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EFFECT Of ELECTROTHERMOPOLARIZATION ON THE ELECTROPHYSICAL PROPERTIES AND STRUCTURE Of HDPE/HfO_2 – BASED POLYMER NANOCOMPOSITES

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ABSTRACT

This study investigates the effect of electrothermopolarization (ETP) on the structural and electrophysical properties of polymer nanocomposites based on HDPE and hafnium oxide (HfO_2) nanoparticles. We analyze the interactions between the polymer matrix and the nanoparticles, detailing structural changes within the bulk and the interphase zones both before and after the ETP process. Scanning Electron Microscopy (SEM) reveals a diverse morphology, ranging from spherical to irregular shapes, highlighting the distribution of HfO_2 within the matrix. Furthermore, the impact of HfO_2 on the polymer's crystalline and amorphous phases was characterized using X-ray diffraction (XRD) and Differential Scanning Calorimetry (DSC). These results demonstrate that the filler significantly influences the supramolecular structure and thermal behavior of the HDPE, which in turn governs the stability of the electret state.

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1. INTRODUCTION

As it is known, the inclusion of nanoparticles in a small amount of polymer allows its physical properties to be improved, its life to be extended. The properties of modified polymer nanocomposites are related to the compatibility and distribution of their components. Meanwhile small amount of nanoparticles acts as a crystallization center. As the amount of nanoparticles increases they accumulate in the amorphous part of the polymer, which leads to changing amorphous and crystalline states in the polymer to create a separate structure in the polymer environment [1, 2].

It should be noted that depending on the nature, size and distribution of nanoparticles, the resulting polymer nanocomposite can be electroconductive, antistatic or dielectric. In this connection the study of the nanocomposite properties obtained by adding metal oxide nanoparticles to polyethylene is of particular interest [3, 4]. The change in the physical properties of polyethylene materials by nanoparticles is mainly due to two reasons: nanoparticles act as a source of structuring and the nanocomposite interphase layer has a large number of charge traps with different activation energy. This work deals with the polymers asa high-density polyethylene (HDPE) matrix and (HfO_2) hafnium oxide nanoparticles, having improved physical parameters, such as high chemical and thermal stability, large band gap, high permittivity as well. We suppose that these distinctive properties of HfO_2content should be reflected in the properties of HDPE+HfO_2modified polymer nanocomposites. Metal oxide nanoparticles play a very important role in many areas of physics and material science [5, 6].

In terms of PE electrical properties as a non-polar polymer is a high-quality, high-frequency dielectric; permittivity and dielectric loss tangent vary only slightly under the changes in the electric field frequency, temperature within minus 80°C up to 100°C and humidity. However, catalyst residues in HDPE increase the dielectric loss tangent, especially with the temperature change, which leads to some deterioration of insulating properties. Therefore to improve both the structural, electrostatic and physical properties of polyethylene, we use hafnium metal nanooxide as an addition.

In modern literature, despite the large number of published works on materials based on hafnium nanooxide, HDPE/HfO_2 – based nanocomposites have not been practically studied. HfO_2 hafnium dioxide properties determine its use in many fields of science and technology. Thus in microelectronics, due to its wide band gap, high permittivity, and low leakage currents, HfO_2 is considered to be alternative dielectric to replace the traditionally used silicon dioxide SiO_2. HfO_2 is also used as a surface coating for optical fibers and dielectric mirrors of ultraviolet lasers.

Our research is dedicated to the change in HDPE/HfO_2 nanocomposite structure, dielectric, electret and thermal properties under the influence of ETP process as a result of polarization processes occurring at the interface. Modified polymer nanocomposite dielectric properties are closely related to the behavior at the interface between nanoparticles and polymer matrix [7, 8]. Many polymer nanocomposites based on metal oxide nanoparticles have a high permittivity and thus a huge potential for using in modern microelectronics to develop new capacitors for energy storage, gates in MOSFETs, memory cells, supercapacitors and etc. [9].

As it is known polymers and nanocomposite materials obtained on their basis after exposure to external constant electric field at sufficiently high temperatures are polarized and have electret properties. The main characteristics of the electret material are the magnitude of electric charge and its stability. To meet the need for electrets with these characteristics more sophisticated and comprehensive study of their thermal properties is required [10-12]. Recently the acquisition and study of new nanocomposite materials by introducing nanoparticles into both low-density and high-density polyethylene yield better results (DSC, SEM) that are of particular importance from practical and scientific standpoint.

