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**PRIORITY DIRECTIONS
OF SCIENCE AND TECHNOLOGY
DEVELOPMENT**



**PROCEEDINGS OF XI INTERNATIONAL
SCIENTIFIC AND PRACTICAL CONFERENCE
JULY 11-13, 2021**

**KYIV
2021**

PRIORITY DIRECTIONS OF SCIENCE AND TECHNOLOGY DEVELOPMENT

Proceedings of XI International Scientific and Practical Conference

Kyiv, Ukraine

11-13 July 2021

Kyiv, Ukraine

2021

UDC 001.1

The 11th International scientific and practical conference “Priority directions of science and technology development” (July 11-13, 2021) SPC “Sci-conf.com.ua”, Kyiv, Ukraine. 2021. 755 p.

ISBN 978-966-8219-84-9

The recommended citation for this publication is:

Ivanov I. Analysis of the phaunistic composition of Ukraine // Priority directions of science and technology development. Proceedings of the 11th International scientific and practical conference. SPC “Sci-conf.com.ua”. Kyiv, Ukraine. 2021. Pp. 21-27. URL: <https://sci-conf.com.ua/xi-mezhdunarodnaya-nauchno-prakticheskaya-konferentsiya-priority-directions-of-science-and-technology-development-11-13-iyulya-2021-goda-kiev-ukraina-arhiv/>.

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ELECTRICAL CONDUCTIVITY IN CORONA POLED POLYVINYLIDENE FLUORIDE – LEAD TITANATE COMPOSITE

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Introduction. The search for new materials to manufacture piezoelectric and pyroelectric sensors and study of their properties is an important task in sensor electronics. We believe that polymer – ceramics ferroelectric composites could combine good mechanical properties of polymers [1] with high electroactivity of ceramics [2] in order to produce piezoelectric and pyroelectric sensors and actuators. Perspective components of such composites are polyvinylidene fluoride, a typical ferroelectric polymer, and lead titanate, a conventional ferroelectric ceramics.

Important stage in preparation of samples is poling, because it directly affects the magnitude of pyroelectric and piezoelectric coefficients. Conventional sandwich poling has a serious disadvantage of high breakdown probability. However, the destructive breakdown is limited in case of corona poling, extensively used in the field of electrets and ferroelectric polymers [3]. Electrical conductivity during poling must be as low as possible in order to make the conductivity component of the poling current much smaller than the polarization component of the current.

Aim. The aim of this work is to study electrical conductivity in polyvinylidene fluoride – lead titanate composite corona poled for the first time to polarize a polymer – ceramic composite with ceramic particles produced by the sol-gel technique [4]. The poling behavior of the composite is compared with that of PVDF matrix, while particular attention is paid to anomalous behavior of conductivity in both materials. This feature is analyzed and explained by trapping of charges injected from the virtual electrode during poling.

Materials and methods. The gel of lead titanate (LT) was prepared by the sol-gel process [4]. The powder has been produced by heat treatments of dried gel near the crystallization point of 500°C. Samples were kept at this temperature for 2 h. X-ray diffraction of the powder has shown the well define perovskite structure.

To prepare a composite with 0-3 connectivity, the ceramics powder, sieved at 30 µm, and PVDF powder were mixed with acetone added until a uniform slurry was formed. After drying, the solid mass was hot pressed to obtain composite films of about 100 µm thickness. Al electrode was deposited on one face of the samples by vacuum evaporation. Then the sample was placed in a corona triode with its free surface exposed to corona ions [5]. The control grid was made vibrating to measure the surface potential by Kelvin's method. The samples were poled at 120° C for 15 min at grid voltage of 4 kV and corona voltage of 15 kV followed by 30 min cooling under applied field. Additionally, I-V characteristics were measured at 30°C and the temperature dependence of the current was studied at the constant voltage of 4 kV.

