

## Universal installation for studying structural defects in electrical and optical fiber materials

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**Abstract.** A universal device for experimentally studying the migration of microscopic structure defects and the features of dielectric relaxation is proposed. It allows using the thermo stimulated depolarization method, in combination with the measurement of the tangent of the angle of dielectric losses and the thermo stimulated polarization current, to perform dielectric spectroscopy of hydrogen-bonded crystals and perform analysis of the properties and parameters of structure defects. A smaller (in comparison with the existing installation) additional compact device for measuring small values of electrical capacitance and the tangent of the angle of dielectric losses, including an electrometer B7-30, was designed, measurement was carried out using a q-factor meter VM 560. When measuring  $\tan \delta \geq 0.1$ , the VM-507 device was used. An experimental methodology is proposed that allows, in combination with the method of minimizing the comparison function (MFC – method), with a high degree of accuracy, to calculate the molecular characteristics of structural defects in composite materials based on semiconductors and dielectrics used in the electrical and optical fiber industry, electric power and insulation technology.

### 1. Introduction

Modern methods of experimental and theoretical research in the field of optical fiber technology are based on the achievements in the field of optoelectronics, wave technology and nonlinear optics. When performing schematic diagrams of optoelectronic devices for monitoring and controlling parameters of various levels of complexity of technological processes, it is important to address the issue of developing high-speed systems for automated control of the wave characteristics of the light flux (pulse) entering and leaving the fiber-optic channel. The question remains open about the development of universal theoretical methods for describing nonlinear electron-optical processes that are implemented in the working channels of control and measurement fiber-optic devices operating in a wide range of optical radiation parameters. The issue of developing effective methods for computer prediction of material properties for working elements of fiber-optic devices is relevant. This is a separate theoretical problem closely related to experimental and production tests and based on methods of solid state theory, wave physics and quantum electronics [1].



The theoretical and methodological foundations of a nonlinear physical and mathematical model of optical (wave) processes occurring in a diffraction grating (with rectangular holes), which acts as a fiber-optic sensor of external action, with variable characteristic parameters of the system are laid in the work [1]. In addition, we also used methods of research and processing of experimental data considered in the works [2, 3].

Defined interest is attracted by the study of kinetics of low-temperature dielectric relaxation in layered crystals in alternate polarization field. The effects of nonlinear polarization in a dielectric with hydrogen bonds have already been described earlier in [4]. The results obtained are used by us in our research. We also used the methods of research and processing of experimental data discussed in [5]. We used graphing methods and the choice of approximations described earlier [6].

In this paper, we propose a universal installation for experimental study of migration of microscopic structural defects and features of dielectric relaxation, which allows the method of thermo stimulated depolarization in combination with the measurement of the tangent of the angle of dielectric losses and thermo stimulated polarization current to conduct dielectric spectroscopy of crystals with hydrogen bonds and analyze the properties and parameters of structural defects. A smaller (in comparison with the existing installation) additional compact device for measuring small values of electrical capacitance and the tangent of the angle of dielectric losses, including an electrometer B7-30, was designed. the measurement was carried out using a q-meter VM-560. The device VM-507 was used for measurements. An experimental methodology is proposed that allows, in combination with the method of minimizing the comparison function, with a high degree of accuracy, to calculate the molecular characteristics of structural defects in composite materials based on semiconductors and dielectrics used in the electrical and optical fiber industry, electric power and insulation technology.

## 2. Justification of the proposed solutions. Statement of the research problem

The technical capabilities of modern software and hardware and computer technology allow us to develop and implement high-speed multi-functional schemes of automated control of the experiment, with the possibility of simulation and control (by analog-to-digital signal conversion) in real time, quite complex physical processes occurring on the functional elements (working body) of electrical circuits of various types of devices (control and measuring; electronic-computing; electronically controlled systems of the microwave range; electronic-optical converters, etc.) [7-14].

The experimental measuring device proposed in [15] is accepted as the basic one.

Of particular interest are composite materials of heterogeneous functional elements of technological systems operating under extreme conditions: ultra-low temperatures (near helium); strong electric fields and ultra-high temperatures (near breakdown values); intense laser (coherent) radiation; strong magnetic fields [16].

The study of the electrophysical properties of dielectrics with a complex crystal lattice structure, in particular crystals with hydrogen bonds [17], is based on a complex comparison of theoretical (calculated) with experimental results obtained by measuring the spectra of thermostimulated polarization currents, depolarization and complex dielectric permittivity of materials [15].

