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HOW TO FAIL THE MEASURE OF DC CONDUCTIVITY OR RESISTIVITY OF POLYMERIC MATERIALS

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Abstract

The advent of HVDC and MVDC applications arrived with a challenge for the dielectric's community: the determination of the conductivity of insulating materials under DC stresses, particular challenging for non-linear materials. Usually obtained from current-voltage (I-V) tests, this work shares a series of faults (and learnings) resulted from the struggle to tailor a reproducible and repeatable protocol for electrical current measurements of polymeric insulating samples. I-V measures have been conducted for epoxy and silicone samples under various electric fields, temperatures and water contents. Results shows that, if the objective is to fail in I-V measurement, it is enough to: Underestimate the structure/microstructure relationship of departure; underestimate key physical parameters during the test; and underestimate the structure/microstructure changes during the test. For each case, illustrations and quantification have being given. Because the electrical current seems to be highly biased by the system characteristics, and since the conductivity calculated from the Ohm's law has no physical meaning in non-Ohmic regions, the intrinsic conductivity of polymeric insulating is hardly attainable.

1 Why this Publication?

Measuring the electrical current and determine the conductivity of a dielectric when a voltage is applied sounds easy. But it can be challenging, because as any material property, the electrical current through a dielectric can be very sensitive to many aspects coming from the processing, structure, and microstructure. These aspects are hugely impacted by sample preparation and operational conditions taking place during the test. So, in an electrical test like this one, which parameters have to be controlled and which does not need strict control? How much the deviation is and how much is acceptable?

This article is the fruit of all the struggles, mistakes, attempts, and learnings lead by the first author to tailor a (supposed) trustworthy electrical test protocol that would give reproducible and repeatable results. It does not pretend to dictate how to measure conductivity, neither to negatively criticise third part results.

It is just an unusual approach, since the scientific community is used to share good, clean and credible results in the first shot. The risk is that the same faults, as improperly highlighted and discussed, will be repeated more or less systematically. During the journey, the reader is free to judge if some attempts were too rigorous, or too tolerant.

The volume conductivity σ (or its reciprocal, resistivity ρ) has been the main important property selected to evaluate the performance of dielectric systems under DC stresses, having the same importance as relative permittivity ϵ and loss factor $\tan\delta$ for AC applications. Even if the main technique of

conductivity characterization is such an old one as the current measure, obtaining experimentally the conductivity of solid dielectrics can be controversial:

- Standards such as ASTM D257-14 or IEC 62631 suggests a polarization time equals to 60 seconds. For most of polymeric systems, a pure resistive behaviour is not attained within this time-length; results will only reflect a polarization index.

- Most works focus on the instrumental point of view, which depends on the instrumental parameters, but the result will also reflect the sample. More precisely, not just the sample nature, but the material's (micro)structure, which comes from the process and conditioning. Because the control of these parameters is not always rigorous, results can diverge worldwide.

Finally, the focus of this article is the sample. What is it? Do we know what we are measuring? In this article, a failure means we have lost the control about what we are measuring because of changes in the material, the test protocol or set-up, or details of data treatment. As the results risk to be non-representative, the authors felt themselves less confident to use them to understand the behaviour of the sample, as well to extrapolate them for real products.

2 Getting Started

Two test cells have been used: the Keithley 8009 Resistivity Test Fixture (Figure 1-a), and a customized metallic cell that can be closed to isolate the environment around the electrodes (Figure 1-b and c). The electrode systems are the same: 10 Ω

conductive silicone, having 54 mm of diameter on the HV side, and 50 mm in the measure side. The Keithley 6517B Electrometer/High Resistance Meter having a voltage source integrated measured the current and also applied the voltage for voltage levels under 1 kV. Above this value (and systematically later on, after the arrangement of a test protocol) the voltage source was replaced by the PS365 SRS High Voltage Power Supply (up to 10kV). Additional set-up will be presented along the text.

This article presents results of two different materials: Two kinds of silicone rubbers (one composite with silica, and another pure without filler, both being a vinyl-methyl siloxane), and an epoxy resin (DGEBA) filled with silica with a T_g around 110°C. Originally provided in plates of 1 mm of thickness, samples are shaped in small discs of 70 mm by laser cut (SiR) or water jet (epoxy).