1. EXPERIMENTAL PART:

1.1. Materials

HDPE polyethylene granules (SOCAR, "Azerikimya" Production Union "Etilen-Polyethylene" plant, 15803-020), CCl_4 organic solvent (Code 141245, 99.5%, CAS Common Chemistry-P.L.C); hafnium oxide (HfO_2) nanoparticles, size 10-20nm, Luoyang Tongrun Info Technology Co., Ltd. China, CAS 12055-23-1, 99%).

1.2. Synthesis of polymer nanocomposites

HDPE/HfO_2 polymer nanocomposites have been obtained as follows: polyethylene granules are dissolved in 60 ml of organic solvent as carbon tetrachloride (CCl_4)at T=70°C. HfO_2 nanoparticles are added to the polymer solution of various content and mixed for 8 hours until the homogeneous mixture is prepared. The mixture is transferred to Petri dish and dried in the vacuum oven for 24 hours. Then nanocomposite thin films are produced from these samples by hot pressing at polyethylene melting temperature and pressure 15MPa for 10 min. After hot pressing the films are cooled in water. Cooling rate of nanocomposite films is 20deg/min. Film thicknesses is 90–105 μm (Fig 1).

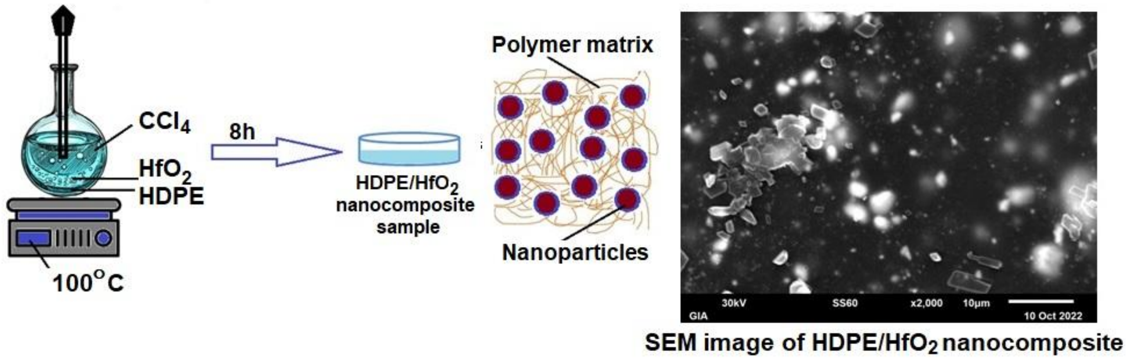

Fig.1 *Synthesis scheme of HDPE/HfO₂ nanocomposites*

1.3. Nanocomposite Characterization

X-ray diffraction studies of nanocomposites have been performed on D2 Phase X-ray diffractometer in reflection mode (Bragg-Brentano geometry) using Cu-kα radiation (average wavelength λ=1.5406Å, nickel β-filter). Diffraction pattern registration is carried out in continuous mode in the range of angles 2θ = 5–80°.

SEM analysis of nanocomposites is made on scanning electron microscope JEOL JSM 6610-LV at accelerating voltage 30 kV.

Polymer nanocomposites undergo electrothermopolarizaton (ETP) by using external electric field strength $7\cdot10^6 \div 12\cdot10^6$V/m at T=100°C and then cooling down to room temperature for 1 hour. Electret characteristics have been measured on EFPM-1 device.

Differential scanning calorimeter (DSC) data are obtained on heat flux calorimeter NETZSCH DSC 204 F1 Phoenix by analyzing melting and crystallization thermograms. The investigations are carried out with heating rate 10 deg/min at T=20÷180°C. Experimentally obtained DSC curves represent the dependence of heat flux (mJ/s) on temperature. Heating in nitrogen atmosphere at the predetermined rate according to the controlled program is taken place and the heat flows of the reference and the material under consideration are compared during the measurement process.

3. RESULTS AND DISCUSSION

The following mechanisms can be used to improve the electret properties of nanocomposites: 1. Change in supramolecular structure due to the addition of HfO_2 nanoparticles to HDPE, as well as the appearance of various local centers of polarized and specific charges; 2. Oxidation of free radicals and polymer chains in nanocomposites obtained by the method of hot pressing at high temperature (close to melting temperature of matrix). Oxidative-destructive processes may at times break the non-polarity of polymer matrix. When oxygen-binding groups are formed in the polymer chain along the macromolecule, polar regions arise, which in turn leads to the formation of dipole-orientational polarization to improve nanocomposite electret properties. This mechanism is connected with the migration polarization of HDPE/%wt. HfO_2 heterogeneous system. It determines the existence of electrets over a period of months and even years.

