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THE IR SPECTROSCOPY AND ELECTRICAL PROPERTIES OF THE COMPOSITES ON THE BASIS OF P (VDF-TeFE) / NANO-SI

A.M. Maharramov, I.M. Nuruyev, R.N. Mehdiyeva, M.A. Nuriyev

Institute of Radiation Problems of ANAS

Abstract: It has been investigated the interrelationship between the changes occurred in IR spectra of initial and gamma irradiated composite samples obtained on the basis of Si nanoparticles with copolymer P(VDF-TrFE) of polyvinylidene fluoride with tetraftoretilen and electrical properties It is shown that, optical properties of P(VDF-TeFE)/nano-Si composites change depending on the amount of filler and irradiation dose and there is a one-valued relationship between this change and electrical properties of composites.

Keywords: composite, IR spectra, wave number, α , β and γ phases, special resistance, activation energy, construction, destruction

1. Introduction

Nowadays the development of the electronics and electro technology requires the creation of single or composite materials with thin layers that have electroactive properties. The study of the effects of structural changes which are formed as a result of change in technological parameters and composition on electrical properties during getting these active composites is one of the topical problems of nanoelectronics [1,2]. IR spectroscopy and other optical methods are widely used to study formation characteristics of the structure and properties of polymer and nanoscale filler-based composites. The IR spectrum of composites allows to obtain valuable information about their structure, different crystalline forms and inter-phase interaction between components. The comparative analysis of IR spectra allows us to reveal relationship between components. The typical frequencies of oscillation which are belong to intercomponent relations in the spectra depend largely on the chemical composition of the sample and the properties of the polymer matrix.[3-6].

Polyvinylidene Fluoride (PVDF) and its copolymer P(VDF-TeFE) with politetrafluoretileno and the study of composites on the basis of them by IR spectroscopy method can lead to many interesting results in this approach. This copolymer has electro active (piezo, pyroelectricity and electrets) properties. The oscillation of some groups of polymer chains is common for both β and γ phases of polymers according to the analysis of IR spectra given in its scientific literature [6,7] and the oscillation of other groups is related to the amorphous phase of the polymer [6-8]. The classification of IR absorption band which characterizes α , β and γ phases has been widely described in scientific literature [6-9]. Further, there can be one or more than one crystalline structures with different characteristics in the same film depending on its preparation conditions. As is well known, both γ and β phases are different oscillation forms (it indicates the same number of peaks) of the same polymer chain and occur around the same wave number. The 510cm⁻¹ peak of β phase which is very close to 512cm⁻¹ peak of γ phase in the PVDF spectrum can be shown as an example [10-13]. Some authors consider 840cm⁻¹ peak in PVDF spectrum as the same for both β and γ phases and while the others consider it to be typical only for β phase [13]. Although the 840cm⁻¹ peak is considered as the base for the β phase in the PVDF spectrum, the arm with the frequency of 833 cm⁻¹ is added to this peak in γ phase and this arm is characteristic for y phase of P(VDF-TeFE). In general, while 489, 614, 766, 795 and

855cm⁻¹ peaks are characteristic for α phase, 470, 510, 600, 745, 840, 1180, 1235cm⁻¹ peaks for β phase, 431, 512, 776, 812, 833, 1234cm⁻¹ peaks for γ phase in PVDF and its copolymer [13-17]. This research was devoted to investigating the relationship between the changes in initial and composite samples irradiated with gamma rays obtained on the basis of copolymer P(VDF-TrFE) of polyvinylidene fluoride with tetraftoretilen and Si nanoparticles with dispersity of d=50nm that occur in the IR spectra and electrical properties.

2. Experimental part

We used acetone (C₃H₆O) as a solvent during getting samples. Firstly, 5% solution of powdered P(VDF-TeFE) in acetone is prepared for getting samples. For this purpose, the polymer and acetone taken in a certain weight ratio are mixed with intervals in the magnetic mixer until the single solution forms 2% or 10% nano-Si is added on polymer dissolved in 5% P(VDF-TrFE) and is mixed for a long time with the help of magnetic mixer. Finally, obtained mixture has been mixed with intervals in "Vortex – 6" vibro shaker for an hour and it has been kept for 48 hour in hermetically sealed conditions for collapsing large-scale agglomerates. The top, liquid and relatively transparent part of the prepared solution maintained for 2 days is filtered into Petri containers and is kept for 24 hours for complete evaporation of the solvent. The samples are prepared by thermal pressing method at the temperature of T=190÷200°C and pressure of P=10Mpa in order to form a more perfect structure out of the obtained composite layer.