The authors [13,18] proposed an effective method for experimental determination of the parameters of microscopic structure defects (defects of Bjerrum and dipole water molecules) in dielectrics of the KHB class, based on a measurement scheme that allows a high degree of accuracy in terms of current strength (the measurement error of the installation was no more than  $\pm(3 \div 5) \cdot 10^{-15}$  A), to measure the temperature spectra of thermally stimulated polarization and depolarization currents. The rate of natural heating of the sample was adopted  $0.1 \cdot \frac{K}{c}$ . However this installation did not allow measuring the electrical capacity  $C < 10^{-11}$  F and  $\text{tg} \delta < 10^{-3}$  [13,18].

The purpose of this work is to develop a schematic diagram and design an improved universal installation for precision measurements of the density spectra of thermo stimulated depolarization currents (TSDC) and the tangent of the angle of dielectric losses in dielectrics with a complex crystal

structure, in a wide range of values of the polarizing field strength (100 kV / m-1000 MV/m)) and temperatures (1 – 1500 K). The proposed installation will be integrated into the automated measuring circuit in the future [12]. Experimental density spectrum TSTD obtained using this measurement scheme  $J_{\text{exp}}(T)$  and  $tg\delta_{\text{exp}}(\omega;T)$ , after their comparison with the temperature dependences calculated from the theory,  $J_{\text{theory}}(T)$ ,  $tg\delta_{\text{theory}}(\omega;T)$  will be used for numerical calculation of the parameters of relaxers in dielectrics, in the temperature range  $T=1...1550$  K [19-21].

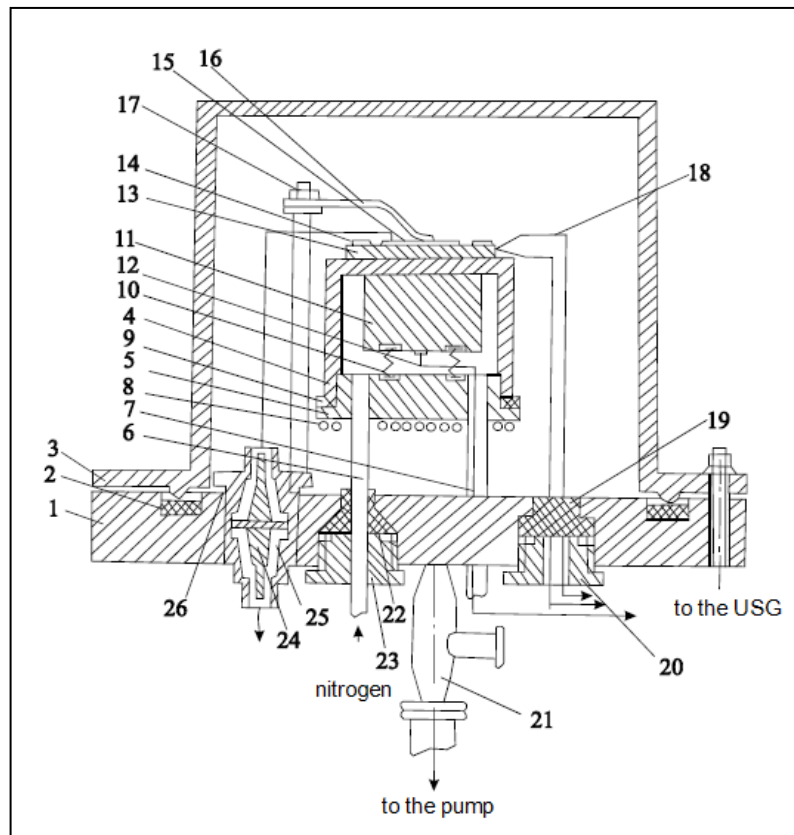
The technical characteristics of a universal measuring device must correspond to a sufficiently high degree of accuracy of the previously developed numerical optimization scheme based on the method of minimizing the function of comparing the theory with the experiments given, in the work [22-24].

### 3. Improved experimental setup for measuring thermally stimulated currents and the tangent of the dielectric loss angle

The authors have developed and tested a universal installation (Figure 1), which allows to determine the complex dielectric permittivity ( $\hat{\epsilon} = \epsilon' - i\epsilon''$ ), the specific volume electrical conductivity ( $\sigma$ ), to measure the temperature spectra of thermally stimulated polarization and depolarization currents, with the possibility of exposure to the sample by ultrasound. Measurements were made at a temperature of  $T = 70 - 550$  K, in the frequency range of the polarizing electric field of 50 kHz–35 MHz. On a steel base 1 with a rubber gasket 2 and a vacuum shielding cap 3, a hollow lower electrode 4 is attached, to the cover of which 5 are welded tubes 6,7, designed for the input and output of nitrogen vapors.

