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МАТЕРІАЛОЗНАВСТВО

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Influence Factors on Electrophysical Parameters of Composite Varistors

Purpose. The work is aimed to evaluate the influence of the structural state of polymer phase on the response voltage and nonlinearity coefficient of a multilayer varistor based on zinc oxide. **Methodology.** Zinc oxide consisted of 97% zinc oxide and 3% total oxides of Bi_2O_3, Co_3O_4, MnO_2, B_2O_3, SbO_3, ZrO_2, Al_2O_3. To obtain the composite of thermoplastic polymers and zinc oxide, non-polar and polar polymers, high pressure polyethylene and polyvinylidenefluoride were used. The composites were obtained by hot pressing at the melting temperature of the polymer phase and a pressure of 15 MPa. After that, using silver paste measuring electrodes 10 mm in diameter were applied to the surface of the synthesized samples and then current–voltage characteristics were measured. Modification of composites under the action of gas-discharge plasma was carried out in a special cell that creates a dielectric-gas-composite system. The composites structure was studied by X-ray diffraction analysis and IR spectroscopy. **Findings.** The obtained experimental results show that the size of inorganic phase particles significantly affects the current-voltage characteristics of the composite varistor: at a given thickness of the composite varistor, the operation voltage decreases markedly, and the nonlinearity coefficient increases. The influence of electric discharge plasma on the polymer zinc oxide-composite results in a significant change in the permittivity and the concentration of local levels at the separation boundary of the composite. The results research showed that electrical plasma effect on the response voltage depends on the polarity of polymer matrices. Moreover, plasma processing itself significantly changes the structure of the polymer phase in the composite. **Originality.** The magnitude of the potential barrier at the phase boundary is mainly determined by the volume fraction and size of the main structural element of ZnO ceramics. The change in the structural state of the polymer matrix allows adjusting the response voltage and nonlinearity coefficient of volt-ampere characteristic of the multilayer varistor. **Practical value.** The discovered development of electron-ion processes in the polymer phase of the varistor indicates the need of taking into account the change in its service characteristics from the duration and intensity of use. The result obtained shows not only the reason for the properties change, but also the need to develop the measures to increase the service life of varistor.

Keywords: amorphous state; boundary; phase; varistor; ceramic; electric current; conductivity

Introduction

It is known that ceramic semiconductor materials such as zinc oxide (ZnO) and silica carbide (SiC) are varistor materials. Numerous experimental results show that the formation of the varistor effect in these materials is directly related to the presence of a potential barrier at the boundaries of the crystallite-amorphous phase [4, 10, 11]. Such a structure of Zinc oxide and Silica carbide caused bipolar conductivity in these materials and the ability to sharply change the resistance at certain voltages. However, these materials in many cases do

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not meet the requirements of their practical application. It should be noted that the idea of developing a composite varistor comes from a model of the formation of the varistor effect in semiconductor ceramics. In composite varistor, a polymer matrix functions as an amorphous phase in ZnO or SiC ceramics; and as zinc oxide and silica carbide crystallites – microscopic particles from ZnO or SiC. This combination of components contributes to the formation of a potential barrier at the polymer-semiconductor ceramic interface, and hence the varistor effect. The purpose of this work is to reveal the formation features of the varistor effect in a strong heterogeneous system of polymer-semiconductor ceramics, taking into account the influence of individual phases on the varistor properties of this system. It is possible to represent the first phase as a crystallite, and the second – as inter crystallite boundaries. Transfer processes in amorphous-crystalline materials, in particular in composites, can be conventionally divided into two groups: through conduction carried out by the transition of electric charge carriers through the potential barrier of inter crystallite boundaries; conduction along polymer boundaries without participation of zinc oxide particles [1, 6].