The amount of nanoparticles found in the composite layers can be determined either by the difference in weight between the initial polymer and composite or by the residue of the combustion product during DTA analysis [18]. The amount of the filler calculated according to the weight difference between initial polymer and composite in composite is ~1,07% by volume for the samples obtained by adding 2% volume nano-Si and ~4,82% for 10% volume nano-Si added samples.

P(VDF-TeFE) and optical properties (infrared spectra) of P(VDF-TeFE)/nano-Si particles on the basis of it have been drawn in 400-4000cm⁻¹ range of wave number (phase frequency) by using "Varian 640 FT-IR" spectrometer. The electrical properties of these samples – the temperature dependence of the specific volume resistivity $lg\rho_v = f(1/T)$ have been measured in temperature increase of ~2,5°/min with linear velocity by E6-13A teraohmmeter. Sandwichshaped samples which had plane parallel electrodes were used during measurement.

3. Experimental results

Now let's pass to the analysis of the IR spectroscopic spectra of the samples which obtained by thermal pressing of polymer and P(VDF-TeFE)/nano-Si composites which taken out of solution. To this end, let's first analyze the IR spectrum of the P (VDF-TeFE) copolymer (Figure 1a). The absorbing bands of 440-600, 800-900 and 1000-1450cm⁻¹ which are characteristic to the frequency range of 400-4000cm⁻¹ are observed. There are three maximum - 509, 840 and 1279cm⁻¹ which are belong to the β phase of P (VDF-TeFE) in the IR spectrum. The peak appropriate to 509cm⁻¹ wave number of β phase is relevant to δ CF2 oscillation (deformational oscillation of CF₂groups) and the peak corresponding to the 838,8cm⁻¹ wave number is relevant to rCF2 + vsCC oscillation that we observe from the spectrum. A large number of maximum which characterize oscillation belonging to CF, CF₂ and CF₃ bonds in the frequency range of 1000-1400cm⁻¹ in the spectrum of P(VDF-TeFE) samples are observed. There are strong maxima for the oscillation of CF₂bonds in the frequency range of 1120-1280 cm⁻¹. It is

known that mainly ~840cm⁻¹ conformational sensitive absorption band characterizes crystal β -phase of P(VDF-TrFE) polymer and comparison between its intensity and the intensity of the conformable non- sensitive ~880 cm⁻¹ absorption band allows estimating the amount of crystallization phase in the structure [10-15,19,20].



Fig. 1. P (VDF-TeFE) polymer (a) and nanosilicon (b) IR spectrum

Now let's consider the IR spectrum of the nanosilicon (Si) (Fig. 1b). We observe the absorption bands of frequency of 487, 1020, 1080, 1134, 1204 and 1280cm⁻¹ characterizing asymmetric and symmetric valent oscillation of Si-O-Si bridge bonds from the IR spectrum of Si nanoparticles [21]. The analyses of IR spectra that we have already described for matrix and filler allow us to analyze P (VDF-TeFE)/Si composites. P(VDF-TeFE)/Si composites are formed in two contents. The IR spectra of samples were described in figure 2 and the volume amount of Si nanoparticles was ~1.07% (Figure 2a) for the first sample, and it was ~4.82% (Figure 2b) for the second sample. Comparing the IR spectrum of the samples added Si nanoparticles to the P (VDF-TeFE) samples, we see that the intensity of the peak of 841cm⁻¹ has increased. In addition,

new absorption bands of the 487, 1020, 1080, 1134, 1204 and 1280cm⁻¹ have also occurred, which can be explained only by the presence of the filler. As we stated above, relatively high electroactive properties of the copolymer P(VDF-TeFE) compared to other polymers are determined by its polarization and crystalline structure.



Fig. 2. IR spectra of P (VDF-TeFE) / nano-Si composites where the volume amount of the filler is ~ 1.07% (a) and ~ 4.82% (b)

We assume that Si nanoparticles in the P (VDF-TeFE)/nano-Si composites can take part as crystallization centers in the matrix and it increases the intensity of the corresponding maxima in the spectrum. The intensity of the maximum corresponding to the phase frequency of 840 cm⁻¹ in the sample which was added ~ 4.82% filler increased about 6 times than the P (VDF-TeFE) sample, and approximately 4,5 times than the sample which was added ~ 1.07% nano-Si. The intensity of the maximum corresponding to the frequency of 510cm⁻¹ was approximately 7 times higher than pure P (VDF-TeFE) and ~ 1.07% nano-Si added sample. All this allows us to say that β phase raises as the concentration of the filler increases in the composite [22].