For each voltage level, the method by default consisted of 1 hour of polarization and 1 hour of depolarization (so called 1h/1h method, or PDC cycles of 1 hour). Details about the test and its particularities can be found in [1]–[3].

3 The Roadmap of Failure

3.1 Not caring about the start: the sample preparation paradigm.

The electrical test will measure something that outcomes from processing and storage steps, which can or cannot be controlled by the experimenter. The dispersion and distribution of fillers throughout the matrix, the adhesion between filler and polymer, contaminations, the reticulation degree, thermal and mechanical stresses, differential crystallization and orientational effects are examples of aspects inherited from fabrication that are able to affect any property of a polymer. Often, the experimenter does not have much flexibility about aspects coming from fabrication, but the sample can be treated to limit some of them. In this section, the impact of some aspects of (failed) sample preparation will be discussed.

3.1.1 Ignoring the way the sample has been fabricated: Two 1-mm thick plates of pure silicone rubbers, i.e. without any filler, produced from the same raw material, have been fabricated by gravitational molding and curing in oven at 160°C, but with different curing times: sample A has been cured for 1 hour with no post-curing; and sample B, 6 hours with 8 hours of post-curing at 80°C.

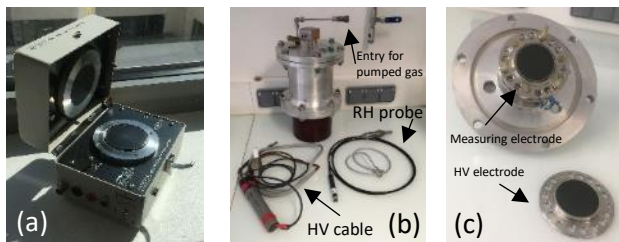


Figure 1. Picture of a Keithley 8009 Resistivity Test Fixture (a), of the metallic cell (b), and the electrodes of this cell (c).

These plates have been shaped into disks of 70 mm of diameter. Figure 2-a shows the polarization as a function of time for such samples when exposed to 1 kV during 1 hour of test. It can be noted that the current of sample B at the end of 1 hour differs from the current of the sample A by a factor equals to 2.15. In addition, the higher the voltage level, bigger is this difference: when tested at 10 kV/mm, the current of sample B was 10.57 times higher than sample A. This difference is broadening because, as presented in Figure 2-b, the apparent conductivity of sample A decreases with voltage, almost if the sample A were liquid-like, while sample B has a conductivity that increases with voltage as most of solid dielectrics.

The difference of samples being attributed to the curing time, a first hypothesis concern about different reticulation degrees, with sample B having a higher reticulation degree than sample A. A second hypothesis concern about the proportions of raw materials: as it is a bi-component silicone, if the quantity of each raw material is not well dosed, the final product shall deviate from an elastomer and give a gel-like material. A third hypothesis concern about the demolding: neutral detergent has been applied on the mold, further dried before molding. The presence of detergent in the bulk and the impact of it on surface is unknown. In any event, the exploration of these results is the focus of another work. Here, what has to be pointed out is that the same material can present contrasting results as a consequence of distinct fabrication means. If the fabrication is unknown, the understanding is limited.

3.1.2 Not evaluating the need of pre-discharging: Fabrication and storage can produce electrical charges that will be trapped inside the bulk (also known as stored charges). Once present, they will change the electric field distribution, so as the value of the absorption current [4].

Figure 3 shows the polarization current in function of time of another silicone sample filled with silica (with completed curing process!) that has been exposed to 1 kV/mm for 1 hour. A pre-PDC discharge during 700 seconds decreased the value of current by a factor equals to 1.83. If annealed at 80°C and discharged for 300 seconds, the current decreased by a factor equals to 4.32 compared to a fresh sample, and 2.35 compared to a discharged sample.

