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MINIMIZATION OF THE EFFECT OF DIELECTRIC SPECIMEN ELECTRIZATION ON THE RESULTS OF MEASUREMENT OF VOLUME RESISTIVITY

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Abstract

The impact of electric charge, gathered in a tested specimen before a testing voltage has been applied to it, on the results of measurement of its volume resistivity is considered. It was shown that the specimen should be discharged before the measurement. The alternating polarity test method, employed by Keithley's *6517 Hi-R Test* program, does not eliminate completely the effect of electric charge. A modification of the method, enabling the elimination from the measurement results of the electric charge which could be the reason of de-electrization current flow (background current) and resorption current, has been proposed.

Keywords: solid dielectrics, volume resistivity, measurement error, electrization impact, elimination of specimen electrification.

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1. Introduction

Measurement of volume resistance R_v is the basis for evaluation of volume resistivity ρ_v. The resistance R_v is defined as the quotient of the value of a constant voltage U, applied across a nonelectrified specimen of dielectric and a steady-state volume current I_u [1-3].

Applying a constant voltage U to conducting electrodes, adjacent to the surface of the specimen of a nonelectrified dielectric results in a flow of volume current $I_v(t)$, which decreases asymptotically in time up to a certain steady-state value I_u (Fig. 1). The current $I_v(t)$ is a superposition of absorption current $I_a(t)$ and steady-state DC resistance current I_u [4-7].

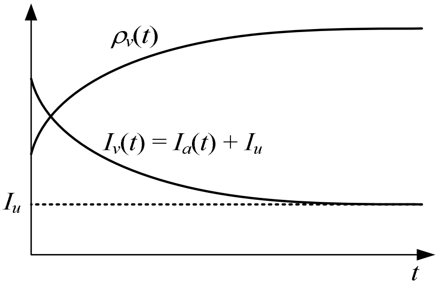

Fig. 1. Time dependencies of volume current $I_v(t)$ and volume resistivity $\rho_v(t)$ for a specimen of nonelectrified dielectric.

The reduction of current in time is a result of dielectric polarization and other relaxation processes. In case of materials with volume resistivity ρ_v below 10^{12} Ωcm, the volume current achieves the steady-state usually within 60 s from the application of voltage to the electrodes.

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In materials with higher volume resistivity, the current may decrease during the following minutes, hours, days and even years.

In case of materials with very high resistivity, which achieve the steady-state of volume current after a very long time, the actual volume resistance is defined as the ratio of voltage applied to the electrodes and the actual current $I_v(t)$. The result of resistivity measurement is displayed in the form of a time characteristics $\rho_v(t)$.

In materials with high resistivity values, the characteristics $I_v(t)$ and $\rho_v(t)$ depend not only on the kind of material but also on the state of its electrization before measurement. Actual characteristics $I_v(t)$ and $\rho_v(t)$ may differ from the ones shown in Fig. 1, corresponding to a nonelectrified dielectric. The standards [1-3] and literature [4, 6-8] suggest either discharging of the specimen before measurement or subtraction of background current from the absorption current or carrying out the measurements at positive and negative voltage [9, 10]. It lacks, however, a detailed analysis of the effect of specimen electrification on the measurement results and some of the proposed methods of elimination of the impact of charge gathered in a specimen on measurement results, like for instance alternating polarity, recommended by Keithley [9, 10] do not seem to be correct.

The author presented in [7] the problems connected with the measurement of volume resistivity of solid dielectrics and a general analysis of the factors affecting the uncertainty of its measurements. In the current paper, the impact of dielectric specimen electrization on the results of measurement of volume resistance and methods of minimization of the effect, have been discussed in detail.

2. Electrization of dielectrics

Dielectric materials are capable of gathering and storing of electric charge even without an electric field. The electrifying of a dielectric can be unintended and may occur during the processes of its fabrication, storing, preparing of specimens (deposition of electrodes) and during measurements in an electric field (for instance, of volume or surface resistance). The electric charge can be collected in the specimen of the tested dielectric in a subsurface layer (surface charge) or can be embedded in the bulk of the whole specimen (space charge). The elimination of such space charge is very difficult and therefore the charge can be a source of considerable measurement errors.