Not so long ago, it was found that among the many known organic compounds and composites based on them, there is a small class of multiphase materials that have a very peculiar combination of properties: bipolar non-ohmic conductivity, heat resistance, fairly high mechanical and electrical strengths, and a symmetrical potential barrier at the phase boundary. Such substances, called active composites, include, in particular, varistor based on polymers dispersed by semiconductor ceramic particles ZnO [1–3]. A new class of composite elements was formed, the distinguishing feature of which is bipolar non-ohmic conductivity. Unlike traditional non-linear devices, they do not contain asymmetric potential barriers and current-voltage characteristics (CVC). The scope of these materials extends from nano-electronic, integrated circuits to low-power spark gaps (switching elements) for various purposes [6, 9–11]. The prospect of polymer composites with bipolar conductivity is due to both wide functionality (for example, in the function of energy-intensive and low-power varistor) and relatively simple and low-temperature manufacturing technology. The pronounced nonlinearity of conductivity and the symmetric nonlinear current-voltage characteristic of composites based on polymer-ZnO attract close attention as an active composite for the varistor. This allows changing nonlinearity coefficient of the current voltage characteristic, the opening voltage (Uop) (transition from linear to non-linear current-voltage characteristic) and operating temperature range in a wide interval. With the accumulation of experimental data, the model representation of the formation mechanism of the varistor effect and the conductivity of composites changed from a model in which the main role was attributed to polymer layers between ZnO particles to a model in which various grain boundary effects in the zinc oxide particle itself and the processes occurring on the boundary layers of the polymer phase with participation of the ZnO particle. At same time, the structural state of the ceramic composite has a fairly significant impact on the complex of technical and technological properties. For example, such a characteristic as an electric current leakage largely depends on the distribution uniformity of alloying elements, which in turn determines the height of the potential barrier [7, 8]. On the other hand, the observed sensitivity to the presence of oxygen atoms is a factor that has a certain effect on the dielectric permeability of ceramics. In fact, the detected dependence of the specified characteristic on the ratio of grain size to the thickness of the depleted layer is actually dependent on the oxygen absorption mechanism (Fig.1.) [12]. As follows from Fig. 1, the characteristic structure of a varistor is ZnO grains separated by thin films consisting of chemical compounds of alloying elements. In a more detailed analysis, after a certain treatment, one can detect dispersed precipitates of chemical compounds in the most defective places at structure – in the triple junctions of grains. Such precipitates of chemical compounds can affect the development of ZnO grain growth processes during high-temperature treatments during the formation of varistors.

Additional evidence of the importance of structural state of ceramics, especially dispersion of the phase components, are the results of works [8, 13].

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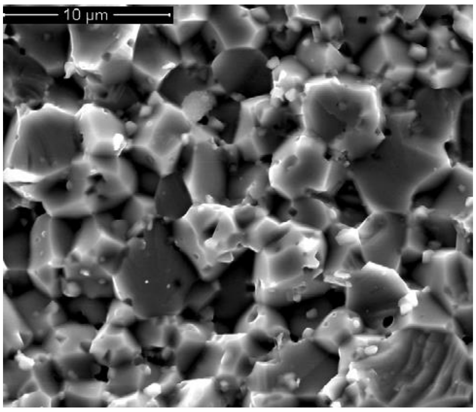

Fig. 1. The typical structure of the varistor [12]

Indeed, the observed low breakdown voltage values for varistor sintered at higher temperatures are due to grain growth and a decrease in the number of grain boundaries between the inner electrodes. In addition to the number of boundaries between phases, the layer thickness acquires a certain value. Based on this, a layer thickness increase should contribute to an increase in the breakdown voltage [8]. Another problem of multiphase varistor is the leakage current. The selection of the optimal structural state of the ceramics makes it possible to effectively decrease it. An example is the stability increase of secondary spinel particles of $Zn_{2.33}Sb_{0.67}O_4$ during cooling of ceramics. Another factor of structure state optimization is location of insulating phase between the skeleton of bismuth-containing phases, which helps to reduce the varistor current leakage by about two orders of magnitude [9]. As in [8, 11, 13], in [5, 12] the importance of the dispersion of structural components is emphasized. Thus, for ceramics consisting of the main ZnO phase and oxides of rare earth elements (Dy_2O_3, Pr_6O_{11}, Pr_2O_3), the grain size significantly depends on the sintering temperature. A sintering temperature increase by only 200°C is accompanied by the grain size increase up to 6 times and nonlinearity coefficient decrease by 2–2.5 times. Based on the analysis of the obtained results, it can be assumed that the most common model of a highly heterogeneous structure is a polymer-semiconductor ceramic composite. The size of the zinc oxide particles should provide a certain ratio of contact area of the polymer to the volume of the ZnO particle. It is obvious that zinc oxide particles appear as the crystalline phase and the polymer is used as the amorphous phase.