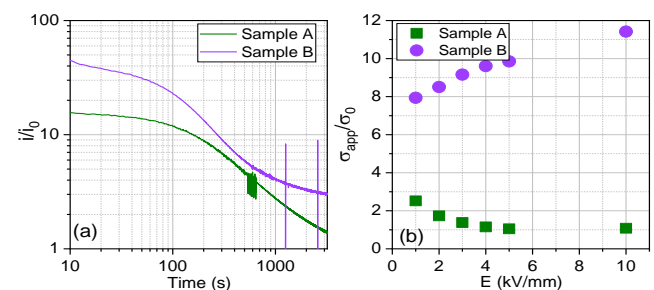


Figure 2. Relative electric current as a function of time for two samples of silicone rubber at 40°C (a), and the apparent conductivity of such samples as a function of field at 40°C (b). Sample A has been cured at 160°C for 1 hour, and sample B for 8 hours with 6 hours of post-curing at 80°C. The values of i_0 and σ_0 are arbitrary.

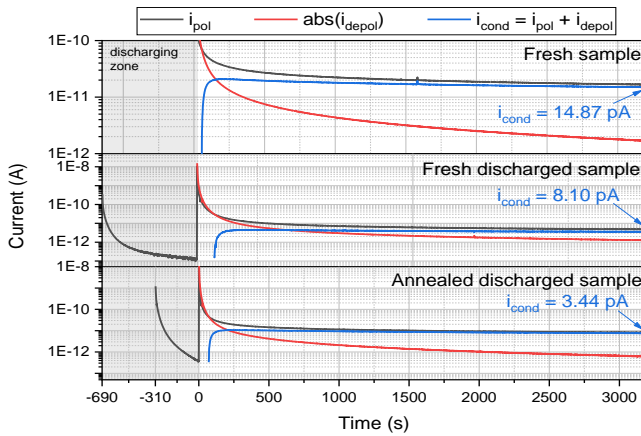


Figure 3. Current as a function of time for a fresh, fresh and discharged, and annealed and discharged sample of silicone rubber filled with silica at 1 kV/mm.

3.1.3 Underestimating the impact of the water content: The authors noticed that the conduction current decreased as the annealing time increased. The samples were drying, and the reduction of current could be related to a reduction of water content [5]–[7]. So, a question raised: what is the impact of water content? Is it relevant? As the water content of a fresh sample is unknown, a protocol for conditioning samples has been designed. It can be seen as an adapted version of the EN ISO 62.

First, samples have dried in an oven under vacuum at 70°C. Supports made of aluminium foil ensured drying on both sides. Samples have been dried as long as they needed, with water lost measured by the weight lost in an analytical balance. The drying procedure is considered finished when the weight remained stable after 3 consecutive measures spaced 1 hour from each other. It is not excluded that volatiles that will be present in real products will be extracted from the sample. The authors considered that it was the cost of having similar samples. Silicone rubbers filled with silica presented stable weights after 2 days of drying, epoxy samples had to last 14 days inside the oven. The total weight lost will depend on the previous water content, which depends on fabrication and the environment of pre-conditioning. In this case, it has been observed that silicone samples lost $0.85 \pm 0.03\%$ of weight. In the particular case of epoxy microcomposite, it has been reported that most of the water shall be absorbed by the matrix, and just a minor fraction should fill the interface with the particles. Because of it, the weight gain has been reported to the matrix, and the total weight lost has been $0.47 \pm 0.01\%$.

In the second step, samples have been humidified in the controlled environment of climatic chambers formerly configured at the same temperature and relative humidity (RH) of the following electrical test. The kinetics of water absorption will be subject of a future work. Here, it is important to mention that two main RH values have been tested (50%, and 90%) and four temperatures (20, 40, 60, and 80°C). The silicone filled with silica took 3 hours to have the weight stabilized on both RHs, epoxy samples took 5 days, and the two pure silicones presented in Figure 2 have not presented

neither a weight lost after 3 days in the oven, neither any gain in weight after 1 week of humidification at 90% RH. At the highest RH of the study (90%), the weight of the silicone composite increased 1.2%, and epoxy (reported to the matrix) 1.1%.