The reasons of dielectric electrization may be diverse like triboelectrization, the effect of an outer electric field or transfer of electric charge from one electrified body to another – not electrified [11]. The triboelectrization takes place during a contact of two different bodies in the result of charge transfer from one body to another. Two nonelectrified bodies connected with each other become electrified after their separation. It is an effect of formation of a double charge layer at the interface of two bodies with different emission work (different Fermi levels) [12]. The effect of triboelectrization can be enhanced by friction or a stroke. One of the reasons of such enhanced electrization during the friction is an increase in the number of contact points, what intensifies the formation of charge by contact electrization. The friction facilitates the charge transfer from one surface to another. Another reason of the friction effect on electrization is an increase in surface temperature during this action. The temperature of particular parts of one body surface may achieve such a value that ions from the parts with enhanced temperature would be able to pass to another surface with lower temperature. Some heterogeneities in friction faces facilitate such occurrence.

The contact electrization of dielectrics depends on their electric permittivity. In the system of two dielectrics being in contact, the dielectric with higher value of electric permittivity obtains a positive charge. The density of the surface charge is proportional to the difference of permittivities of both dielectrics being in contact. During the contact of solid dielectrics with metals, the metal gains either a positive charge (noble metals) or negative charge (common metal).

Electrization in the effect of friction can be classified as follows:

- Electrization by friction can lead to formation of charge with high value.
- Electrization can take place both during the friction of similar and quite different materials. The electrization can also occur during friction of a dielectric material with metal or during a stroke by crumbled material, for example dust or liquid.
- The charges occurring during friction are not uniformly distributed on the friction faces. In different parts of the face they can have different densities and even a minus or plus sign.
- The effect of electrization during the friction process depends on surface roughness. The same material can occupy different places in the triboelectric series, depending on the state of the surface.
- If besides the friction, another reason of electrization occurs, the phenomena superimpose. For example, friction in an electric field may result in the creation of a larger of smaller charge, depending on the field's direction. The electrization can also be a result of friction of dielectric by air containing dust particles or condensed water vapor.

The electrization by contact and friction is a natural phenomena and takes place without the contribution of an external electric field. The specimens prepared for measurement can be subjected to such effects.

Electrifying of a dielectric can also occur in the result of an external electric field, for instance during measurement of resistance. The dielectric subjected to the outer electric field is being charged. It gathers both surface and space charges. After switching off the source of the outer electric field, the electric charge is preserved in the dielectric and it decays with time The rate of the decay depends on the resistivity of the dielectric. In the dielectrics with very high values of resistivity, the charge can be maintained for many hours, days and even weeks, months and years (electrets). The value of the surface potential resulting from such charge may exceed even 1000 V. In such case, application of the 1000 V voltage to the specimen results in a current flow in the circuit in the opposite direction, compared with the case when the dielectric is not charged.

Electric charge transfer from an electrified body can also be the reason of electrization of a dielectric specimen. For example, a person charged in the result of friction of his/her body by clothing made of plastic fabric, who carries out the tests and touches the tested specimen, can transfer its charge to the specimen causing its electrifying.

The charge collected in the specimen can be a source of a de-electrization current $I_{de}(t)$, called also background current [9]. Therefore, the current flowing across a charged specimen in the result of a constant voltage applied to the electrodes, contains additionally the $I_{de}(t)$ component. The occurrence of the current before applying a voltage can be stated only by short-circuiting the specimen by a measuring instrument. The de-electrization current $I_{de}(t)$ can alternate its direction in a prolonged period of time (Fig. 2a) and may have an important impact on the resultant current $I(t)$ in the specimen (Fig. 2b).

The specimens prepared for resistivity testing should be deprived of electric charge because such charge falsifies the measurement results. De-electrization of the specimens is performed by short-circuiting of electrodes, between which dielectric has been placed, for a longer time and then their grounding. The method does not always turn out with success, specially for dielectrics with very high values of resistivity, like for example polytetrafluoroethylene (PTFE). The dielectrics can be discharged by placing the short-circuited specimen at elevated temperature. In case of PTFE the temperature amounts to 250 °C. However, such high temperature may cause structural damages in the material.