Purpose

Influence evaluation of structural state of the polymer phase on the response voltage and nonlinearity coefficient of a multilayer varistor based on zinc oxide.

Methodology

Zinc oxide used as a dispersant was obtained as follows: the composition of semiconductor ceramics was chosen – 97 mol% zinc oxide and 3 mol%, oxides Bi_2O_3, Co_3O_4, MnO_2, B_2O_3, SbO_3, ZrO_2, Al_2O_3 as an alloying component of molecular weight 0, 96% used ZrO_2 or Al_2O_3 based on these compositions at a temperature of 1573°K, semiconductor ceramics ZnO [3] was synthesized. The zinc oxide powder with a particle size of ≤ 60 μm was prepared from the mixture obtained by milling. Composites based on thermoplastic polymers and the zinc oxide were obtained in the following way: non-polar and polar polymers, high-pressure polyethylene (PE) and polyvinylidene fluoride (F2M), respectively, were used as polymers. The composites were obtained by hot pressing at the melting temperature of the polymer phase and pressure of 15 MPa [1, 6]. After that, using silver paste, measuring electrodes 10 mm in diameter were deposited on the surface of the synthesized samples, and then the current-voltage characteristics were measured. Modification of composites under the action of gas discharge plasma was carried out in a special cell that creates a dielectric-gas-composite system [2]. Of particular importance is the determination of the electrical strength of composite varistors. The article determined the instantaneous breakdown voltage. The number of samples used in the test is at least 10. To determine the instantaneous breakdown voltage, a UPU-10 device was used. The cell used for testing is shown in Fig. 2.

The electrodes and their configuration correspond to known conditions. The diameter of the high-voltage electrode is approximately 3 times smaller than the diameter of the ground electrode. The electrodes are made in the form of a Ragowski electrode and ensure the uniformity of the electric field in the high-voltage electrode-composite-grounded electrode system. The structure of the composites was studied using the methods of X-ray diffraction analysis and IR spectroscopy.

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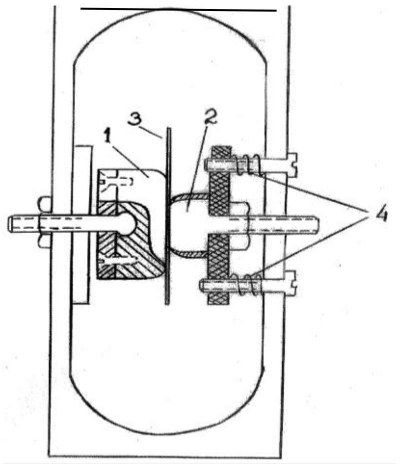

Fig. 2. Device for determining the electrical strength of samples (*1* – ground electrode, *2* – high voltage electrode, *3* – sample, *4* – compression spring)

Discussion of experimental results

To reveal the role of the polymer phase in the formation of the varistor effect and to determine the effect of the polymer matrix on the parameters of the composite varistor, the properties of the composite varistor were studied using polymers of different structure and polarity.