Once dried and humidified, the electrical test are performed at the same conditions to avoid having a new metastable sample. The metallic cell presented in Figure 1-b and c have been installed inside another climatic chamber, the final set-up looking like the one in Figure 4-c. At voltage levels up to 3 kV, the cell is maintained opened so as the interior of the cell is in equilibrium with the temperature and RH of the climatic chamber, but at higher voltage levels, the pressure had to be increased inside the cell to reduce the risk of discharges in the air, so the cell has been closed and dry air is pumped for these values of voltage level. The authors are aware that it can dry the sample, but the analysis of J(E) with PDC cycles of 1 hour did not suggest any further impact.

The current density at 90% RH for silicone samples is in average 5.3 ± 1 times higher than the dried sample at xx°C. In addition, it looks like the increase in current slows down when 1% of water content is attained. More detail about the water absorption impact of this material can be found in [8]. In the case of the epoxy composite (Figure 5), the samples conditioned at 90% RH presented a current density 765 times higher than the dried samples at voltage levels of 250 V, 9114 times higher at 3 kV, and 7448 times higher at 10 kV. It corresponds to an increase of 3-4 orders of magnitude for the same voltage level at the same temperature. The difference of dried/wet samples can be more or less significant due to the impact of water in the main conduction mechanism. These aspects are explored in [9].

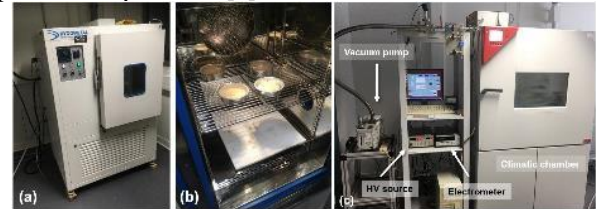


Figure 4. Climatic chamber view from the outside (a) and inside with samples (b). The final test set-up, including another climatic chamber with the metallic test cell inside it (c).

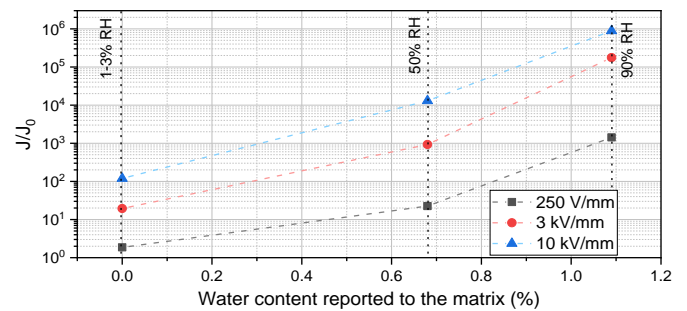


Figure 5. Relative current density as a function of the water content for an epoxy composite filled with silica at 40°C. PDC cycles of 1 hour. Samples at 1 and 3 kV/mm have been tested with an opened cell. The cell was closed and filled with dry air when $V = 5$ kV/mm. J_0 is arbitrary.

3.1.4 Underestimating the impact of electrode material and contacts: Aiming to identify the potential impacts of the electrode material, as well as to improve the contacts between them and the dielectric roughness' surface, samples have been metallized by sputtering with the help of the turbomolecular pumped coater Quorum Q150T ES (Figure 6-a and b). In practice, following the conditioning procedure, samples have been coated by a 200 nm thickness of gold, copper, silver, and platinum on both sides. Because this metallization step is able to dry the sample, they had to be reconditioned for a couple of hours again before being installed in the electrical test cell. The dimensions of the coating being the same as the electrode's dimensions (Figure 6-c), attention had to be paid to the alignment of coating and cell electrodes during the installation of the sample.

For the case of epoxy resin (Figure 7), the impact of the electrode material is like to widen at higher voltage levels for the epoxy resin: at 1 kV/mm, the higher current (conductive silicone tape) differs from the lowest (copper) by a factor equals to 1.34. At 5 kV/mm, this factor increases to 2.00 between silver and gold. In contrast, for the silicone composite, this factor ranges between 11.91 at 1 kV/mm and 3.11 at 5 kV/mm. Here, the dispersion seems to narrow with the increasing voltage. In addition, the current mechanism (defined by the slope in the bi-log representation) is changing from gold (with a slope equals to 3.65) to the other metals (with a slope close to 2).