On the other hand, the active interaction that can arise between the polar groups in the matrix and the highly effective surface of the nanosilicon may result in the formation of new bands in the IR spectrum. As we know from the literature, the new absorbing bands with the frequency of 487, 951, 1020, 1080, 1134, 1204 and 1280 cm⁻¹ observed in the IR spectrum of composites characterize oscillation and interaction of Si-H and Si-O-Si bonds [23]. The 861, 1069 and 1086cm⁻¹ absorption bands of the IR spectrum of the composite indicate the presence of Si-O-C (1069 and 1086cm⁻¹) and Si-C (761 cm-1) bonds. [24]. It is clear from the spectra that the intensity of these bonds is higher in the composite where the filler is much. The arm of the absorption bands with the frequency of 1272, 1190, 950cm⁻¹ and the frequency of 890÷895 cm⁻¹ characterizes valent oscillation relevant to Si-CH₃, SiO-CH₃ and (CH₃)₃-SiF bonds. We consider that the absorbing band of~1074cm⁻¹ characterizing the Si-O bridge bonds and its surroundings are equivalent to the valent oscillation of the Si-O-Si bond with the maximum in the frequency of 1080cm⁻¹ and is a sign of the relationship between Si nanoparticles [25]. Therefore, we consider that the absorption bands in the IR spectrum of P(VDF-TeFE)/4.82% of nano-Si composites form oscillation belonging to filler-filler relationships, as well as oscillation and oscillation corresponding to filler-matrix relationships. The high value of the intensity of maximum of some absorption bands (1080 cm⁻¹ and its surrounding) gives us a reason to say that what we say is right. In addition, the wide maximum observed in the wave number range of 3650-3850 cm⁻¹ in the IR spectrum of the ~4,82% nano-Si composition was probably belong to the valent oscillation of -OH groups in the Si-OH bonds and it is related to the porous structure of the composite and high adsorption ability of the filler surface. The presence of the maximum of the deformational oscillation in the frequency range of 830-950cm⁻¹ of –OH groups in this composite proves the accuracy of what we say [24]. All of these give us a reason to say that there is a strong bond between the filler and the matrix in the composite.

The effect of ionizing radiation on these materials is very important from the point of view of their property management. The effect of ionizing radiation on polymers, as we know, is accompanied by the formation of a number of processes, such as construction, destruction and oxidation, depending on the power of the absorption dose. These processes either slow down or increase depending on the amount and type of the filler in the structure of the composites. On the other hand, ionizing radiation is often used for modification of the materials, ie, to manage the properties that are interesting. Therefore, the effect of gamma radiation on the composites we have investigated is very important from the point of view of topicality, and the results of these studies are described below. The structural changes occurring in the composites due to gamma radiation were made according to the results of comparative analysis of IR spectra that were taken before and after radiation (Fig.3). The results of the analysis have been presented in the table.

Table

The intensity change of initial and radiated P (VDF-TeFE)/1.07% nano-Si samples in the IR spectra for Si-C ($761\pm5cm^{-1}$), Si-O-Si ($1075\pm10cm^{-1}$) and Si-OC and (1069 and $1086cm^{-1}$) and C=O ($1710-1740cm^{-1}$)bonds

D, kGy			0	100	300	500
	K, cm ⁻¹	F, volume%	А	А	А	А
1	761±5	~1.07	0.739	1.68	2.82	3.47
2	1075±10	~1.07	9.61	37.39	69.1	21.2
3	1710-1740	~1.07	2.6	14.83	34.9	52.7

To evaluate the structural changes in the P (VDF-TeFE)/nano-Si composite, we used the results of the change in the location and intensity (maximal area) of C = O carbonyl groups in the range of 1710-1740cm⁻¹in IR spectra of radiated composite samples by initial and different doses. The data in the table show that double C = C carbonyl groups are formed after the radiation exposure and the last C = O due to the breakage of the polymer chain in the IR spectrum of P (VDF-TeFE)/1.07% nano-Si samples. The formation of these groups shows itself in the increase of intensity of the absorption band in the wave number range of 1720-1740cm⁻¹in the spectrum. The intensity of the maxima of oscillation of C = O carbonyl groups is expected to increase, as the radiation dose rate increases. The kinetics of increase in intensity of the maximum in the wave number of 1725cm⁻¹ is quite high depending on the absorbed dose power. We assume that the cause of it is dose power being mainly in polymer matrix in the low values of concentration, polymer destruction and formation of C=O carbonyl groups faster as a result of the subsequent oxidation.