Table show the current-voltage characteristics and the nonlinearity coefficient of composites based on non-polar (PE) and polar (F2M) (modified polyvinylidene fluoride) polymers and zinc oxide. The results obtained show that the polymer matrix significantly affects the response voltage of the composite varistor. The same results were obtained for these composites when comparing their coefficient of nonlinearity of the current-voltage characteristics. In the first approximation, we believe that changes in U_{op} and β depending on the structure and properties (polarity) are associated with interfacial processes at the polymer-ZnO interface.

Table. 1

Opening voltage and nonlinearity coefficient of ZnO–PE and F2M composite varistors

N	ZnO (with admixture of Al_2O_3) + PE			N	ZnO (with admixture of Al_2O_3) + F2M		
	Volume percent of samples	U_{op}, V	β		Volume percent of samples	U_{op}V	β
1	30%C + 70% PE	140	4,86	4	30%C + 70% F2M	140	3
2	50%C + 50% PE	120	7,6	5	50%C + 50% F2M	132	5
3	60%C + 40% PE	117	8,7	6	60%C + 40% F2M	120	6,4

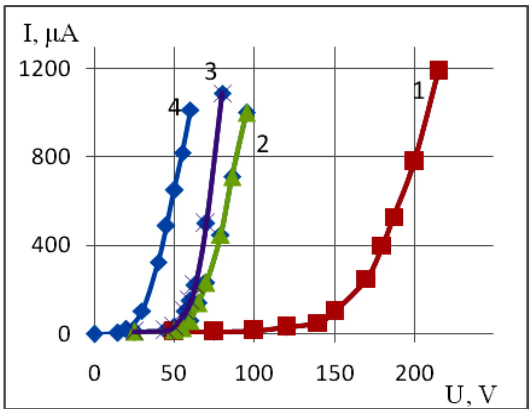

Fig. 3. Grain size influence of the matrix (*1* – 63, *2* – 100, *3* – 200, *4* –315 μm) on the current-voltage characteristic of a composite based on polyethylene 30% C + 70% PE

It is known that interfacial interactions significantly depend on the size of the inorganic phase of the composites [1, 3]. Therefore, for composites based on polyethylene, the current-voltage characteristics were studied depending on the size (d) of Zinc oxide particles (Fig. 3). The obtained experimental results show that the size of the particles of the inorganic phase significantly affects the CVC of the composite varistor. At certain (constant) thickness of the composite varistor, the operation voltage decreases markedly, and the nonlinearity coefficient increases. Numerous experimental results obtained by us show that the impact of electric discharge plasma on the polymer-zinc oxide composite leads to a significant change in the per-

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mittivity and the concentration of local levels at the interface of the composite [1–3].

Fig. 4 shows the dependence of the dielectric permittivity on the volume content of Zinc oxide for samples treated under the action of electric discharge plasma. It can be seen that for all volumetric contents, the treatment with electric discharge plasma leads to a noticeable increase in ε. This parameter increases with increasing duration of the discharge. Fig. 5 shows the changes in the coefficient of nonlinearity (β) of the I–V characteristics of the composites before and after their treatment under the action of a discharge for various volumetric contents of the ZnO phase. It can be seen that plasma treatment significantly increases the values of β.

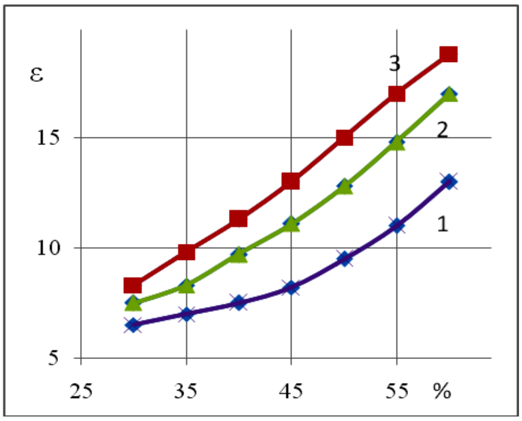

Fig. 4. Dependence of dielectric constant on the volume of content filler in the ZnO – high-pressure polyethylene composite and treatment by electric discharge (*1* – untreated, *2* – treated for 3 min, *3* – for 10 min)

