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Derivatographic analysis of PE polymer nanocompounds with $Co(AlO_2)_2$ filler

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Polyethylene (PE) films filled out with $Co(AlO_2)_2$ in amounts of 0,5 vol.% and 1 vol.% exposed to electrothermopolarization have been investigated by derivatography method. By electrothermopolarization at $E=7\cdot 10^6$ v/m there has been taken place thermaldestruction of PF matrix crystal part, as a result thermal stability decreases to 4,4 % comparing with PF not exposed to electrothermopolarization. It is established that the effect of electric field by addition of 0,5 % $Co(AlO_2)_2$ in PE appears in the form of the curve on DTA by endothermal effect accompanying by loss of mass due to forming intermediate product volatility in amounts of 17%. In PF + 1% $Co(AlO_2)_2$ composition polymer matrix there has been taken place the process of polymer crystal part amorphism accompanying by forming intermediate product volatility in amounts of 23%.

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1. Introduction

It is known that use of different methods of polymer material modification leads to pronounced development of the field of their application. In this case directional change of structure and polymer properties are carried out either during the synthesis process or by the effect on finished product with the introduction of different chemical character fragments into the macromolecule. Recently when creating polymer materials with given properties special attention was given to modification of their surface as the structure of surface layer just determines their behavior under the working conditions [1-4] in many ways. Nowadays modification of electret and electrophysical properties of two – phase systems of polymers with different fillers is of greater importance. In polymer by strong electric field there have been ageing processes covering deeper layers of polymer. As a result of latter there have been taken place changes in structural part of polymer related to the changes in crystal and amorphous part relation of polymer [1], also with the formation of interlaminar crystal phase in polymer matrix and etc. Study of polymer composite material thermal properties gives us the scope for evaluating the quality of filled material and working out recommendations on their use and production technology [2-4].

2. Methods of sample production and measures

By hot pressing PE samples including additions of $Co(AlO_2)_2$ low-molecular colorant to the extent of 0.5 and 1.0 vol.% at melting temperature of polymer matrix under the pressure 15MPa for 3 min. with subsequent quick cooling at the rate 2000 deg/min. have been made. Obtained samples have been preliminarily exposed to the electrothermopolarization at T_i=335 K under the electric intensity $E_i=5\times10^6$ V/m, $E_i=7\times10^6$ V/m for t_i=1haur.

Derivatograms have been recorded on Q-derivatograph of MOM-typed Paulink-Paulink Erdey system (HUNGAPY) within T 20÷450 °C. Weighted amount of films under the investigation depending on thickness is from 70 up to 180 μm. Channel sensitivity is DTA-1/5; DTG-1/15; mg-200. Rate of heating is 10% min; as a standard Al_2O_3 roasted at T1000 °C for 12 hours has been used.

3. Experimental results and discussion

In Fig. 1 PE derivatographs are given. As it is seen on DTA curve at E=0 two endothermal effects at T105 °C and T405 °C and two exothermal effects at T187 °C and T327 °C have been found out. Endothermal effect at T105 °C is in agreement with polymer matrix crystal phase melting. Exothermal effect within T155 °-255 °C with the peak at T187 °C is in agreement with the formation of hydroperoxide groups, i.e. the first stage of oxidation. The second blurred exothermal effect at T327 °C is in agreement with the break of weak bonds. Vigorous endothermal effect at T405 °C is in agreement with polymer depolymerization.

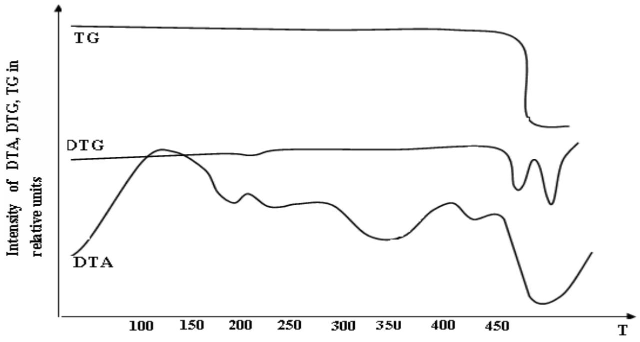

Fig. 1. Derivatograph of PE films obtained under water quenching conditions without treatment in constant electric field.

In Table 1 there are shown the results of PE thermogravimetric and differential-thermal analyses filled by $Co(AlO_2)_2$ in amounts of 0.5 vol.% and 1 vol.%. On DTG curve the process of weak bond break and depolymerization appears as the endothermal effects at T400 °C and T430 °C accompanying by loss of mass on TG curve in amount of 15.6% , the balance of PE unburnt mass by TG calculation is 84,4%.