While investigating electret properties of polymer nanocomposites [13-15,21-23] one can conclude that there is a very broad relationship between the physical and chemical structure of polymers and the size of collected charges. It is shown that the change of polymer electret properties is primarily influenced by trap structures. Therefore the stability and amount of charges captured by the traps are largely defined by the polymer supramolecular structure peculiarities.

The crystal structure, size and orientation of HfO_2nanoparticles and HDPE (Fig. 2.a) are investigated by X-ray diffraction (XRD) method. X-ray diffraction pattern shows that the nanoparticles have a polycrystalline monoclinic phase. The observed data are in good agreement with the standard JCPDS card no: 06-0318. In Fig. 2.b there have been shown X-ray diffraction spectra of HDPE and PE/HfO_2 - based polymer nanocomposites. Analysis of X-ray diffraction spectra reveals (Fig. 2.b) that the partial narrowing of diffraction peak half-width due to hafnium oxide (HfO_2) may be associated with the increase ofbond intensity in final nanocomposites [16-20,24].

The nanocomposite matrix demonstrates that X-ray diffraction peak intensity increases as HfO_2 nanoparticles are incorporated into HDPE matrix.

X-ray spectrum of HDPE/HfO_2 nanocomposite shows that as the concentration of HfO_2 increases, peaks are observed in X – ray spectrum at an angle of 2θfor HDPE:21,59° (110), 23,98° (200); HDPE/3%HfO_2:28°(111),31,89° (111), 34,93° (020), 36° (200), 41,06°(102), 49,97°(022),50,82° (220), 54,69° (300), 56° (221); HDPE/5%HfO_2:28,6° (111), 31,5° (111), 34,48° (020), 35,5° (200), 41° (102), 49,6° (022); 50,5° (220), 54° (300), 55,89° (211), 60° (131), 63,3°(113) 66°(331); HDPE/7%HfO_2:24°(011), 28,63° (111), 31,7° (111), 34,5° (020), 35,6°(200)38,9° (021), 41,2° (102), 49,7°(022), 50,7° (220), 54,6° (300), 56° (221), 60,5°(131), 63°(113), 66°(331)respectively, that assumes the typical structure of orthorhombic HfO_2. Sharp and very intense X-ray peaks indicate that the materials under investigation are the nanocomposites characterized by HDPE semicrystalline structure.

The average particle size of resulting HDPE/HfO_2 nanocomposites is calculated using Debye-Scherrer equation. Debye-Scherrer equation is the most simple way to determine particle size:

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (3)$$

where D is the particle size, λ = 1.5406 Å, β is the peak width at half maximum, θ is the diffraction angle (in radians), K is the constant approximately equal to 0.9. With increasing filler content, the diffraction intensity of HDPE/HfO_2 phases decreases.

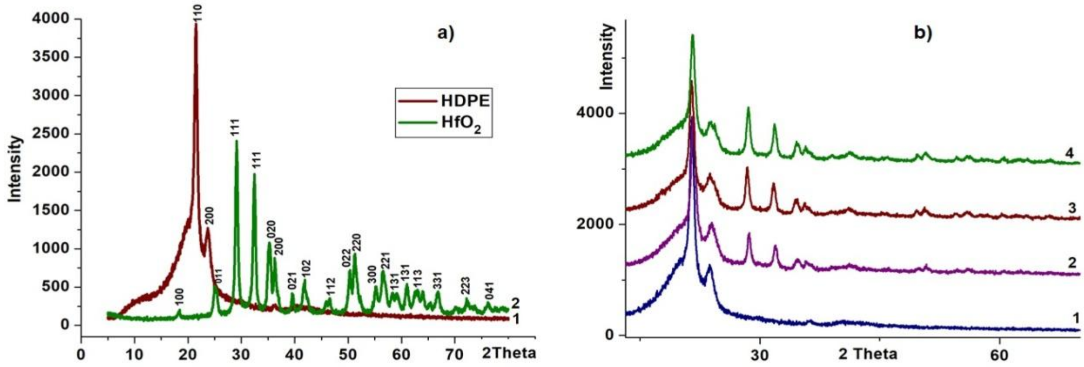

Fig. 2 *XRD patterns of HfO_2 nanoparticle in powder form, HDPE (a) and XRD patterns of nanocomposites HDPE/%wt. HfO_2 (b): 1. HDPE; 2. HDPE/3%HfO_2; 3. HDPE/5%HfO_2; 4. HDPE/7%HfO_2*