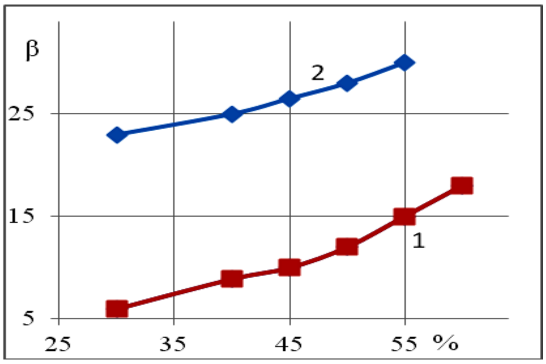

Fig. 5. Dependence of nonlinearity coefficient on the volume content of filler in the ZnO – high-pressure polyethylene composite and treatment by electric discharge (*1* – untreated, *2* – treated for 3 minutes)

Such a change in ε and β testifies to the determining role of boundary effects in the formation of varistor properties of composites. This assumption is also confirmed by the results of changes in the operation voltage of varistor before and after their processing under the action of electric discharge plasma (Fig. 6 *a*, *b*). The figure also shows that the effect of electric discharge plasma on U_{op} depends on the polarity of the polymer matrices (high-pressure polyethylene and polyvinylidene fluoride). From the beginning, let us consider a possible change mechanism of U_{op} and β of composites after their modification under the action of electric discharge plasma. It is known that plasma treatment significantly changes the structure of the polymer phase of the composite (Fig. 6, *b*). It can be seen that intense oxidation of polymer chains occurs: CO, OH, COC, etc. appear. Based on the fundamental idea that any changes at structure are accompanied by the appearance of local levels in the quasi-forbidden band of the polymer phase, the shift of the Fermi level towards the conduction band is quite justified.

a

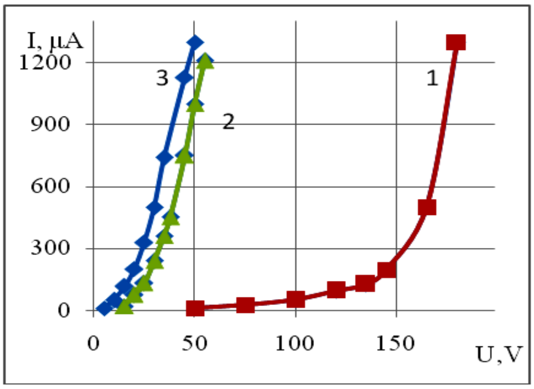

Fig. 6, *a*. The treatment effect of the electric discharge (*1* – without treatment, *2* – treatment for 3 min, *3* – 10 min) on the current-voltage characteristics composite 60% ZnO + 40% polyvinylidene fluoride

Based on this, it should be expected that structural changes in the polymer matrix will lead to a decrease in the response voltage and increase in nonlinearity coefficient of the current-voltage characteristic.

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b

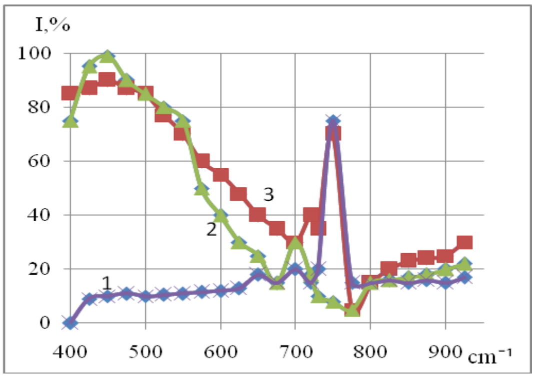

Fig. 6, *b*. Spectra of components and composite ceramics (*1*– 100% high-pressure polyethylene, *2* – 100% ZnO, *3* – 10% ZnO+90% of the high-pressure polyethylene) depending on the inverse value of the wave length (1/λ), where λ is the wave length of the spectral lines)

Some confirmation of this effect is the higher sensitivity of plasma modification of composites based on non-polar PE polymer (Figs. 6, *a* and 6, *b*). Comparison of the results of the change in the structure of the polymer phase after exposure to the electric discharge plasma shows that, all other things being equal (the value of the discharge power), the structure change degree in the case of composites with a polymer matrix of polyethylene is greater. This manifests itself in a relatively large change of opening voltage and coefficient nonlinearity.