On the lower surface of the cover of the lower electrode is a spiral heater 8, fed by a direct current. The cover 5 is tightened hermetically using a fluoroplastic gasket 9. Inside the electrode 4, a radiating ultrasonic transducer 11 is fixed with the help of springs 10. Isolated input 12 from the generator of ultrasonic vibrations (USG) was carried out through the nitrogen output tube 7, which simplifies the design of the cover 5. Vacuum lubrication is used to improve the contact of the ultrasonic transducer with the electrode body. Nitrogen is pumped by increasing the pressure of its vapors by heating a resistance spiral lowered with a tube into the liquid nitrogen in the Dewar vessel. The test sample (dielectric) 13 with a guard 14 and measuring 15 electrodes is placed on the lower hollow electrode 4 and pressed by a thin plate spring 16, fixed on an insulated rack 17. The temperature measurement was performed by means of a chromel-alumel thermocouple 18 fixed in a hole in the upper part of the lower electrode.

The input of the thermocouple is carried out through a connector consisting of a dense rubber gasket 19 and a pressure nut 20. Air is pumped out through the connection 21, welded to the base. Tubes 6 and 7 for pumping nitrogen are attached to the base using fluoroplastic gaskets 22 and pressure nuts 23. The vacuum electrical input consists of a metal core 24, fluoroplastic gaskets 25 and a pressure nut 26. The lower part of the connector has a thread for connecting the connector from a power supply or an electrometer. To measure small values and we have designed an additional compact device (Figure 2), smaller in size, in comparison with the installation shown in Figure 1. Case 1 (top view) was made of fluoropolymer. The housing has rubber mounts for the thermocouple 2 and a connection 3 for pumping air. The device of the lower hollow electrode 4 is similar to that shown in Figure 1. Nitrogen input and output tubes are attached to the body by means of pressure nuts 5. The vacuum electrical input is assembled on the basis of a plate 6 of fused quartz having a dielectric loss angle of  $(1-2) \cdot 10^{-4}$ . The electrical contact 7 is inserted through a hole in the center of the plate. The plates and the inlet are sealed with rubber gaskets 8 and pressure nuts 9. The pressure is about 1 PA.



**Figure 1.** Installation design for measuring thermally stimulated polarization currents, depolarization and dielectric loss tangent [15]

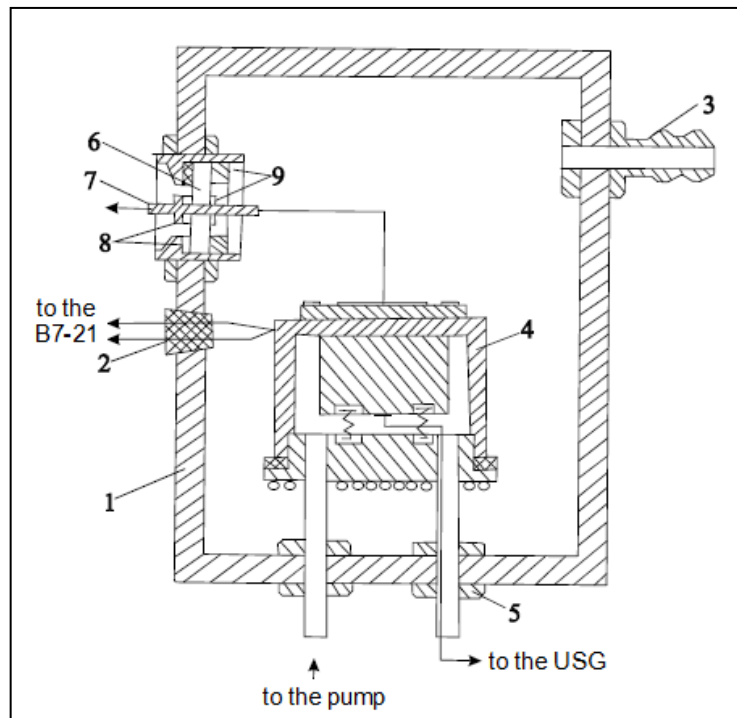
The cover is made of plexiglass and pressed against the body 1 through a rubber gasket. The entire device fits into a metal screen.

