Toward non destructive high resolution thermal methods for electric charge measurements in solid dielectrics and components

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Abstract— During many years, non destructive direct space charge distribution measurement techniques have been successfully used to study electrical phenomena in insulating materials and components. This paper deals with recent applications of these techniques in the field of microelectronic structures and thin layers and on the information they are able to provide when used instead or in complement to existing indirect or destructive methods. The issue of developing new experimental set-ups allowing high spatial resolution measurements is addressed in particular.

I. INTRODUCTION

The development of charge-based or charge-controlled devices needs a perfect knowledge of the amount and of the distribution of electric charge in the different material layers and at their interfaces. This need is particularly acute when high electric fields establish in the structures during operation. This is mainly the case for the electronic components and the next generations of components and devices, such as MEMS, devices for controlling micro-fluids in lab-on-a-chip, high-voltage high-current integrated devices, micro-electro-thermal heat sinks for micro components etc.

On the other hand, electric charge accumulation in dielectrics (or space charge, as it is commonly known) can have dramatic effects when uncontrolled. Thus, the electric field induced by the development of space charges is superimposed to the electric field applied to the material in its usual operating conditions. The resulting value of the field can therefore approach and even exceed the breakdown threshold, leading to an alteration or to the
destruction of the material and, as a consequence, to a possible failure of the system in which it is included [1]. The risk is increased when high electric fields are applied to the component, at the interface between different materials, or in the presence of external factors able to induce significant amounts of charges in the insulating layers (e.g. radiations). Even without reaching breakdown, the charges accumulated in the insulating layers of a device may provoke malfunction affecting system reliability. This is a critical problem in contexts with considerable economical dimensions and where human security considerations are essential, such as in electric power transport, airborne and space applications.

Determining the space charge distribution is therefore an important matter, either for designing efficient sensors or for optimizing dielectric materials and structures. Non destructive measuring techniques are preferable because the evolution of the charge distribution in the material under real operating conditions can be followed. This has led to the establishment, over the last decades, of several non destructive charge measurement techniques, commonly known as “stimuli methods” [2-16]. These methods are based on the application of a mechanical or thermal stimulus which slightly perturbs the electrostatic equilibrium of the measured sample, giving birth to an electrical transient response which is recorded and analyzed, thus allowing to determine the electric field and charge distributions across the sample.

Historically, the stimuli methods have been used for a wide variety of electrical engineering applications involving thick insulating layers (> 100 µm). However, increasing interest is now given to the application of stimuli methods to thin layers (< 1 µm) and electronic structures [16-17], as the classical methods used in micro-electronics are either destructive or of insufficient resolution. Thus, for dielectric layers thinner than 100 nm, the etch-off technique [18-19] allows obtaining significant spatial resolution, but that technique is destructive. The capacitance-voltage technique [18] is also widely used in micro-electronics; this technique is non-destructive, but only the centroid of the charges close to the semiconductor interface can be located. Moreover, results are highly dependent on physical models.

As they are direct, non destructive and of potential high resolution, the stimuli methods can bring important information when used instead or in complement to the above quoted techniques. Herein after, the fallouts of these methods in the domain of thin layers and electronic structures are addressed. Results obtained in the concerned field are presented, then paths for increasing spatial resolution are proposed and discussed.

II. PRINCIPLE OF THE THERMAL METHODS

There are mainly three families of non-destructive space charge distribution measurement methods: thermal methods, pressure methods and electro-acoustic methods [2]. All of them are based on the non homogeneous perturbation of the electrostatic state of the structure under test. For that purpose, adjacent electrodes are necessary. The tested material (or insulating structure) with its adjacent electrodes is referred hereafter to as the sample.

Thermal methods use thermal diffusion as the perturbation [3]. The sample is subjected to a low variation of temperature on one of its faces. The diffusion of heat through the insulator expands the material in a non-uniform manner, which slightly displaces the charges. Additionally, the dielectric constant of the material varies locally. This induces an
electrical response, either a current between the electrodes in short-circuit conditions or a voltage in open-circuit conditions. That is illustrated in Fig. 1.

![Principle of the thermal methods](image)

Fig. 1. Principle of the thermal methods. (a) Typical set-up: heat diffuses in the sample, displaces the charges which induces in turn an electrical current. (b) Typical signal: with a heat pulse, the amplitude at the beginning of the signal is directly proportional to the interface electric field.

Different techniques have been developed in order to generate heat diffusion of various time dependences. When pulsed lasers are used, the method is referred to as the thermal pulse technique [4-6]. When modulated lasers are used, the method is referred to as the laser-intensity-modulation-method (LIMM) [7-8]. When temperature steps are used, the method is referred to as the TSM thermal step method (TSM) [9-11]. Whatever the forms of the thermal stimulus, the expression of the measured signal is of the form [12]:

\[
    i(t) = -C \int_{0}^{\infty} \alpha(x) E(x) \frac{\partial \Delta T(x,t)}{\partial t} \, dx
\]  

where \( C \) is the fraction of the sample capacitance corresponding to the heated or cooled area, \( E \) is the electric field distribution across the sample, \( \alpha \) is the equivalent linear expansion coefficient which takes into account pyroelectricity, and \( \Delta T \) is the imposed temperature variation across the sample at every instant. Though the limits of the integral have been extended towards infinity, the electric field has a non-zero value only inside the sample. Coefficient \( \alpha \) can be considered as constant in uniform materials and for reasonable temperature variations and is typically of the order of \( 10^{-4} \) K\(^{-1}\) to \( 10^{-3} \) K\(^{-1}\). As the charge density is related to the electric field via the Poisson equation, obtaining the field repartition from equation (1) allows to determine the charge density distribution.