On PE derivatograph (Fig. 2) exposed to electrothermopolarization at $E=7\times10^6$V/m melting temperature of polymer matrix crystal phase shifts to low temperatures by 5 °C, i.e. is equal to 100 °C. The first stage of oxidation occurs at T240 °C. The onset of weak bond break on both curves DTA and DTG fits T272 °C. The peak on curve DTG is observed at T290 °C but on curve DTA is observed at T305 °C. The process of weak bond break is followed by mass volitality in amounts of 20% but the balance of unburnt mass is 80%. From forming volatile components of TG and DTG curve course calculation follows that by applying electric power $E=7\times10^6$V/m, polymer thermodestruction comparing with PE at E=0 is more than 4%. By addition PE+0.5%$Co(AlO_2)_2$, (E=0) there have been revealed three endothermal effects on DTA curve at T120 °C, T210 °C and T270 °C (Fig. 3). Endothermal effect at T120°C is due to melting of polymer matrix crystal phase, at T210 °C the first stage of polymer oxidation, at 270 °C polymer depolymerization are taken place. These processes on DTG curve appear as one endothermal effect at T260 °C accompanying along TG curve by volatility of volatile components in amounts of 17%. As a result of polymer depolymerization the balance of polymer unburnt part by heating up to T380 °C is 83%. It should be noted that $Co(AlO_2)_2$ in filler amounts of 0.5% increases melting temperature of polymer matrix crystal phase by 15 °C but the temperature of the 1st oxidation stage by 3 °C comparing with the initial PE.

Table 1. TG and GTA analyses of polyethylene films.

Composition on PE-based compounds	Differential-thermal analysis - DTA				Differential-thermo gravimeter analysis - DTG, TG		
	Meling temperature °C of crystal phase	Ist stage of oxidation T°C	Break of wear bonds T°C	Depolymerization T °C	Endothermal effect-DTG, T°C	Amount of volatile component by TG in %	Balance as a remit of depolymerization by heating up to 360-380°C, in %
PE, E=0	105	187	380	405	400,430	15,6	84,4
PE, $E=7\times0^6$ V/m	_	_	240	305	290	20	80
PE+0,5% $Co(AlO_2)_2$, E=0	120	190	240	290	240	17	83
PE+0.5% $Co(AlO_2)_2$, $E=7\times10^6$ V/m	_	170	205	230	230	30	40
PE+1% $Co(AlO_2)_2$, E=0	70	90	188	290	280	23	77
ПЭ+1% $Co(AlO_2)_2$, $E=7\times10^6$ V/m	70	140	195	230	230	33	67

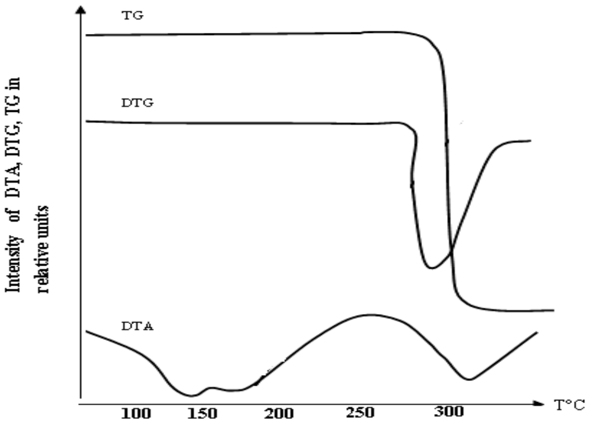

Fig. 2. Derivatograph of PE films obtained under water quenching conditions treated in constant electric field at $E=7\times10^6$ V/m.

Break of weak bonds and depolymerization have been taken place at lower temperature, lower than in initial polymer, i.e. by 97 °C and 115 °C, respectively. The latter indicates that thermal stability of polymer decreases.

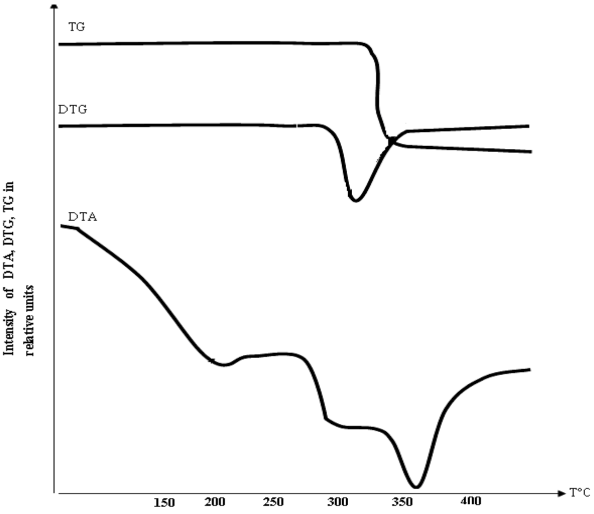

Fig. 3. Derivatograph of PE+0,5% Co $(ALO_2)_2$ nanocompounds obtained under water quenching conditions without treatment in constant electric field.

In the derivatograph PE+0.5%Co$(ALO_2)_2$ composition nanocompound has been exposed to electro thermopolarization at $E=7\times10^6$V/m, (Fig. 4), on DTA curve there has been found out one exothermal effect at T170 °C appropriate to the 1st stage of nanocompound oxidation, then there has been followed endothermal effect at T230 °C appropriate to compound depolymerization. Given processes on DTG curves are accompanied by one endothermal effect at T230 °C due to the loss of volatile components in amounts of 30% calculated according to TG curve. The balance of nanocompound unburnt mass by heating up to T360 °C is 70%.

