Experimental device for study of the thermal stimulated direct current in the composite materials

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Received: 10 September 2021 / Accepted: 30 June 2022 / Published online: 4 August 2022 © Akadémiai Kiadó, Budapest, Hungary 2022

Abstract
Presents the device for analysis the thermally stimulated electrical conductivity of composites in the wide temperature range. The main advantage of the module in relation to those described earlier is that it allows experiments both in the mode of measuring the current of thermally stimulated depolarization (TSDC) at low temperatures, and in the mode of measuring the thermally stimulated conductivity current (TSCC) up to very high temperatures. Using PMMA/PVDF mixtures as an example, it is shown that the TSDC method can be used to temperature transitions analysis in the many composite materials at low temperatures. It was also shown on the example of the two-component epoxy networks that the described module makes it possible to study the thermal transitions in the composites materials using the TSCC mode to the high temperatures.

Keywords Thermal transitions analysis · Thermally stimulated depolarization current · Thermally stimulated conductivity current

Introduction
The rapid development of nanoengineering leads to the development of different kind of composites with unique technological properties. This, in turn, stimulates the development and subsequent implementation of new experimental methods for measuring the properties of multicomponent composite materials (MCCM).

To predict new properties of materials, it is necessary to study information about the dynamics of thermo-molecular motion. Among the well-known methods for studying the dynamics of thermo-molecular motions, the method of the measuring of the thermally stimulated direct current techniques is widely used. Using of this high sensitivity technique allows to gain an understanding of the mechanisms of transition and relaxation and thermal processes, charge carrier generation, trapping, energy activation of the process of electrical conductivity, charge storage and transportation behavior and other related processes that take place in in the materials.

The thermally stimulated direct current techniques mentioned above can be divided into two classes. It is the thermally stimulated depolarization current (TSDC) method and the thermally stimulated conduction current (TSCC) method. Both methods are based on the same principle—measuring DC currents between the electrodes of an electrical capacitor, which heats up at a constant rate. An MCCM with previously measured geometric parameters is used as a dielectric in such a capacitor.

Recently, these methods are increasingly used to study the electrical properties of various composite materials [1–14].
The TSDC method consists in measuring the depolarization nanocurrents flowing between the capacitor parallel electrodes through a dielectric that was pre-polarized in a powerful electric field and then heated at a constant rate from the temperature of liquid nitrogen to complete depolarization of the sample. In the process of heating, they are sequentially defrosting and depolarization of the elements of the structure of the MCCM occurs. The currents TSDC is recorded as a function of temperature, and the relaxation processes can be seen as peaks in TSDC curves. The position and intensity of the peaks on the curves characterize the molecular mobility in the composites. TSDC technique is used to study the molecular behavior of a wide class of composites based on both low- and high-molecular compounds [1–5, 10–14].

The TSCC method is based on measuring leakage nanocurrents through a capacitor connected to a DC current source. By the magnitude of losses and their temperature dependence, they characterize the quality of the investigated composite.

It has recently been discovered that MCCM are effective and innovative materials in the novel nanotechnologies for the manufacture of lithium-ion batteries [15], for composite films with superior energy storage performance when used in electrostatic capacitors [16], for enhancing electrochemical properties of the graphite anodes [17] and as polymer composite electrolytes for solar cells [18], as elements of nanoelectronics for controlling processes in antibacterial and cryochambers, in particular, in low-temperature vessels for storing vaccines against COVID 19 etc. [19, 20]. This has led to the growing interest of scientists in the development of methods for producing MCCM and the study of their electrical and dielectric properties [21–25].

In this article, we present the experimental equipment developed by us for the study of composite materials by methods TSDC and TSCC in a wide temperature range, as well as some results of the thermal analysis obtained using this equipment.

**Experimental technique for conductivity characterization**

A measuring module has been developed and manufactured at the scientific institute of physics, which allows you to investigate electrical conductivity by TSDC and TSCC methods in a wide range of temperatures (Fig. 1).