Thermal methods provide significant sensitivity and good spatial resolution close to interfaces without requiring wide bandwidth measuring systems. In contrast, far from the interfaces the spatial resolution decreases owing to the physics of thermal diffusion. Though the thermal methods have been firstly used to simply characterize the polarization homogeneity of pyroelectric films [3], many studies have been devoted to estimate the field and charge distributions from the signal [13-15, 20]. This is indeed a delicate point to address.
III. APPLICATION TO ELECTRONIC COMPONENTS AND THIN LAYERS

It comes from equation (1) that the homogeneous (single-layer) sample from Fig. 1 can be replaced by any structure, as the measured signal is proportional to the overall capacitance of the sample and to the integral of the field multiplied by the $\alpha$ coefficient, which can vary with the depth coordinate with respect to the nature of the different layers composing the structure. Thus, the technique is fully applicable to electronic structures as metal-insulator-semiconductor (MIS) capacitors. If we take the example of the metal-oxide-semiconductor (MOS) structure shown in Fig. 2, the measured signal $I(t)$ will carry information about the position and the amount of charges contained in the insulating oxide and in the semiconductor.

![Diagram of a MOS structure](image)

Fig. 2. Application of the thermal method to a metal-oxide-semiconductor structure: the measured signal is the derivative of the influence charge with time, providing information on the oxide and semiconductor charge.

Typical dimensions of MOS structures are of the order of nanometers to microns for the oxide and of several hundreds of microns for the doped semiconducting layer (substrate). In order to study phenomena related to reliability and ageing of such components and for designing new structures, it is important to be able to locate as precisely as possible the electric charge within the oxide and at the oxide/semiconductor interface. Indeed, the charges accumulated in the insulating layer and at the interface may provoke malfunction of the component even if the insulating layer does not break down. Determining the doping profile in the semiconductor is also of interest for improving the manufacturing process.

A. Low resolution measurements

Interesting information on the charge within such a structure can be obtained relatively easily by using a thermal step stimulus [16-17]. Fig. 3 shows such a setup where the thermal step is applied with the aid of a liquid circulating in a radiator in contact with the measured sample. The signals measured for different voltages applied to the structure are also shown: it can be seen that a characteristic signal is obtained for a given voltage, as the semiconductor is driven into accumulation, depletion or inversion regime.

By using equation (1) and by assuming that the time thermal constant of the radia-
tor/sample interface (of equivalent thickness $x_0$ [11]) is much higher than that of the sample itself, it can be shown that the amplitude of the acquired signal reads [16]:

$$I(V_g)_{Max} = C \left[ \alpha_\text{ox} (V_g - V_S) + \alpha_\text{Si} V_S \right] \left[ \frac{\partial \Delta T(x_0,t)}{\partial t} \right]_{Max}$$

(3)

where $C$ is the sample capacitance, $\alpha_\text{ox}$ and $\alpha_\text{Si}$ are the equivalent linear expansion coefficient which take into account pyroelectricity for the oxide and the substrate, $V_g$ is the voltage applied to the structure (gate voltage), $V_S$ is the substrate voltage and $\left[ \frac{\partial \Delta T(x_0,t)}{\partial t} \right]_{Max}$ is the amplitude of the thermal wave.

Fig. 3. Thermal step method applied to a metal-oxide-semiconductor structure and transient current signals measured for different voltages $V_g$ applied to the MOS sample. From reference [16].

The $I(V_g)_{Max}$ data can be plotted to obtain a characteristics of the structure (Fig. 4), then by combining it with low frequency capacitance-voltage measurements one can obtain the total charge of structure and the charge characteristics to the oxide and the interface (Fig. 5).
Fig. 4. Maximum of the measured thermal step currents $I_{\text{Max}}$ plotted versus the gate voltage $V_g$ [16].

Fig. 5. Total charge of the structure ($Q_0$) and oxide + interface charge ($Q_0-Q_s$) versus substrate potential [16].

Fig. 6. Distorted signal measured in inversion ($V_g = -5$ V) on a MOS capacitance of the type presented in Fig. 3 after submission to a -50 V dc stress during 41 hours at 25°C. Comparison to the signal measured before stress.

It is also worth to mention that the sensitivity of the thermal methods is at least one order of magnitude above that of the capacitance-voltage method, thus allowing putting into evidence changes into the electrical state of the insulator that cannot be observed other-
wise [17, 21].