Given the design of the meter q VM-560, the device is mounted directly on the device, which allowed to reduce the length of the lead wires to 5–6 cm. In the result managed to provide a measurement 11, and the electrical capacitance to a fraction of picofarad.

Error  $\frac{\Delta(\text{tg}\delta)}{\text{tg}\delta}$ , when measuring  $\text{tg}\delta$  in the specified range, is quite significant. So, at  $\text{tg}\delta < 10^{-3}$ ,

$\frac{\Delta(\text{tg}\delta)}{\text{tg}\delta} = (40 \div 50) \%$ , and at  $\text{tg}\delta \geq 10^{-3}$ ,  $\frac{\Delta(\text{tg}\delta)}{\text{tg}\delta} = (6 \div 10) \%$ . However, given the complexity of

measurement  $\text{tg}\delta$  and  $\epsilon'$ , in the low temperature range (70–100 K) in a vacuum, greater accuracy, at present, can not be achieved. The influence of surface conductivity is excluded by protective ring electrodes or by the choice of sample sizes that provide a large path difference across the surface and between the electrodes. A diameter-to-thickness ratio of 20:1 was selected.



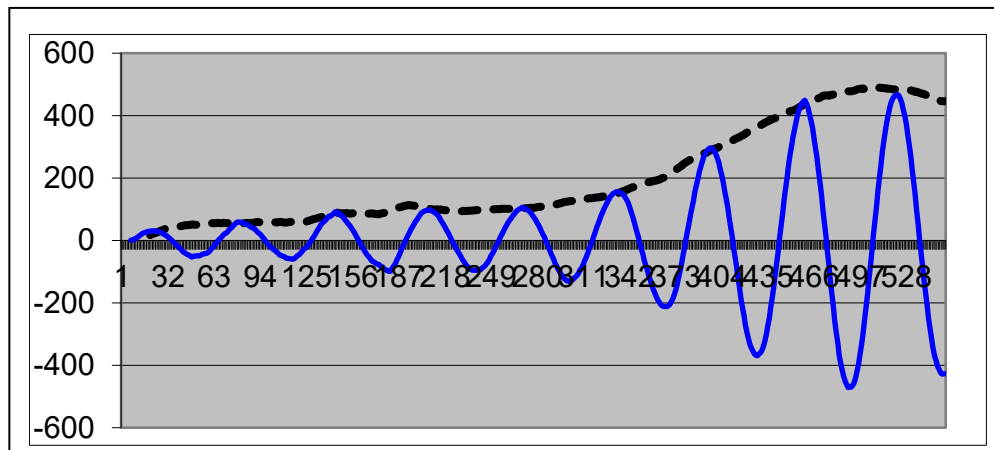
**Figure 2.** The design of the system to measure small values  $\operatorname{tg} \delta$  and  $\varepsilon'$ . This element is additional to the schema [15]

#### 4. Description of the measuring scheme and theoretical bases of numerical processing of experimental results

To measure the dielectric characteristics of materials, we have assembled a measuring circuit that includes the following elements [15]: 1) a sample placed in a measuring cell; 2) stabilized voltage source of the model UPS-2; 3) The device for measuring currents (electrometer V7-30 or the quality meter VM-560, for measuring  $\operatorname{tg} \delta < 10^{-3}$ ; 4) remote device (potentiometer KSP-4), shunt, ultrasonic transducer, ultrasonic generator, protection unit. To measure the temperature, a voltmeter V 7-21 was used with a differential thermocouple, one spike of which was placed in a zero-thermostat (ZT), where the temperature of the melting ice was maintained. Calibration of the thermocouple was performed using a DC bridge MOD-61 with an accuracy of  $\pm 0.5$  K.

In order to increase the degree of accuracy in the experimental determination of parameters of microscopic defects of the structure, in addition to the scheme described in [15], we have proposed and tested in practice, an automated control system for control and measurement processes based on the method of a sound card embedded in the computer's micro-processes. Out of the total number of existing methods, this method is the simplest in terms of its technical implementation with the help of improvised tools - no microcontroller firmware is required, the use of ADC, etc., and the sound card with ready-made drivers is completed as part of almost any computer [13].