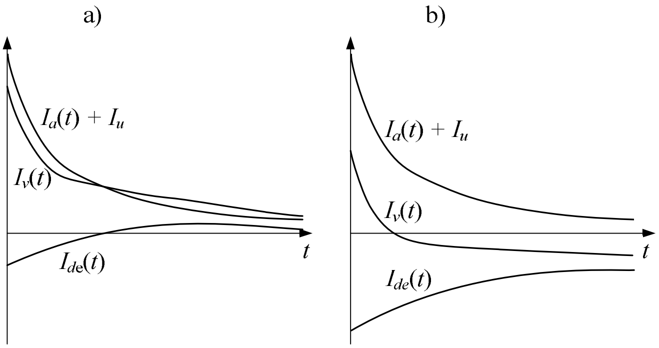

Fig. 2. Effect of de-electrization current on the resultant current in the circuit of dielectric specimen: a) de-electrization current $I_{de}(t)$ alternates its direction, b) de-electrization current $I_{de}(t)$ does not alternate its direction but produces an alternate direction of volume current $I_v(t)$.

If the source of voltage was switched off and then repeatedly switched on, for instance with alternated polarity, then the background current would be the sum of the de-electrization current $I_{de}(t)$ and the resorption current $I_r(t)$, whereas the resultant volume current in the specimen:

$$I_v(t) = I_u + I_a(t) - [I_{de}(t) + I_r(t)]. \tag{1}$$

The state of the tested specimen can be represented by the equivalent circuit shown in Fig. 3 [4]. The resorption current $I_r(t)$ is decaying much faster than the de-electrization current $I_{de}(t)$.

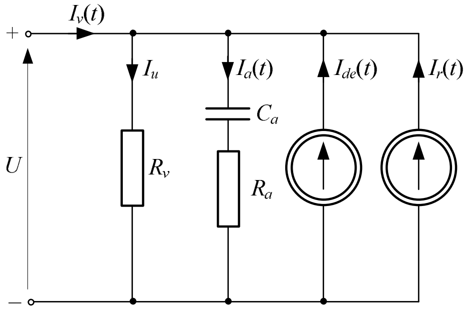

Fig. 3. Equivalent circuit of an electrified specimen of dielectric: U – the voltage applied to the electrodes, $I_v(t)$ – resultant volume current, I_u – a component of steady state volume current, I_u – absorption current, $I_{de}(t)$ – de-electrization current, $I_r(t)$ – resorption current, R_v – volume resistance, C_a and R_a – capacity and resistance of the relaxation branch.

For simplicity, the short-lasting current charging the condenser formed by the electrodes and the tested dielectric, has been neglected in equation (1).

3. Effect of specimen electrization

The charge gathered in a specimen is the source of de-electrization current $I_{de}(t)$ (Fig. 4a), which can be replaced by a source of equivalent voltage $U_z(t)$ (Fig. 4b) [7, 8].

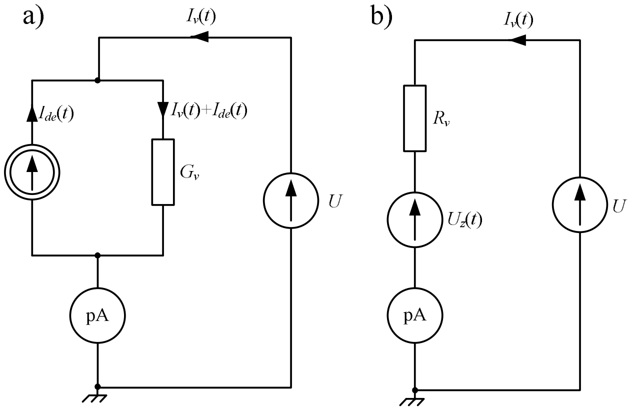

Fig. 4. A simplified circuit for measuring of volume resistance including the charge If the electrodes are placed on both sides of the specimen, then the de-electrization current I_{de} can be easily measured by short-circuiting the electrodes (before applying the voltage) by a current-measuring instrument. gathered in the dielectric
a) parallel equivalent circuit of dielectric, b) series equivalent circuit after employment of Thevenin's theorem.