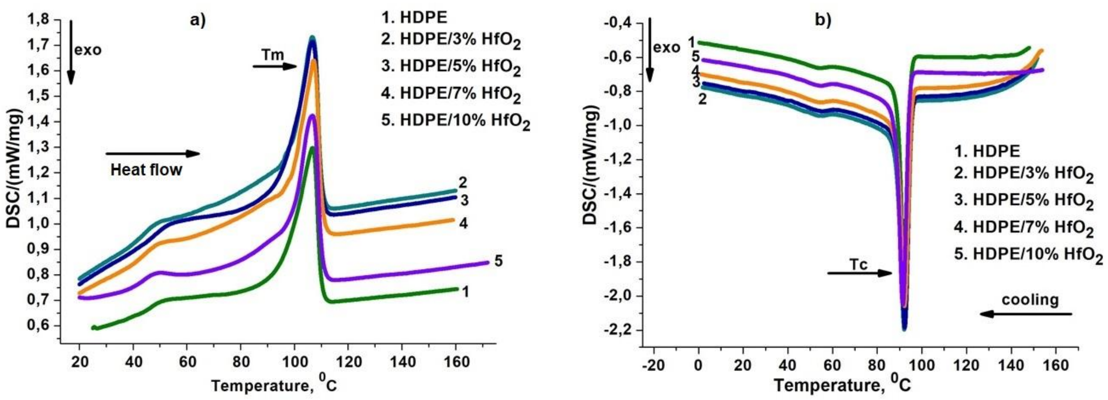

Fig.3 *DSC curves heat flow (a) and cooling (b) of pure HDPE, HDPE/HfO_2 nanocomposites*

HfO_2 metal oxide-containing nanoparticle used in this work is located in the interphase layer of HDPE structural elements and causes the formation of heterogeneous centers of crystal formation in the nanocomposite alloy that leads to the growth of the crystallization center during the nanocomposite cooling process [25-34]. To determine the thermal stability of HDPE/HfO_2-based polymer nanocomposites, the tests are carried out using the differential scanning calorimetry (DSC) method. In Fig.4 and Fig.5 there has been shown the thermal stability of nanocomposite materials of polyethylene and HDPE/HfO_2 nanocomposites depending on HfO_2 particle concentration. These maxima can break old bonds and form new ones, as well as can change the molecular mass in HDPE/HfO_2 nanocomposites. This means that the interaction of polymer matrices with the nanoparticle charged surface leads to the formation of polymer nanoparticle nearby the nanoparticle surface and it is this interfacial nanoparticle that determines the maxima.

Having determined the fusion heat it is possible to calculate the degree of crystallinity of the nanocomposite under study (Table 1). Since the cooling process in the calorimeter is slow, the amount of crystalline phase formed in this case is significantly less than in the process of starting material production [35-37]. It should be noted that there is a slight decrease in the melting temperature of the polymer nanocomposite, which can be explained by partial decrease in the chain length and a greater mobility of the polymer molecules, respectively.

Table 1. *Results of DSC analysis of heat flow and cooling*

Sample	Tm, °C	Tc, °C	Melting enthalpy, ΔH, J/g	Degree of nanocomposite crystallinity, %
HDPE	107,8	92	39,47	31,57
97%HDPE/3%HfO_2	106,3	92,1	50,24	36,38
95%HDPE/5%HfO_2	106,5	92,3	44,48	36,44
93%HDPE/7%HfO_2	107,7	92,3	46,16	40,76
90%HDPE/10%HfO_2	106,8	91,5	46,59	31,57

As the mobility of polymer chains increases, the melting temperature decreases that is observed in the experiment. The observed decrease is slight and should not lead to significant change in polyethylene physical properties. For HDPE/HfO_2 - based polymer nanocomposites the degree of crystallization at all HfO_2 nanoparticle concentration in the polymer increases and reaches maximum value at 7%wt. In Table 1 there have been presented the results of DSC analysis of experimental samples. The obtained data can be explained by the fact that the filler particles are distributed throughout the matrix and can be heterogeneous crystallization centers. Given results are confirmed by SEM spectra.