Fig. 3. IR spectra of P (VDF-TeFE)/nano-Si composites takenbefore (a) and after (b, c) radiation: b-D = 300kGr; c-D = 500kGr;

The electron hunger of the fluorine in the chain during the gamma radiation of the polymer causes the breakdown of C-F bonds and decrease in the amount of CF2 groups and conversion some of them into CF radical. The recombination of these radicals in the matrix results in the formation of interchain construction. The interchain construction increases the rigidity of the polymer matrix and the interactive effects of the active centers after radiation result in an increase in the crystallization phase as a result of rise in Si-O-C(1069 and 1086cm⁻¹) and Si-C (761cm⁻¹) bonds, as well as, Si-O-Si (1075±10cm⁻¹) bonds at the matrix-nanoparticle interphase boundary. An increase in the intensities of absorption bands of 509cm⁻¹ and 840cm⁻¹ characterizing crystal β -phase in the matrix in the IR spectrum of P (VDF-TeFE) / Nano-Si system is an evidence of this[13,23]. The intensities of both Si-O-Si and Si-O-C (1069 and 1086cm⁻¹) bonds decrease passing a maximum dose of 300kGr with the increase in dose for P (VDF-TeFE)/1.07% nano-Si samples. The decrease in Si-O-C (1069 and 1086cm⁻¹) bond, the relative increase in Si-C (761cm⁻¹) and the rise in corresponding intensities have been observed as the dose increases up to 500kGy.

All these structural changes should also affect the electrical properties of the composites. Below, the temperature dependence of the special electrical resistivity of the P (VDF-TeFE)/1.07% nano-Si and P(VDF-TeFE)/4.82% nano-Si nanocomposites in the Arrenius coordinates has been described (Figure 4 and Figure 5). Our goal is to investigate the reason of the variations that occur by changing the concentration of nano-Si particles in 5% P (VDF-TeFE) solution of the polymer with the same concentration by analyzing temperature dependence of the electrical conductivity. The temperature dependence of the specific electrical resistivity of the initial P (VDF-TeFE) was also added to both dependencies for comparison (curve 1). As seen from dependencies, while there is a minimum in the curve (curve 2) corresponding to the first measurement in the low-temperature region in P-VDF-TeFE / 1.07% nano-Si composite, it has not been observed in P (VDF-TeFE) / 4,82% of nano-Si composites. We observe the loss of this minimum for both composites after approximately one hour's interval in the dependence of samples taken on the basis of P (VDF-TeFE) / 1.07 nano-Si during the second measurement (Figure 4 and Figure 5, curve 3).



Fig. 4. The temperature dependence of specific electrical resistivity of P (VDF-TeFE) / 1.07% nano-Si nanocomposite: 1- P (VDF-TeFE); 2- P (VDF-TeFE) / 1.07% nano-Si, I heat; 3-II heat.



Fig.5. The temperature dependence of the specific electrical resistivity of the P (VDF-TeFE) / 4.82% nano-Si nanocomposite: 1- P (VDF-TeFE); 2- P (VDF-TeFE) / 4.82% nano-Si, I heat; 3-II heat.

We consider that the reason of formation of this minimum in the dependence of $lg\rho_{\nu} = f(1/T)$ of P (VDF-TeFE) / 1.07% of the nano-Si composite is the desorption of absorbed water molecules in the sample during getting and conserving after getting. The relative high concentration in P (VDF-TeFE) / 4.82% nano-Si composites leads to the increase in Si-OH bonds. This results in a relative increase in the intensity of oscillation of Si-OH bonds and the value of the initial resistivity of the sample around the 3800cm⁻¹ absorption band in IR spectra.

On the basis of the comparison (figure 4 and 5) of the curves corresponding to the curves derived from the second measurement of samples in the high temperature zone we can say that the activation energy of electric conductivity of P(VDF-TeFE)/1%nano-Si composite is greater than the activation energy of the P(VDF-TeFE)/4,82%nano-Si composite. While the activation energy of P(VDF-TeFE) / 1% nano-Si composite is equal to the activation energy of the initial P

(VDF-TeFE) layer according to the angle coefficient taken on the curves, it is bigger than the activation energy of the P (VDF-TeFE) / 4,82% nano-Si composite. In our opinion, the concentration of filler particles in P (VDF-TeFE) / 4.82% nano-Si composite is quite high, and it causes the increase in the effective surface and decrease in mobility of polymer chains by strengthening the interaction of particle-macromolecules in the polymer matrix-filler interphase boundary, as well as a reduction in the activation energy. The formation of absorbing bands of Si-O-Si, Si-O-C and Si-OH bonds and the increase in their intensities in the IR spectrum is a proof of it. On the other hand, the Si-OH bonds are very small in P(VDF-TeFE)/1% Nano-Si composite, so the change is mainly observed in the region of 3600-3800sm⁻¹ of the P (VDF-TeFE) / 4.82% nano-Si composite in the IR spectrum.