Results and discussion. Usually, conductivity increases with temperature. However, an anomalous decrease of conductivity with temperature was observed in the composite (Fig. 1). The temperature dependence of the poling current at heating and cooling shows that after reaching a maximum at 75°C the conductivity decreases with temperature. It is remarkable that the cooling curve does not follow the heating one indicating that some irreversible changes occur. Anomalous decrease of conductivity during thermally stimulated corona poling was observed also in PVDF.

As seen from Fig. 2, Child's law, i.e. a quadratic $I(V)$ dependence being typical for injected and space charge limited currents (SCLC) [6] is observed at medium fields. However, at high fields the I-V characteristic becomes sub-linear in logarithmic coordinates, indicating that the SCLC model is not valid any more.

It is known that the total poling current density $i(t)$ in ferroelectrics has three components, namely the capacitive, polarization and conductive ones as follows,

$$i(t) = \varepsilon_0 \varepsilon \frac{dE}{dt} + \frac{dP}{dt} + gE \quad (1)$$

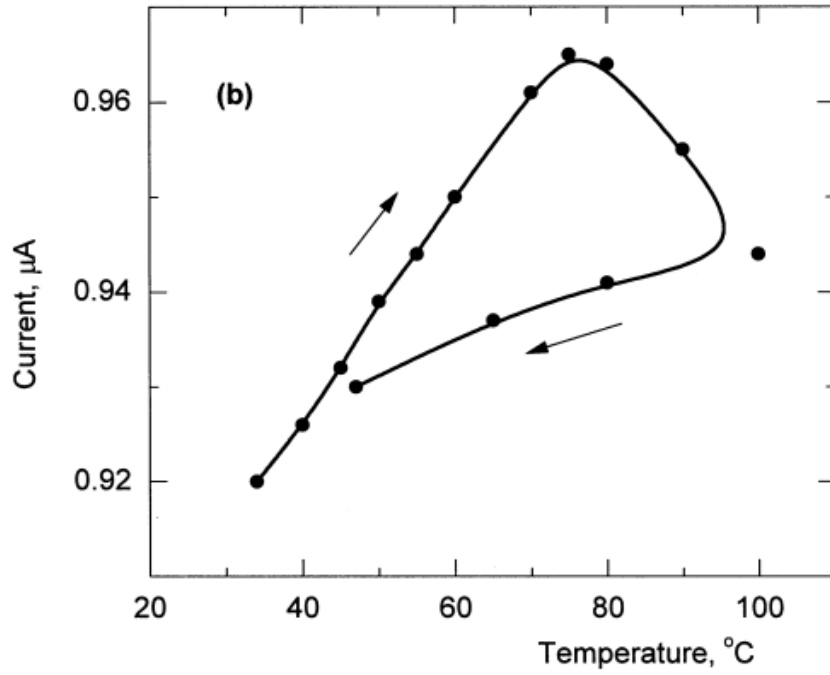


Fig. 1 Temperature dependence of the poling current during heating and cooling

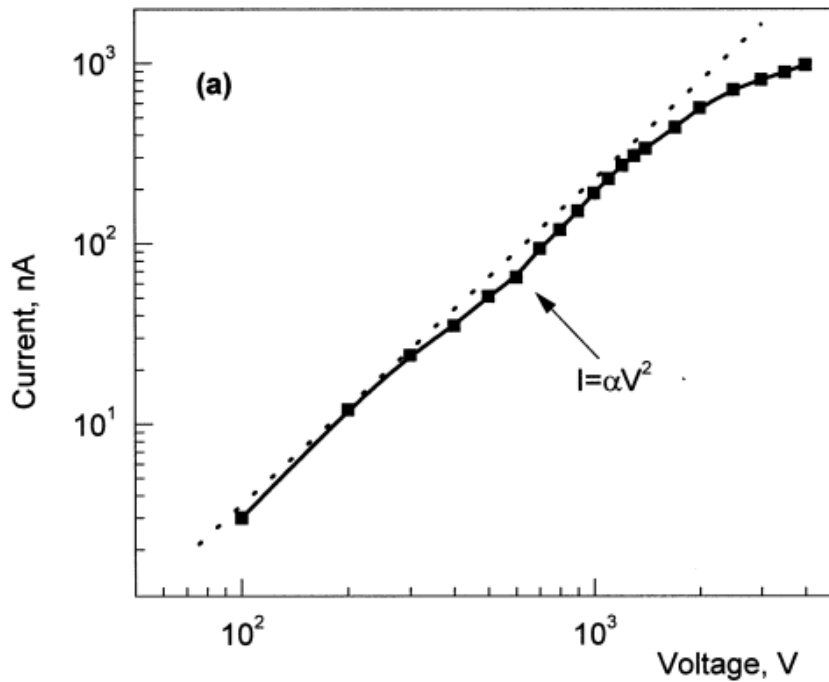


Fig. 2 The current – voltage characteristic of the composite

where ϵ_o , ϵ , E , and P are the permittivity of vacuum, the dielectric constant, the electric field strength and the ferroelectric polarization, respectively. The capacitive component represented by the first term at the right hand side of Eq. (1) can be

neglected, because constant field was applied in all experiments. In our case, only conduction current is shown in Fig. 1, because measurements were made after 30 min of voltage application, while polarization switching time was less than a second.

As clear from the super-linear I - V characteristic shown in Fig. 2, Ohm's law is not valid due to injection of free carriers in the bulk. Moreover, some carriers might be trapped. It is known that the ferroelectric polarization is stable if only the depolarizing field created by the bound polarization charges are compensated [7].

The decrease of conductivity during poling can be explained under assumption that the build-up of polarization is accompanied by the trapping of charge carriers. We have shown that favorable conditions for trapping exist at the boundaries of polarized ferroelectric particles due to the large-scale fluctuations of potential, caused by the alignment of dipoles [8]. The new state is stable, since the trapped charge provides compensation of the local depolarization field.