In real applications, electrode materials are not symmetric as in the laboratory. On the contrary, their shapes and natures change from the inner to the outer surfaces. Aiming to mimetize these configurations, samples of silicone composite have been coated with different materials on each side (gold,

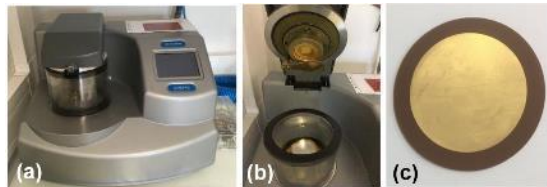


Figure 6. Turbomolecular pumped coater Quorum Q150T ES from outside (a) and inside the chamber (b); sample metallized with gold (c).

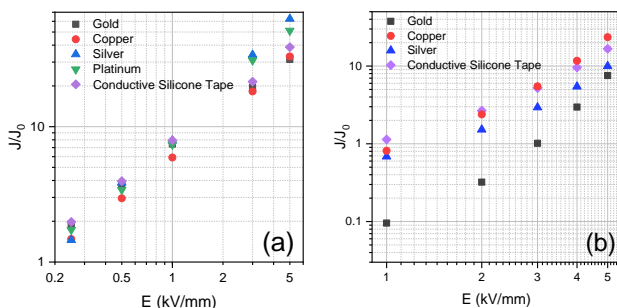


Figure 7. Relative current density as a function of electric field for an epoxy composite (a), and silicone (b) coated by 200 nm of gold, copper, silver, platinum, and by the conductive silicone tape that made the original cell electrodes. Only dried samples at 40°C are presented. J_0 is arbitrary.

copper, and conductive silicone tape), giving what we have called *asymmetric electrodes*. The results and analysis of the samples can be found in [10]. When the data of both publications crossed, it can be noted that the impact of electrode material and configuration increases the current density by a factor up to 52 at 1 kV/mm, and 7.2 at 5 kV/mm.

3.2 Faults during the measure: focus on operational parameters.

3.2.1 Measuring at the room or ambient conditions: we are always tempted to do experiments at the room temperature (i.e. between 20°C and 25°C). The impact of temperature seems to be well acquired by the scientific community, and the exponential relationship written as an Arrhenius-like equation is widely known. For the epoxy resin of this study, the current density obtained at 20°C and 25°C and 3 kV/mm should be far from each other by a factor 1.94 (1-3% RH), 2.71 (50% RH), and 3.38 (90% RH), according to data of [9].

It also has to be pointed out that the ambient temperature can vary from the room temperature, and that the range of ambient relative humidity can be wide, both of them being continuously instable along the year. As an example, along six months between summer and winter, the conditions of one of the ambient-controlled laboratories where the test has been performed presented a variation of humidity from 14.7%/28.7°C (possibly associated with artificial heating on winter), to 61.7%/26.8°C on summer. If the same epoxy samples were in equilibrium with these coupled values, the current shall differ according to a factor equals to 32.1.

However samples take time to achieve equilibrium with environmental conditions. As it can be seen in Figure 8, the water uptake is mostly impacted by RH, with temperature playing with the kinetics of water absorption. It took 10 days (at 40°C and 25% RH) to achieve equilibrium; the water content did not change when temperature is increased to 60°C, and the weight measures were more instable because the equipment sensibility when the material is warm (a variation of 0.08% of weight has been found for dry samples measured at 80°C and 20°C). At 60°C, it took more 5 days to achieve a new equilibrium at 50% RH. The current density of this epoxy at 60°C varied one decade between conditioning at 25% RH and 50% RH.

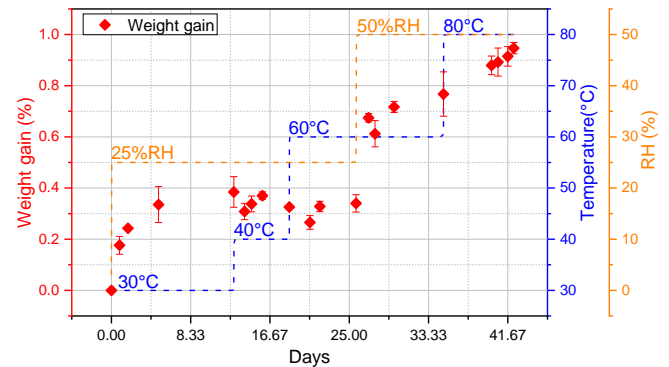


Figure 8. Weight gain (reported to the matrix) of an epoxy resin under many coupled temperature-RH changes. Samples are dried at the beginning.