Similar changes are also observed in the temperature dependence of the initial resistance of the measured variable region of initial and radiated P (VDF-TeFE) / 1% nano-Si composites (Figure 6). The effect of ionizing radiation of the P (VDF-TeFE) / nano-Si composites obtained out of the mechanical mixture of components by thermal pressing method has been studied in previous studies [26]. It has been shown that the causes of the changes occurring in the electrical properties of the radiated samples are the differences in the distribution of the components among absorbed energy dose along with the construction and destruction processes [26,27].



Fig. 6. Temperature dependence of the special resistance of initial and radiated P (VDF-TeFE)/2% nano-Si composites measured in the variable region: 1-initial; 2-100kGr; 3-300kGr; 4-500kGr;

The activation energy which conditions the conductivity according to the inclination in the high temperature is higher in comparison with the initial sample in the $lg\rho_v = f(1/T)$ dependence of the samples radiated in three dosage (100, 300, 500kGr) for P (VDF-TeFE)/2% nano-Si composites. The minimum observed in $lg\rho_v = f(1/T)$ dependence of the initial sample lost in the samples radiated by 100 and 300kGr dose, whereas it had been observed in a very small amount in the sample radiated by 500kGr dose at low temperatures. We assume that the cause of it is the dominance of the construction processes over destruction in the polymer matrix at the dose of 100 and 300kGr, which ultimately leads to increased Si-OH and Si-H bonds. The increase of excess destruction and oxidation processes by dominating construction processes at the dose of 500kGr leads to the re-emergence of the minimum.

Thus, by summarizing all this we can come to this conclusion:

- Based on the analysis of IR spectra obtained before and after gamma radiation of P (VDF-TeFE)/nano-Si composites, we can say that the crystallization rate of the system is increasing as a result of interaction of the active centers formed on the matrix after radiation with the surface of Si nanoparticles on the interphase boundary.
- The optical properties of P (VDF-TeFE)/nano-Si composites obtained based on the P (VDF-TeFE) polymer change depending on the amount of filler and the radiation dose, and there is a one-valued relationship between this change and the electrical properties of the composites.

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ИК-СПЕКТРОСКОПИЯ И ЭЛЕКТРИЧЕСКИЕ СВОЙСТВА КОМПОЗИТОВ НА ОСНОВЕ П(ВДФ-ТЕФЭ)/НАНО-SI

А.М. Магеррамов, И.М. Нуруев, Р.Н. Мехдиева, М.А. Нуриев

Резюме: Исследовано взаимосвязь между изменениями в ИК спектрах и электрических свойствах исходных и гамма облученных композитов полученных на основе сополимера поливинилиденфторида с тетрафторэтиленом П(ВДФ-ТеФЭ) и наночастиц кремния (нано-Si). Показано, что оптические свойства композитов П(ВДФ-ТеФЭ)/нано-Si изменяется в зависимости от содержании наполнителя и дозы облучения. Установлено, что имеется однозначное связ между этими изменениями и электрическими свойствами композита П(ВДФ-ТеФЭ)/нано-Si.

Ключевые слова: композиты, ИК-спектры, волновое число, α, β и γ-фазы, удельное сопротивление, энергия активации, сшивание, деструкция

İR SPEKTROSKOPİYASI VƏ P (VDF-TEFE) / NANO-Sİ ƏSASINDA KOMPOZİTLƏRİN ELEKTRİK XÜSUSİYYƏTLƏRİ

A.M. Məhərramov, İ.M. Nuruyev, R.N. Mehdiyeva, M.A. Nuriyev

Xülasə: Polivinilidenftoridin tetraftoretilenlə sopolimeri P(VDF-TrFE) ilə Si nanohissəcikləri əsasında alınmış ilkin və qamma şüalandırılmış kompozit nümunələrinin IQ spektrlərində baş verən dəyişiklərlə elektrik xassələri arasında əlaqə tədqiq edilmişdir. Göstərilmişdir ki, P(VDF-TeFE)/nano-Si kompozitlərinin optik xassələri doldurucunun miqdarından və şüalanma dozasından asılı olaraq dəyişir və bu dəyişmə ilə kompozitlərin elektrik xassələri arasında birqiymətli əlaqə mövcuddur.

Açar sözlər: kompozit, İQ spektrlər, dalğa ədədi, α , β və γ fazalar, xüsusi müqavimət, aktivləşmə enerjisi, tikilmə, destruksiya.