The measuring module does not contain materials that become fragile at low temperatures which under experimental conditions may approach the boiling point of liquid nitrogen (≈−196°C). Therefore, it is possible to study organic / inorganic composite materials of different nature in TSDC mode from temperature 77 K. On the other hand, the use of high-temperature radio-ceramic as thermal insulators for electrodes, and thermocouples in a ceramics tubes allows to study inorganic MCCMs in the TSCC mode in the temperature range of 77–1300 K. To ensure the stability and the reliability of contacts of the module at high temperatures, a point electrical weld under a microscope, instead of soldering, of stainless steel wires to the electrodes of the measuring module was used. The electrodes, between which the investigated organic or inorganic composite was placed,

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Fig.1  a General view of the measuring module. 1–electrodes, 2–thermal insulation ceramic tubes, 3–heating furnace. b The measuring module with the closed heating furnace. c The measuring module with the thermos with liquid nitrogen
were made of brass and had a round shape with a diameter of 2 cm. The junction of the thermocouple was located near the upper electrode (see Fig. 2a, b). The studied sample was hermetically kept between the electrodes inside the furnace. The electrical circuits for connecting the module are shown in Fig. 2.

To polarize the sample in an electric field in TSDS mode (Fig. 2a) the parallel electrodes, between which is placed the sample connected to the source of the DC high voltage U (to avoid electrical breakdown—no more than 400 V per mm of the sample thickness). Then, with the holding high voltage U on the plates of the electric capacitor the measuring module with the sample is placed in a liquid nitrogen (see Fig. 1c). Online control of the temperature is carried out through an analog-to-digital converter (ADC) using thermocouple. After reaching the minimum temperature, the DC source is turn off, electrodes of the electrical capacitor is shorted then turn up the measuring circuit the highly sensitive nanoelectrometer (nA) as shown in Fig. 2b. After that, the sample is heated at a speed of about 3 degrees per minute for registration of depolarization currents. The electrical connection scheme in the measurement regime by the method TSCC is shown on the Fig. 2c. In contrast to the TSDC mode, in the TSCC mode the temperature dependence of the direct conductivity current through the studied composite is measured. To do this, the sample is constantly under the influence of a constant voltage U. The temperature of the sample increases at the same rate as in the method TSDC. The scheme of TSCC equipment is shown in Fig. 2c.

**Study of the thermally stimulated depolarization current in composites based on poly(methyl methacrylate) (PMMA) and poly(vinylidene fluoride) (PVDF)**

In polymeric materials, the cause of the appearance of conductivity when heated frozen material is the defrosting of the segmental mobility of certain atomic groups (so called α-, β-relaxations etc.). In this case, the moment of defrosting mobility (relaxation temperature) can be accurately determined using TSDS/ TSCC techniques.

The samples PMMA and PVDF and their blends with different ratio of PMMA to PVDF were obtained by using a twin screw extruder with the temperatures maintained at 473 K [26]. The samples were compression-molded at a temperature of 473 K and 30 MPa pressure during 10 min to form the disk-like samples and subsequent cooling of the press form to room temperature in the air on a table top. The polarization of the samples was performed according to the scheme shown in Fig. 2a. TSDC measurements were performed according to scheme shown in Fig. 2b.

In PMMA samples, by the methods of isothermal dielectric relaxation spectroscopy, low frequency mechanical spectroscopy and dynamic rheology, the authors of studies [24–29] found only one peak corresponding to the β relaxations of the segmental motions. In our measurements, due to the low heating rate (equivalent to a frequency of \(5 \times 10^{-2}\) Hz), we observed two peaks—one with a maximum at a temperature of 250 K and the other at a temperature of 265 K (Fig. 3a). In Ref. [3], it was noted that the β relaxation at about 260–280 K is usually ascribed to the rotational motions of the –OCOCH3– side groups of the PMMA. We suggest the other peak, at the lower temperature, 250K, should be ascribed to the β relaxation of the other, more mobile, –CH3– side groups in the chains of PMMA [28].