When poling is completed and the sample is short-circuited, the field everywhere in the bulk becomes zero due to redistribution of the mobile charges occurring with the characteristic Maxwell's relaxation time. The trapped charges reside in transition zones where polarization is non-uniform and $dP/dx = 0$. Thus, conditions for charge trapping are favorable in macroscopic transition zones separating polarized parts of the volume from non-polarized ones.

There must be sufficient density of free charges that can be trapped during poling. The charges can be either injected, as in case of corona poling, or emitted thermally inside the sample during high temperature poling. Although the thermal activation is not required for ferroelectric orientation and switching, it has been found experimentally that samples poled at high temperature show higher magnitude of the remanent polarization. This can be explained by the existence of thermally generated carriers acting as compensating charges after being trapped during poling.

There is another aspect in high temperature poling of composites. In order to be poled efficiently the field applied to ceramic particles must be at least of the order of the coercive field, i.e. 30 MV/m. The fields applied to LT and to polymer matrix are inversely proportional to the corresponding conductivities. Since at low fields,

conductivity of the polymer matrix is much lower than that of the composite, the great part of the field is applied to the polymer, but not to the ceramics particles. However, there will be two positive effects if one applies simultaneously the high field and high temperature. Conductivity of the polymer matrix will become higher, while conductivity of the ferroelectric filler will decrease drastically, as seen in Figs. 1 and 2, so the main part of the poling field will be applied to the ceramics particles.

Conclusions. Anomalous decrease of the apparent conductivity has been observed during corona poling of PVDF-LT composite representing heterogeneous ferroelectric polymers and polymer-ceramics composites. It has been shown that the phenomenon accompanies the build-up of the ferroelectric polarization in the material and explained by a massive trapping of charge carriers at boundaries of the ferroelectric species and at the transition zones where a gradient of polarization exists. The trapped charge contributes to stability of the ferroelectric polarization, since it compensates the depolarizing field.

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