If the sample is not properly conditioned and if the RH is not controlled during the electrical test, what is measured can be a metastable material. Samples are continuously losing and reabsorbing water to follow environmental changes. As an example, one epoxy sample conditioned at 40°C and 90% RH is tested at 60°C with no control of RH. Cycles of 10 hours have been performed, with voltage at polarization cycles equals to 3 kV. Figure 9 shows that the polarization currents continuously decreases, and if the conduction current was calculated, a variation of a factor 1050 would be found between the first and sixth cycle. At the end of cycles, the sample has lost 83% of the total amount of water absorbed during conditioning, even if it was shield by the electrodes.

3.2.2 Not stabilizing the temperature: for better thermal control, tests should be performed at isothermal conditions, but sometimes the temperature takes time to be achieved, or it fluctuates around a given value. Figure 10 shows a sample that is supposed to be heated at 40°C, but the test started 2 degrees Celsius before it. Until it, the sample has been electrically discharged. A thermocouple that goes inside the cell, next to the electrodes, has measured the temperature. Because of the thermal conduction of the insulating, we cannot precise if the temperature in the sample is the same of the one figured by the thermocouple, so some delay is expected. As the test starts before the stability of the targeted temperature, samples are not properly discharged as well. As a result, the discharge current (in modulus) is higher than the charge one, leading to a negative conduction current.

The authors do not believe that this material has special features leading to the creation of energy during discharge; on the contrary, in a first hypothesis, as the discharge is taking place at a higher temperature, the rate of resorption can be higher than absorption, and space charges (especially if some of them are in deep traps) will be evacuated easily. This example put together an unstable temperature, and non-properly discharged sample. We remarked that such phenomena has only been observed at lower voltages.

3.2.3 Comment about the test time-length: Some works have dealing with the inaccuracy associated with the measurement time [11]. For the silicone composite of this study, PDC cycles of 1 hour have been enough to achieve a trustable result (even at 20°C), since values obtained with longer tests remained the same (Figure 11). Actually, cycles of 30 minutes should be enough to obtain the same result.

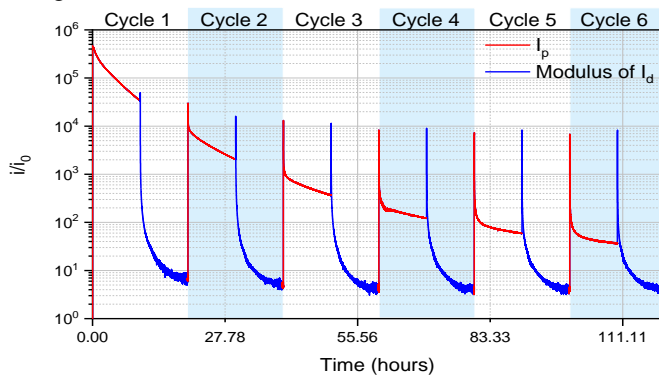


Figure 9. Relative current as a function of time for an epoxy sample conditioned at 40°C and 90% RH and tested at 60°C,

5% RH, with PDC cycles of 10 hours each, 3 kV/mm at each polarization cycle.

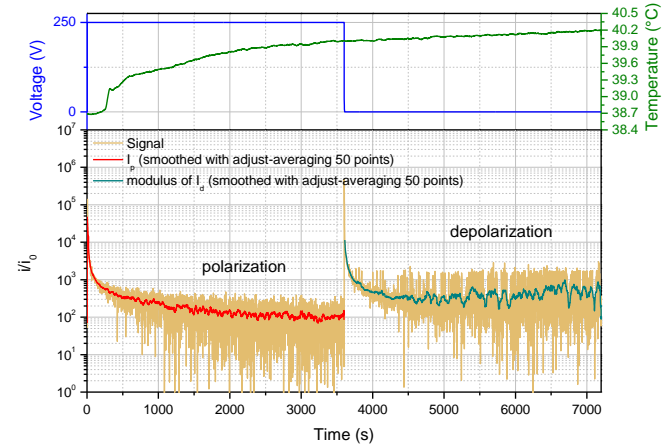


Figure 10. Relative current as a function of time for a sample tested at 250 V with a PDC cycle of 1 hour. Dried bilayer sample.