On the TSDC curve of the PVDF, we noted an intensive peak at the temperature 254 K, which can be ascribed...
to relaxations of the amorphous microphases within the crystalline phase $\alpha_c$ [29]. The more intensive peak, at the temperature 230 K, is usually ascribed to the cooperative $\alpha_a$ relaxation of amorphous segments in the PVDF. We discovered, also, a small peak at the temperature about 220 K, which we suggest to correspond to non-cooperative $\beta$ relaxation motions, partially hidden under the $\alpha_a$ relaxation peak (Fig. 3a).

In the PMMA/PVDF blends, researchers have been able to distinguish eight relaxations. They are: the $\alpha_a$ relaxation, which is associated with segmental motions in the PVDF, the $\alpha_c$ relaxation of segments within the PVDF crystalline phase, the $\alpha_m$ relaxation, which is attributed to the molecular motions in the miscible, amorphous PMMA/PVDF phase, the $\beta$ relaxation in PVDF, the two $\beta$ relaxations in PMMA and the high temperature $\alpha_a$ relaxation in the amorphous PMMA [24–30]. In the temperatures range used here for the TSDC measurements for the pure components and the blends, we found only five of the low temperature relaxations mentioned above: the $\alpha_c$ and $\alpha$ relaxation in the PVDF, $\alpha_m$ relaxation, in the PMMA/PVDF phase and the $\beta$ relaxations in each of the components. The high temperature $\alpha_a$ relaxation in the PMMA we will discuss below based on the results of the DRS.

On the curves of the blends PMMA/PVDF 4/1 and PMMA/PVDF 3/1 (see Fig. 3b), we can see inflections (about 220 K) at the temperature of the $\beta$ relaxation in component PVDF of the all blends. In our opinion, both peaks of the $\beta$ relaxation in the PMMA for the blend PMMA/PVDF 4/1 shifted to lower temperatures—one to the temperature 249 K and other to the temperature 261 K. For the 3/1 ratio of the components these peaks also shifted to lower temperatures—one to temperature 245 K and other to temperature 260 K. The shifts of the maxima of the $\beta$ relaxation peaks of PMMA component on the TSDC curves of the blends, compared to neat PMMA, show the nanoscale miscibility of the chain segments of the different components of the blends [31, 32]. Moreover, in the blend PMMA/PVDF 3/1, there appeared the additional $\alpha_m$ relaxation (mentioned above), which is attributed to the molecular motions in the miscible amorphous PMMA/PVDF phase, at the temperature 252 K (see vertical dashed line on Fig. 3b). These facts may be considered as further experimental evidence for nanoscale composition homogenieties originating from strong, specific interactions between PMMA and PVDF.

Consequently, the presence of many relaxation multi-peaks of depolarization and the offset of maxima of relaxation peaks on the TSDC curves of the samples of PMMA/PVDF shows a nanoscale mixture of chain segments of mixtures components.

Thus, on the example of PMMA/PVDF mixtures, it is shown that by the TSDC method can be tested the many composite materials in terms of the prospects for their use as the elements of microelectronics in the low temperatures area. Also this mode can help with the definition of methods for producing composites with the necessary electrical properties in the field of low temperatures.

Study of the thermally stimulated conductivity current in composites based on two-component epoxy networks

Epoxy resins due to their good adhesion to most materials and chemical inertness are traditionally known as high-quality adhesives and sealants. Due to these properties, epoxy resins are widely used in fuel pipelines and containers for storage. Such objects need special measures of spark safety and protection against fire and explosion, first of all—protection against static electricity. In the recent work, we found that the thermophysical, mechanical
properties and thermal conductivity of the materials based on epoxy resins are mainly determined by the ratio of components in them [33]. It was shown that mechanical and thermophysical properties can be adjusted in wide limits (for example, the glass transition temperature is from 300 to 400 K). From a practical point of view, this is very important, but the questions of their electrical conductivity from the point of view of protection against static electricity remain open. It was of interest the study the electrical conductivity of the DC of these composites by the TSCC method since glass transition is the process of defrosting collective segmentation mobility in the material and can be associated with the transfer of an electric charge in it.