The measured actual volume resistance in the equivalent circuit shown in Fig. 4a:

$$R_v(t)=\frac{1}{G_v(t)}=\frac{U}{I_v(t)-I_{de}(t)}, \tag{2}$$

and in the circuit in Fig. 4b:

$$R_v(t)=\frac{U-U_z(t)}{I_v(t)}. \tag{3}$$

The equivalent voltage U_z can be measured in the dielectric with only one single electrode. The I_{de} current alternates in time t and can change in the time interval between the measurement of its value and measurement of volume resistance. Therefore, it is not easy to take into account its correction. The time characteristics of the current may have sometimes a surprising run. An example of such characteristics, recorded by a computer in 5 hours, for PTFE specimen is shown in Fig. 5 [4, 13]. The de-electrization current initially increases, reaches a maximum and then decreases and alternates its direction. Even after 5 hours it does not approach a zero value.

The charge gathered in a specimen may be so large that the indications of the instrument measuring the current or resistance can be negative. It means that the equivalent voltage U_z is higher than the applied voltage U, which is sometimes as high as 1000 V. In such a case, the person carrying out the measurements will be aware that the obtained result is nonsense. Much worse is, when the equivalent voltage is lower than the measuring voltage or has reverse polarization, since an inexperienced person carrying out the measurements may recognize such result as being correct.

The sample had an evaporated silver electrode on one side whereas clamping electrodes covered with conducting rubber were used as measuring and guard electrodes. Before the measurements the specimen was thoroughly de-electrified *i.e.* almost completely deprived of electric charge by long lasting short-circuiting by an electrometer measuring the current. Then, the specimen was placed in a screened chamber and its volume resistance was measured with a 6517A electrometer by Keithley, equipped with a data-processing system enabling the registration of the volume resistance ρ_v versus time. The measurements were carried out at a voltage of 1000 V. After the measurements, the specimen was once again discharged, the measuring electrode with a guard ring was removed and a corona electrode was inserted in its place. The specimen was intentionally electrified by a corona discharge. Next, the corona electrode was removed, a detector of electric field was put in its place and the value of the equivalent voltage U_z was measured. Further, the measurements of resistivity versus time were repeated. The resistance characteristics of a specimen with negative charge ($U_z < 0$) approached higher values than the characteristics of a nonelectrified specimen, the higher the greater the value of the negative equivalent voltage was (curves 2 and 3 in Fig. 6). In case of electrifying a specimen with positive charge ($U_z > 0$), the resistivity characteristics ran below the characteristics of the nonelectrified specimen (curve 4).

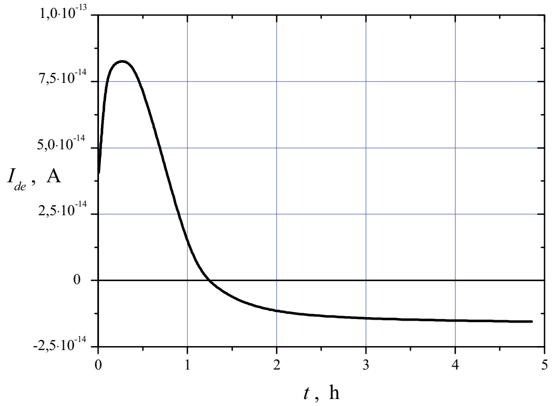

Fig. 5. Time characteristics of the de-electrization current of a PTFE specimen.

An example how the electrization of a specimen affects the measurement results of resistivity is shown Fig. 6.

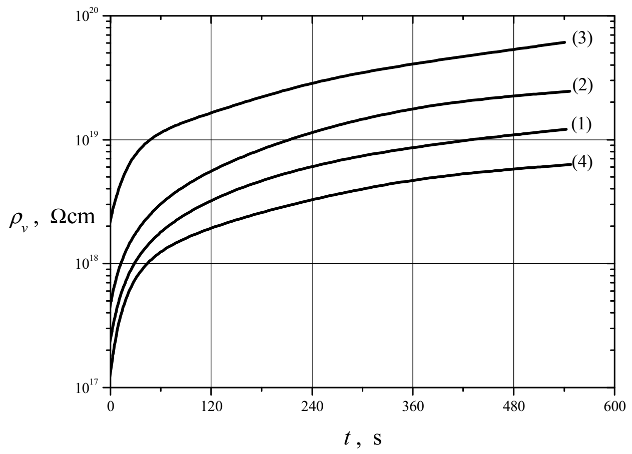

Fig. 6. Impact of specimen electrization on resistivity measurement results: without electrization (1), after electrization, at $U_z = -300$ V (2), $U_z = -790$ V (3), $U_z = +400$ V (4).