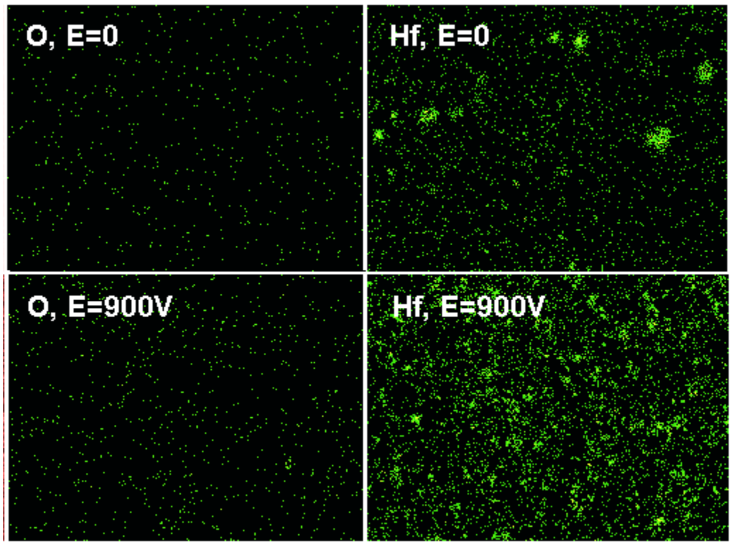

Fig.4 *SEM images of elemental distribution in HDPE/5%HfO_2 nanocomposite before and after electrothermopolarization*

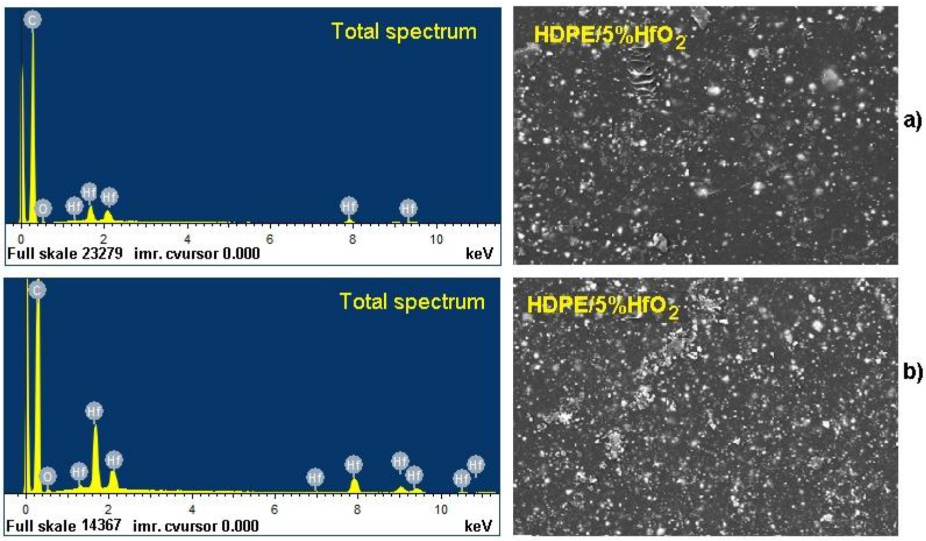

Fig.5*SEM-EDSimages of HDPE/5%HfO_2 polymer nanocomposite before (a) and after (b) ETP*

SEM and SEM-EDS analyses are used to characterize the surface morphology of HDPE/HfO_2 nanocomposites. In Fig.4 micrographs of HDPE/HfO_2 are shown. The surface morphology of nanocomposites before ETP is rough and phase-separated. After ETP the nanocomposite surface has a smooth and controlled morphology [38-43].

The morphology of samples and distribution of HfO2 nanoparticles throughout the volume of the polymer matrix have been studied by scanning electron microscope (SEM) and energy dispersive spectrum (EDS) (Fig.5. a. b). It is also shown from EDS element analysis (Fig.5. a. b) that obtained nanoparticles are hafnium oxide ones. Fine crystalline particles can be observed on the surface in Fig.5. a.b. Based on SEM images, it can be stated that HfO2 nanoparticles are evenly distributed and aggregated the polymer matrix surface.

CONCLUSIONS

It is established that incorporating 3–10 wt.% of HfO_2 enhances the physical properties of the HDPE/HfO_2 nanocomposite by inducing significant structural modifications in the polymer matrix. SEM analysis reveals a high density of specialized structural groups and improved interfacial regions on the surface and within the bulk, which contribute to the high stability of the electret state. XRD and DSC data indicate that the introduction of HfO_2 nanoparticles leads to a redistribution of the amorphous and crystalline phases. Specifically, the observed decrease in permittivity is attributed to the partial dissolution or reorganization of the polymer's crystalline phase. Furthermore, the DSC results suggest that a portion of the HDPE matrix adsorbed onto the HfO_2 nanoparticle surfaces undergoes localized crystallization, creating a more complex interphase.

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