It has been noted, for example, that at 80°C, PC cycles of 15 minutes were enough (without even discharging). Effectively, the higher the temperature, the higher the water uptake, the higher the electrical current value for the same voltage levels, and the lower can be the PDC time cycle. Bearing all the factors that can impact the results, the authors considered that, for the case of epoxy samples, the 1h procedure was sufficient to give reasonable values. Finally, we are not able to suggest a general time-length without considering the particularities of each material and context.

3.3 Additional Features and Artefacts

During these experiments, both polarities (positive and negative) have been tested, but results were not conclusive. The authors consider that, at least for the materials of this study, the polarity had no relevant impact. Some additional parameters have been ignored, and authors are not able to conclude about the impact of them:

- The impact of mechanical pressure made by the electrodes, especially for elastomeric samples, as following changes of the compression set.
- Changes on hydrophobicity of silicone samples after electrical test.
- Possible changes during storage.
- The impact of thickness in the J-E plot.

As this article focus on the sample, almost no information is given about the quality of the equipment. The authors have

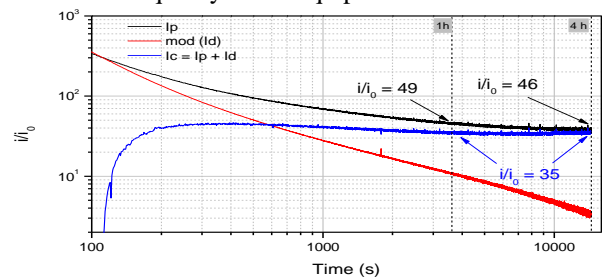


Figure 11. Relative current as a function of time for a silicone composite at 20°C and $V = 1$ kV.

encountered (and solved) many issues associated with the absence of low pass filters, noisy signals and the impact of different smooth strategies, or parasite resistances (mainly at extreme conditions, as 80°C and 90% RH, coming probably from water). They are not treated in this article. No relevant offset has been identified.

4 The Conductivity as a Failed Goal by Definition.

When the insulating is obeying the Ohm's law, the conductivity value can be found easily according to it. But what about when the material is not linear? Can we apply the Ohm's law when the material is not obeying the Ohm's law? When the Ohm's law is applied at non-Ohmic regions, we have been calling the result as the *apparent conductivity* (σ_{app}), which is very useful to design real applications. Therefore, the σ_{app} does not represent the ability with which a material conducts electric current. The current is limited by events in the interface (according to injection mechanisms) or transport in the bulk in such a way that the σ_{app} has no longer a physical meaning. Additionally, the current value seems to tell much more about the electrical insulation system (EIS) than about the insulating itself: from the example of the impact of electrodes, as electrical tests cannot be performed without them (so as the application), the current will always be biased. These observations lead to conclude that the real or intrinsic conductivity is rarely attainable by experiments. What is being measured is mostly a complex system in which results have to be framed.

5 Conclusions

There are many ways to fail a current measurement, as for example: Testing a metastable or instable sample; not evaluating the need of pre-discharge; underestimating the impact of the water content; underestimating the impact of electrode material and contacts; measuring at the room or ambient conditions; not stabilizing the temperature; and underestimating material changes during the experiment, as water (des)absorption, and ageing.

These artefacts have been remarked along many experiments and must be limited to the particular materials of this study: a silicone rubber and an epoxy resin. Each material, at particular conditions and environments, should behave differently, with results reflecting the electrical insulation system. It is up to the experimenter to test and to tell the reader if these parameters were observed during investigation. Because the current is a response of the system, the authors are not able to extract information about the material without further analysis.

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