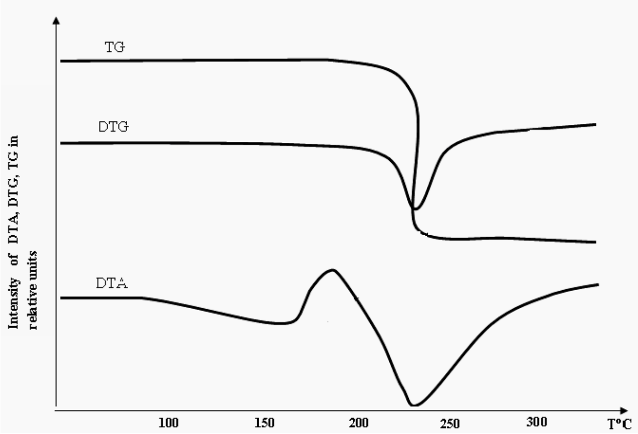

Fig. 4. Derivatograph of PE+0.5% Co$(ALO_2)_2$ nanocompounds under water quenching treated in constant electric field at $E=7\times10^6$ V/m.

By adding to PE+1%Co$(ALO_2)_2$ composition when E=0 there has been taken place polymer amorphysm process (Fig. 5) that on DTA curve appears as decrease of matrix crystal phase melting temperature up to T50 °C and blurred endothermal effect at T50 °C. 1st stage of polymer oxidation manifests itself by exothermal effect at T70 °C, break of weak bonds takes place at T160°- 188 °C with the peak of endothermal effect at T188 °C but depolymerization is followed by endothermal effect at T290 °C. The processes on DTG curve like these appear as one endothermal effect at T290 °C accompanying by volitality of volatile companents in amounts of 23% calculated according to TG curve. Solid balance of depolymerization processes unburnt part by heating up to 360 °C is 77%.

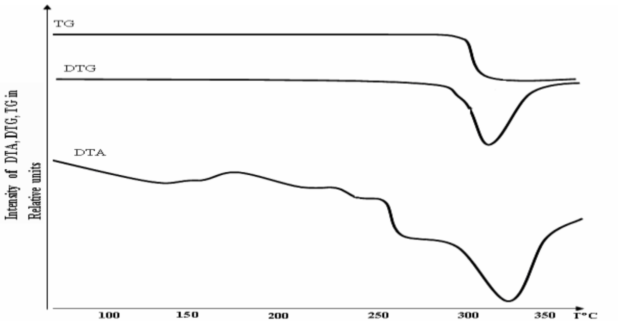

Fig. 5. Derivatograms of PE+1% Co$(ALO_2)_2$ nanocompounds obtained under water quenching conditions without treatment in constant electric field.

In the derivatograph of PE+1%Co$(ALO_2)_2$ composition nanocompound exposed to electrothermopolarization at $E=7\times10^6$ V/m, on DTA curve (Fig. 6) endothermal effect appropriate to polymer matrix crystal phase melting (amorphism of compound matrix crystal phase appeared to be happened because of high electric discharge) has not been found out. 1st oxidation stage of nanocompound takes place at 140 °C but the break of weak bonds is revealed at 195 °C, effect of depolymerization is revealed at 230 °C. The processes on DTA curve like these manifest themselves by one endothermal effect at 230 °C that by TG curve calculation is in agreement with loss of volatile components in amounts of 33%, the balance of depolymerization by heating up to 360 °C is 67%.

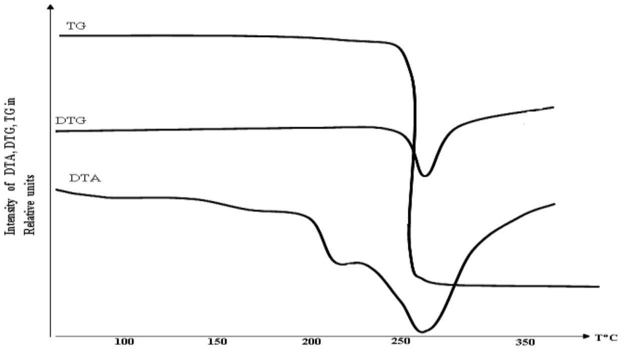

Fig. 6. Derivatograph of PE+1% $Co(ALO_2)_2$ nanocompounds under water quenching treated in constant electric field at $E=7\times10^6$ V/m.

From thermal data given in Table 1 and Fig. 1-6 follows that compared with initial PE $Co(ALO_2)_2$ filler addition in amounts of 0.5% in PE leads to increase of matrix crystal phase melting temperature by 15 °C. In this case in all PE+0.5%$Co(ALO_2)_2$ and P+1%$Co(ALO_2)_2$ compositions under investigation exposed to electrothermopolarization at $E=7\times10^6$V/m maximum thermal destruction is produced. This points out to the low temperature depolymerization and large amount of volatile component volatility (30– 33%).

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