Originality and practical value

The discovered development of electron-ion processes at polymer phase of the varistor indicates the need to take into account the change in its service characteristics from the duration and intensity of use. The result obtained has a certain practical significance, since it indicates not only the reason for the change in properties, but also the need to develop measures to increase the service life of the varistor.

Findings

1. The analysis of the results obtained indicates the dependence of operational characteristics of multilayer varistor on the development of the electron-ion exchange processes at polymer phase.
2. The magnitude of the potential barrier at the phase boundary is mainly determined by the volume fraction and size of the main structural element of ZnO ceramics.
3. Changing the structural state of the polymer matrix allows adjusting the response voltage and nonlinearity coefficient of volt-ampere characteristic of the multilayer varistor.

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Фактори впливу на електрофізичні параметри композитних варисторів

Мета. У роботі передбачено оцінити вплив структурного стану полімерної фази на напругу відбиття та коефіцієнт нелінійності багатошарового варистора на основі оксиду цинку. **Методика.** Оксид цинку складався з 97 % оксиду цинку і 3 % з оксидів Bi_2O_3, Co_3O_4, MnO_2, B_2O_3, SbO_3, ZrO_2, Al_2O_3. За температури 1 573 °C здійснено синтез напівпровідникової кераміки на основі оксиду цинку. Для отримання композиту з термопластичних полімерів та оксиду цинку використовували неполярні та полярні полімери, поліетилен високого тиску та полівініліденфторид. Композити отримували гарячим пресуванням за температури плавлення полімерної фази й тиску 15 МПа. Після цього, за допомогою срібної пасти на поверхню синтезованих зразків наносили вимірювальні електроди діаметром 10 мм, а потім вимірювали вольт-амперні характеристики. Модифікування композитів під дією газорозрядної плазми проводили в спеціальній комірці, яка створює систему діелектрик – газ – композит. Структуру композитів досліджували методами рентгенівського фазового аналізу та ІЧ-спектроскопії. **Результати.** Отримані експериментальні результати показують, що розмір частинок неорганічної фази суттєво впливає на вольт-амперні характеристики композитного варистора: за заданої товщини композитного варистора помітно зменшується робоча напруга, а коефіцієнт нелінійності зростає. Вплив плазми електричного розряду на полімерний композит оксид цинку призводить до суттєвої зміни діелектричної проникності та концентрації локальних рівнів на межі поділу композиту. Результати досліджень свідчать, що вплив електричної плазми на напругу відбиття залежить від полярності полімерних матриць. Крім того, сама плазмова обробка суттєво змінює структуру полімерної фази в композиті. **Наукова новизна.** Величину потенційного бар'єру на межі поділу фаз в основному визначають об'ємною часткою і розмірами основного структурного елемента кераміки ZnO. Зміна структурного стану полімерної матриці дозволяє регулювати напругу відбиття та коефіцієнт нелінійності вольт-амперної характеристики багатошарового варистора. **Практична значимість.** Виявлений розвиток електронно-іонних процесів у полімерній фазі варистора свідчить про необхідність урахування зміни його службових характе-

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ристик від тривалості та інтенсивності використання. Отриманий результат вказує не тільки на причину зміни властивостей, але й на необхідність розробки заходів для збільшення терміну служби варистора.

Ключові слова: аморфний стан; межа; фаза; варистор; кераміка; електричний струм; провідність

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