The above model assumes that the charge contained in the structure is distributed in two layers, one having the thickness of the oxide and the other the thickness of the space charge area within the substrate. An estimation of the charges in the two regions is therefore at reach. However, when the electric field is disturbed significantly, the incertitude of the result can be important, as the “charged layers” hypothesis is not fulfilled. Such situations result in significant deformation of the signals (Fig. 6), more information being available directly from the acquired data. Thus, provided that the side effects are negligible, it is possible in this case to draw information about the localization of the charge within the oxide and to put into evidence the charges placed close to the gate [17, 21], which the capacitance-voltage method does not allow to follow.

To reduce side effects, the heat flux must be concentrated as much as possible in the gate area. One way to achieve this is to provoke the thermal perturbation by a non contact method, for instance by using a laser pulse. This also presents the advantage of diminishing the thermal inertia of the system, thus allowing tending toward much higher resolutions. An example of such a measurement made on a MIS structure composed of 300 nm-thick Si3N4 coated on a $10^{18}$ cm$^{-3}$ p-doped Si substrate is shown in Fig. 7. The thermal stimulus was generated with 0.4 mJ - 50 ps laser pulses. In this case, the acquired signal contains more information on the electric field distribution across the oxide and the substrate, as the sampling period is much closer to the transit times of the thermal wave across the two layers: provided that the temperature diffusion through the sample is accurately known, such measurement can allow to closely approach the electric field distribution in the sample.

![Diagram](image)

**Fig. 7.** Thermal pulse method applied to a metal-insulator-semiconductor structure and transient voltage signals measured for different voltages applied to the sample.

B. Toward high resolution systems

From the results presented in the previous section, it can be concluded that measurement methods usually implemented for thicker materials can give interesting new information in the case of thin materials. However, only the overall charge quantity can be ac-
cessed with such methods, and the detail of the distribution remains elusive. Obtaining better spatial resolutions requires a significant increase of the bandwidth of both the perturbation and the detection set-up. Indeed, spatial resolution is closely related to bandwidth since time and space are connected through a diffusion process [22].

Though noise level should be taken into account to determine the reachable spatial resolution, direct resolution is roughly given by \((Dt)^{1/2}\), where \(D\) is the thermal diffusivity of the sample and \(t\) is the time. \((Dt)^{1/2}\) represents the position for which the temperature variation is half the one at the interface. \(D\) is of the order of \(10^{-7}\) m\(^2\)/s in polymeric materials and of the order of \(10^{-6}\) m\(^2\)/s in silicon dioxide. Therefore, a resolution below 100 nm in organic layers and components requires a bandwidth higher than 10 MHz, whilst the bandwidth needed for oxides is of the order of 1 to 10 GHz. In the first case, nanosecond thermal pulses are required, while femtosecond pulses are needed in the second case. Conventional heaters or pulse generators are no longer adapted and the bandwidth of the usual measuring equipments is not sufficient. New kinds of implementation are required.

Tests where thermal perturbation is produced by a femtosecond laser pulse have been carried out [23]. When charges are perturbed by heat or by the elastic waves, they emit an electromagnetic wave which can be measured by electro-optic sampling (Fig. 8). Though spatial resolution better than 50 nm in silicon dioxide could be expected, which is equivalent to 22 nm in polymeric materials, the signal to noise ratio is very poor and obtaining convincing results is still a long way to go with all optical instrumentation.

![Principle of thermal methods with femtosecond laser pulses. A pump beam perturbs the sample. The induced charge displacement generates an electromagnetic wave which is measured by electro-optic (EO) sampling. A delay stage provides the time sweep.](image)

The technology for obtaining spatial resolution below 100 nm is therefore at reach, although requiring dedicated equipments such as wide bandwidth acousto-optic modulators for heat excitation. However, even if most of the needed equipments are now available, reaching the proposed goal remains a difficult challenge, as significant experimental and theoretical problems must be solved.

First of all, the perturbation needs to be correctly applied and the signal must be detected above the noise. Measuring the signal is however not sufficient to accurately estimate the charge distribution with the thermal method. Indeed, results drastically depend on the boundary conditions applied in the deconvolution calculations.
The simultaneous measurement of the signal and of the interface temperature is of great help, as it allows accessing directly to the stimulus and thus increases the calculation precision for temperature distribution across the material. Bolometric measurements from the heated electrode have already been proposed but attention must be paid to terminals for limiting additional load capacitance and noise increase (Fig. 9).

Amplitude is not really useful since calibration procedures are used to scale signal amplitude [11], only time evolution of the temperature is required in order to perform the mathematical deconvolution of the equation (1), which is a Fredholm equation of the first kind. An important problem which must be addressed is the improvement of the temperature matrix conditioning, either by mathematical techniques or by the adaptation of the thermal stimulus (boundary conditions), allowing to obtain a less ill-posed inverse problem. A significant work concerning thermal and signal analysis must therefore be performed for improving signal deconvolution.

IV. CONCLUSION

Thermal stimuli non destructive methods are able to provide important information in the field of localization and quantification of electric charge in thin insulating layers and microelectronic components. Spatial resolutions of the order of 20 to 50 nm are today at reach with the aid of developments allowing simultaneous measurement of the signal and of the interface temperature, associated to significant work in the domain of signal processing is carried out.

REFERENCES