IEC 60093 standard [1] recommends de-electrization of a specimen before measurement by short-circuiting its electrodes and measuring the de-electrization current until it approaches its steady-state value, very low in comparison with the expected steady-state value of volume current or with the current after 100 min from the application of the voltage. Sometimes, even the short-circuiting of specimen electrodes for several days does not bring satisfactory results.

It happens that after long-lasting short-circuiting of the electrodes and inserting the specimen into a measuring circuit, the volume current is still flowing in the opposite direction even when a voltage of 1000 V is applied to the specimen.

Such specimen can be discharged in reasonable time by placing it with short-circuited electrodes at an elevated temperature. Fig. 7 presents the results of measurement of the de-electrization current of a PTFE specimen after its annealing in a short-circuited state at 250 °C for 120 min followed by cooling down.

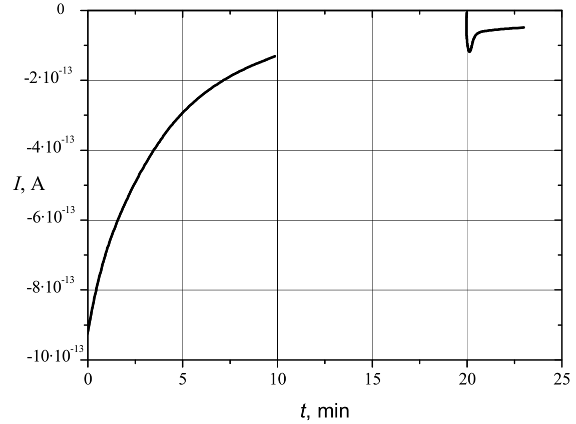

Fig. 7. Time characteristics of resorption current of a PTFE specimen after annealing for 120 min at 250°C.

The values of the de-electrization current were registered for 10 minutes. Then, the specimen was open-circuited for 10 min and short-circuited again by the current-measuring instrument. The open-circuiting of the specimen did not cause an occurrence of the return current and the value of the resorption current was at the level of 10^{-15}A, *i.e.* its value was definitely lower than the measured absorption current. It can be stated that the thermal discharge resulted in the release of a collected electric charge. Such measuring procedure is however time consuming and expensive and may additionally affect the structure of the tested material.

Polish standard PN-E-04405:1988 [3] recommends discharging a specimen so long until the value of the discharge current reduces to a value ten times lower than the expected current value during the measurements. It should be pointed out that at such specimen discharge, the measurement error caused by the charge gathered in the specimen approaches 10%.

If the specimen discharge in a reasonable time is impossible and the de-electrization current I_{de} reached its steady-state value, then further de-electrization should be ceased, the specimen electrodes open-circuited and the voltage source switched on. In such a case the volume resistance could be evaluated from equation (2). However, it should be kept in mind that the current I_{de} can have either a positive or negative value.

4. Alternation of polarization of the applied voltage

If the electric charge did not change during the measuring procedure, the impact of the surface potential U_z – and thus the electrization of the specimen before the measurement - could be eliminated by carrying out the measurements at a supplying voltage U_p with positive and negative polarization, but of the same value. As the result of such measurement an average value should be assumed [4, 13].

Keithley Co. offers the *6517 Hi-R Test* program for measurement of resistance at cyclic alternation of voltage polarization [10]. The procedure includes repeatable measurements of absorption current with periodically alternating polarity of the applied voltage. In the first step, when the polarization voltage is positive, the time characteristics of the volume current is registered in an assumed time. In the second step, the voltage polarization is alternated at the same voltage value, and the volume current characteristics is registered in the same time as in the first step. In the third step, the voltage polarization is alternated once again and the characteristics of volume current is registered. The steps are repeated several times and the number of steps is adjusted by the operating software. In Fig. 8, some results of the cyclic measurements of volume currents of a PET foil with a thickness of 100 μm are presented.

