific and industrial studies.

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