The thermo-EMF from the thermocouple and the thermally stimulated current from the sample, according to the upgraded measurement scheme, are sent to the line input (Line in) of the sound card via the M1 and M2 modulators assembled on the K140UD13 chip, after conversion to an alternating signal. Figure 3 shows a fragment of the signal that illustrates the operation of modulators before amplitude modulation (dotted line) and after amplitude modulation (solid line).



**Figure 3.** Fragment of a modulated signal when measuring the current spectra of thermally stimulated depolarization in layered minerals

The calibration error of the VM-560 generator scales did not exceed  $\pm 0.5\%$  in frequency. The measurement error of  $q$  in percent of the measured values does not exceed  $\pm \left(3 + \frac{Q_x}{Q}\right)$ , where  $Q_x$  – end value of the scale which is the reference  $q$ -factor,  $Q$  – the measured value of the quality factor. The price of division of the measuring capacitor is 0.1 pF, the basic error in capacity does not exceed 1%. Calculations on the  $q$ -meter VM-560 were carried out according to the equation:

$$\operatorname{tg} \delta = \frac{(Q_1 - Q_2)(C_0 + C_1)}{(C_1 - C_2)Q_1Q_2} \quad (1)$$

where  $C_0$  – is the intrinsic capacitance of the replaceable inductors,  $Q_1, Q_2, C_1, C_2$  – is the  $q$ -factor and electrical capacitance of the circuit tuned to resonance, without a capacitor and with a connected capacitor with the object.

The V7-30 electrometer allows you to measure currents of both polarities from  $10^{-15}$  A to  $10^{-7}$  A, DC voltages from  $10^{-4}$  V to 10 V, charges  $10^{-15}$  K1 to  $10^{-9}$  K1, resistances from  $10^6$  Ohms to  $10^{18}$  Ohms. The limits of the values of the permissible basic error of current measurement  $\Delta = \pm(A + 0.05I_x)$ , where  $A$  – is the error of the electrometer's discreteness, equal to 2 units of the lower discharge,  $I_x$  – is the actual value of the measured value. The P589 digital automatic bridge was used to measure electrical capacitance and  $\operatorname{tg} \delta$  at a fixed frequency of 1 kHz. The object was supplied with a voltage of 80V. the Limits of measuring the tangent of the angle of dielectric losses and capacitance are equal to  $3 \cdot 10^{-4} \leq \operatorname{tg} \delta \leq 10^{-1}$ ,  $10^{-14} \leq C \leq 10^{-5}$  F. errors of capacitance measurement and  $\operatorname{tg} \delta$ , respectively.

$$\frac{\Delta C}{C} = \pm \left(1 + \frac{2}{C_x}\right) \quad (2)$$

$$\frac{\Delta(\operatorname{tg} \delta)}{\operatorname{tg} \delta} = \pm(0.02 \operatorname{tg} \delta_x + 3 \cdot 10^{-4}) \quad (3)$$

To measure large values of  $\operatorname{tg} \delta > 10^{-1}$  used  $q$ -meter VM-507 with a frequency error of 1% and an angle of  $\pm 2^\circ$ .

When numerical processing of measurement results, the calculation using equations (1)-(3) was performed in conjunction with the method of minimizing the comparison function (MCF) [22] and methods of statistical analysis [16, 24].

## 5. Conclusions

1. A compact device additional to the measuring unit (Figure 1) has been designed (Figure 2), which allows precision measurements of the density spectra of TSTD and  $\text{tg}\delta(v;T)$  in a wide range of temperatures  $T = 1 - 1550$  K and the strengths of the polarizing electric field  $E_0 = 100$  kV / m – 1 GW/m, at the frequencies of the field  $\nu = 50$  kHz – 35 MHz. The additional device allowed to expand the range of values of values  $3 \cdot 10^{-4} \leq \text{tg}\delta \leq 10^{-1}$ ,  $10^{-14} \leq C \leq 10^{-5}$ .

2. A measuring circuit for measuring currents  $I \geq 10^{-15}$  A was developed and tested in practice, including an electrometer B7-30, and the measurement  $\text{tg}\delta \geq 3 \cdot 10^{-4}$  was carried out using a q-meter VM-560. When measuring  $\text{tg}\delta \geq 0,1$  used the device VM-507.

3. Developed experimental methodology which will be used for further research based on the comparison of the theoretical (calculated) and experimental (measured) results by minimizing the comparison functions (MCF - method), with the aim of achieving a higher (compared to existing) degree of accuracy in the numerical optimization of parameter values (molecular characteristics) of microscopic structural defects in advanced composite electrical materials.

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