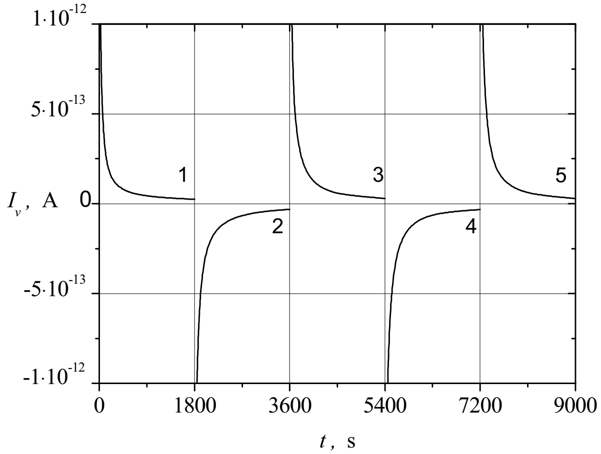

Fig. 8. Dependences of volume currents of PET foil specimens obtained with the use of the *6517 Hi-R Test* program in successive steps (1, 2, 3, 4, 5) at a polarization voltage $U = \pm100$ V.

As a final measurement result, the *6517 Hi-R Test* program computes the weighted average of final values of currents of each step of the last four measuring cycles from the equation:

$$\bar{I}_v = \frac{I_1 + 3(-I_2) + 3(I_3) + (-I_4)}{8}. \tag{4}$$

In Fig. 9, the characteristics of specific volume currents obtained in successive measurement steps are compared. As the tested foil specimen was thoroughly de-electrified before the measurements, the characteristics $I_v(t)$ of the first step does not reveal any charge effect.

During polarization, an electric charge is introduced into the specimen. After alternating the polarization of voltage U (according to the mentioned procedure), the charge gathered in the specimen makes that the run of volume current characteristics at the second step is different from the run in the first step. It can be clearly seen in Fig. 9 where the time characteristics of volume current during the first step is completely different from the characteristics in successive steps. For this reason, the value of volume current described by equation (4), used for the estimation of volume resistivity by the *6517 Hi-R Test* program is burdened by a significant error. In spite of the fact that the considered method enables elimination of the de-electrization current from the measurement results, it does not eradicate the harmful resorption current from the background current. The errors resulting from this fact may be quite significantly the higher, the longer the cycle lasts. Unfortunately, Keithley Co. does not inform the software users about the fact.

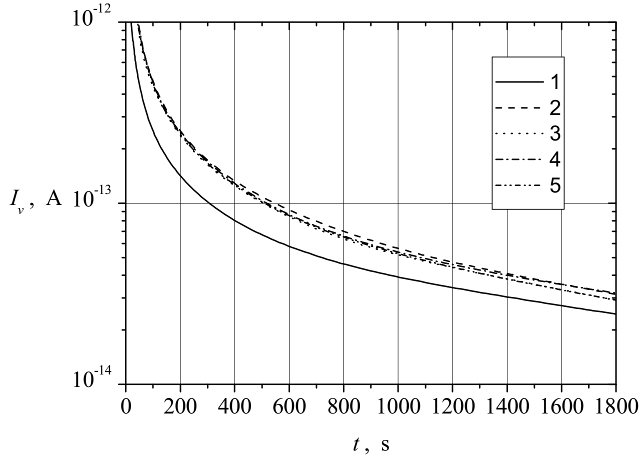

Fig. 9. Comparison of time characteristics of volume currents of PET foil in five successive measuring steps at the test voltage $U = \pm 100$ V.

5. Correct method of alternate polarization of polarization voltage

To enable correct measurements of volume and surface resistivity by the method of alternating voltage polarization, the author has proposed an essential modification of the method applied in the Keithley software. The modification consists in the fact that the specimen is short-circuited before the polarization voltage is applied to it and the value of the resorption current is measured. An exemplary run of a measurement of PET foil is shown in Fig. 10. In Fig. 11, the time characteristics of volume currents obtained in successive measurement steps are compared. The characteristics almost overlay, so they can be averaged and the correct value of current at an assumed time t as well as average values of volume current can be estimated. The values can be used in the assessment of correct results of volume resistivity.