Two epoxy oligomers, i.e., diglycidyl ether of poly(propylene glycol) (DGEPPG) (from Aldrich) and diglycidyl ether of bisphenol-A (DGEBA) (Araldite MY 790-1 from Vantico Ltd.) are used. Hexahydrophthalic anhydride were supplied by Vantico Ltd. 2-Ethyl-4-methyl-imidazole (2,4-EMI or EMI) was purchased from Janssen Chimica, Belgium (purity higher than 99%). All reagents were used as received.

The compositions of the two-component epoxy networks, represented by the mole ratio of DGEPPG to DGEBA, are 0/100, 30/70, 50/50, 70/30, 90/10, and 100/0, respectively. For researches, round discopa-like samples with a diameter of about 1 mm were prepared. The measurements were carried out according to the electrical circuit shown in Fig. 2c.

In Fig. 4a shown the curves of electrical conductivity samples. The temperature dependences of the conduction of the DC were treated in terms of the Arrhenius equation:

\[ I_{dc} = I_0 e^{(-E_{act}/kT)} \]  

(1)

where \( I_0 \) is zero loss of electrical conductivity, \( E_{act} \) is energy activation of the process of electrical conductivity, \( k \) is Boltzmann constant, \( T \) is the temperature.

In Fig. 4b shown the electrical conductivity samples in the Arrhenius equation coordinates.

Experimental results of electrical conductivity are well consistent with the Arrhenius equation (Fig. 2b). The magnitudes \( E_{act}/k \) characterize the sensitivity of the conductivity material to change the temperature. As can be seen from Fig. 4, the conduction temperature began grows with increasing ratio of DGEPPG/DGEBA. This result is quite consistent with increasing the temperature of the glass transition obtained by us DSC and DMA (Fig. 5a) [33]. From a physical point of view, it is quite natural that the defrosting of collective mobility of the molecular segments in the material during glass transition is accompanied by a rapid increase in the transmission of an electric charge in it under the influence of external voltage from the power source U (see Fig. 2c).

In our previous work [33], it was shown that the improve in mechanical and thermophysical properties with an increase in the content of the DGEBA in composites based on epoxy resins is the result of an increase in the thermodynamic rigidity of the network of the chemical bonds. Therefore, to defrost the segmental mobility of epoxy composites with increasing DGEBA content, heating to higher temperatures is required. As a result, with increases of the content DGEBA the temperature of the beginning of electrical conductivity increases (Fig. 5a), also increases the value of the conductivity but decreases the sensitivity of electrical conductivity \( E_{act}/k \) (Fig. 5b).

Thus, this studies have shown that the described module using the TSCC method allows to explore MCCMS to study the composites with minimal electrical losses in the wide temperature interval up to high temperatures area and for create sensors and electronic control elements of technological processes with specified properties.
Conclusions

This article presents the device to study the electrical conductivity of composite materials into the wide area of temperatures. The use of a highly sensitive nanoelectrometer along with a multichannel and high-speed analog for a digital converter allows to obtain high-precision and reliable results and provide online control of the measurement process. The module provides the ability to place a measuring cell into a liquid nitrogen vessel. This allows experiments in the measurement mode of thermally stimulated depolarization current (TSDC). On the other hand, the use of high-temperature materials in device, as well as the use of innovative methods of microwelding contacts, provides the ability to carry out experiment in measurements mode of thermally stimulated conductivity current (TSCC) to very high temperatures.

On the example of PMMA/PVDF mixtures, it is shown that by the TSDC method can be tested the many composite materials in terms of the prospects for their use as the elements of microelectronics in the low temperatures area. Also this mode can help with the definition of methods for producing composites with the necessary electrical properties in the field of low temperatures. Also it was shown described module using the TSCC mode allows to explore MCCMs to determinate composites with minimal electrical losses in the high temperatures area and for create sensors and electronic control elements of technological processes with specified properties.

Author contributions Valery Korskanov and Olena Fesenko designed and directed the researches. Volodimir Dolgoshey, Ponomarenko Sergiy and Tamara Tsebrinko carried out experiments. Valery Korskanov, Oksana Budnyk and Olena Fesenko wrote the manuscript.

Funding This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sector.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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