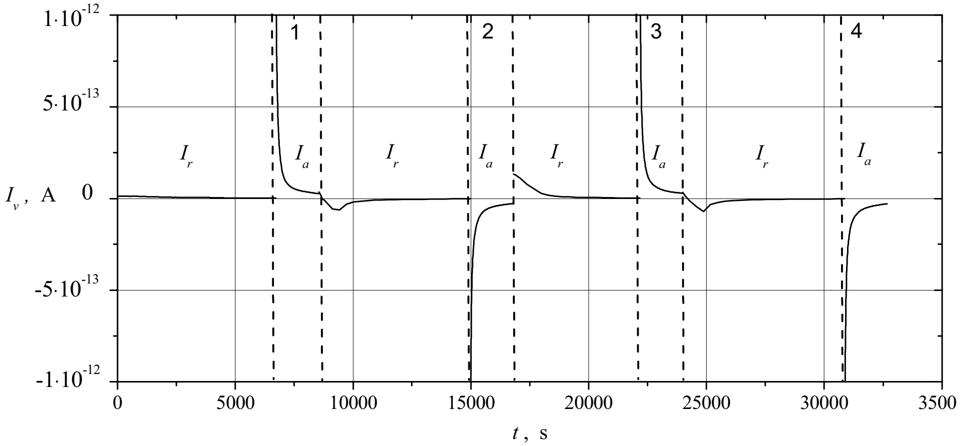

Fig. 10. Time dependences of volume currents of the specimens made of PET foil, obtained by the method of alternate polarization with specimens discharging before each step, at a polarization voltage $U = \pm 100$ V.

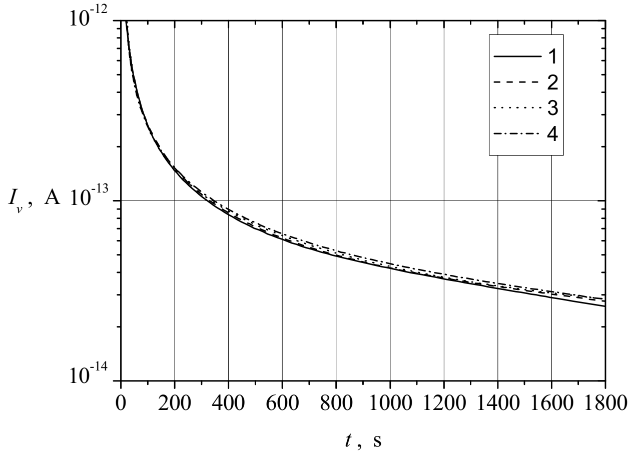

Fig. 11. Overlay time characteristics of volume currents of four successive steps obtained by the specimens of PET foil by alternate polarization method at a voltage $U = \pm 100$ V.

The proposed method of resistivity measurement eliminates the effects of electric charge gathered in the specimen before the measurements during its preparation and storing, which is the source of a de-electrization current, as well as the charge, created directly before the measurement, being the source of resorption current. The method fulfills the recommendations of the IEC 60093 standard so as the resistivity measurements were performed at two voltage polarizations. It also enables one to obtain an average result from multiplied repetitions of the measurement for the same specimen, what significantly improves the accuracy of resistivity measurements and makes them more reliable. The method is accomplished automatically by a specially developed software, enabling the co-operation with the Keithley 6517A electrometer and a personal computer.

6. Conclusions

The charge collected in a specimen before a resistivity measurement can have an important effect upon the measurement results of resistivity. Therefore, the tested dielectric specimen should be thoroughly discharged before the resistivity measurement. Employing a cyclic alternating polarization does not completely eliminate the effect of the charge on measurement results. The alternate polarization introduces to the specimen an additional charge from the preceding cycle. The most correct results can be obtained for the first cycle which should be preceded by thorough discharging of the specimen. Repetition of the measurements without repeated discharging of the specimen may lead to incorrect measurement results. The improvement in accuracy of the results can be obtained by multiple repetition of the measurements and the average values can be recognized as the most reliable. For the realization of the method, the author suggests a procedure with discharging the specimen after each cycle by short-circuiting it and measurement of the resorption current until it reaches a negligible value. The method enables elimination of the charge effect on the measurement results of the time characteristics of absorption currents. The average values of the currents in successive measuring cycles can be taken for the estimation of correct values of volume resistivity of the tested material specimens.

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