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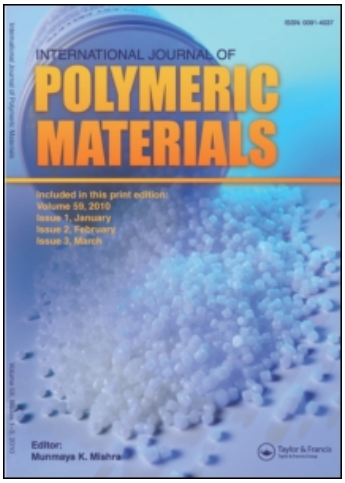

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T. A. Rudakova[a]; O. V. Demicheva[a]; V. V. Egorov[b]; N. A. Khalturinskii[b]
[a] Institute of Synthetic Polymer Material of RAS, [b] Institute of Chemical Physic of RAS,

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The Influence of Pressure and Oxidizing Atmosphere on Foam Coke Layer Formation under Heat Action on Polymer Coating

T. A. RUDAKOVA[a], O. V. DEMICHEVA[a],
V. V. EGOROV[b] and N. A. KHALTURINSKII[b,*]

[a] *Institute of Synthetic Polymer Material of RAS;*
[b] *Institute of Chemical Physic of RAS*

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This work is devoted to investigation of external condition influences, in particular: of external atmosphere pressure, composition of oxidizing environment, influence of additions on a process of formation of carbonized layer during stage by stage study of polymer material diffusive combustion.

Keywords: Material combustion; coke formation; external factors polymer coatings

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It is known, that combustion of a polymer material occurs in oxidizing atmosphere of environment. It is natural that characteristics of this oxidizing atmosphere, such as composition, pressure, velocity of accumulating flow, play an essential role in the process of combustion. For a large class of polymer materials which do not form under combustion and pyrolysis a coke skeleton, an influence of these characteristics on the process has not been studied well enough. For materials forming under heating and combustion volume coke layer only a few investigations have been carried out.

*Corresponding author.

EXPERIMENTS METHODS

Experimental research of influence of pressure and composition of oxidizing atmosphere on quantitative characteristics of a polymer material thermo-degradation under the pressure below 1 atm. are carried out in vacuum chamber (Fig. 1).

Removable glass bell-shaped chamber 1 is ground to the metal substrate. The contacts for bringing voltage to heater are installed in the substrate. The system is provided with gas-conducting tube and is connected to vacuum pump. Initially, the chamber is pumped out to pressure 10^{-2} torr; then it is filled in by a mixture of nitrogen and oxygen at required proportion up to the necessary pressure. We investigated regularities in combustion of cylinder samples of polymer materials and quantitative characteristics of volume thermo-degradation process. To this aim the disperse condensed products are trapped using quartz tube with a filter (Fig. 2).

At a pressure higher than the atmospheric one, investigation were carried out in a hermetic steel chamber, *i.e.*, in high press chamber with constant volume. The high press chamber (Fig. 3) has inspection windows 1 for visual inspection and photo-registration, supplied with the screw presssing nut 10 and cover 9. The isolated and sealed electrical-inlets 2, 3, gate for shedding pressure 6 and substrate for

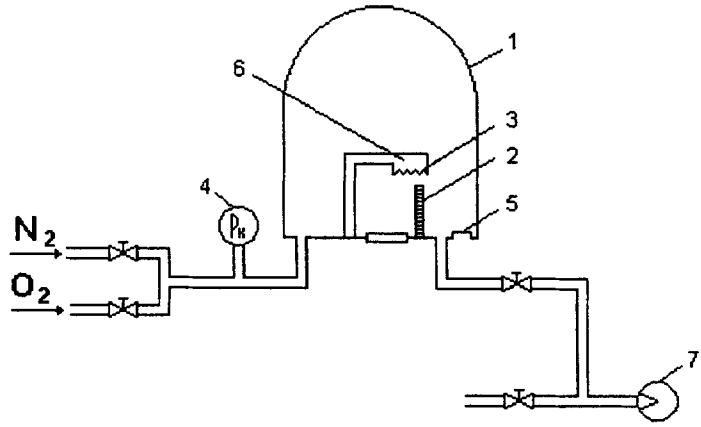

FIGURE 1 Vacuum chamber. 1-combustion chamber; 2-observable sample; 3-spiral for sample burning; 4-vacuummeter; 5-substrate; 6-system for spiral removement; 7-vacuum pump.

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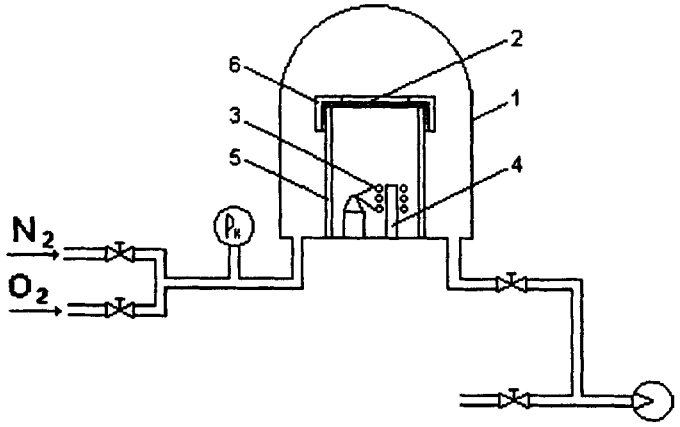

FIGURE 2 Scheme of the system for disperse phase extraction during thermo-degradation. 1-bell-shaped chamber; 2-filter; 3-heater; 4-sample; 5-quartz glass; 6-pressing device.

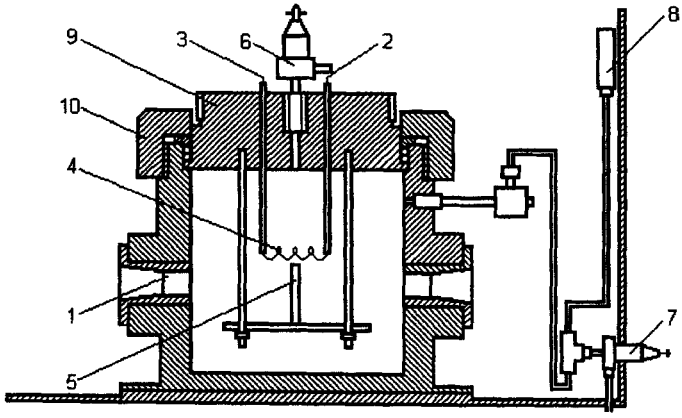

FIGURE 3 High press chamber. 1-inspection window; 2,3-electrical inlets; 4-spiral for sample burning; 5-sample; 6-gate for shedding pressure; 7-gate for supplying compact gas; 8-manometer; 9-cover; 10-pressing nut.

sample are installed into the cover. The chamber has the gate for supplying compact gas from standard gas balloons.

Before an experiment the chamber is filled up by the gas mixture of necessary composition and pressure, and then the thermo-degradation is realized in required regime.

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EXPERIMENTAL RESULTS

In this work we studied the behavior of foamed coating on the base of carbamide-formaldehyde resin (Russian patent No. 98102202) and epoxy resin ED-20 with additions of PPhA, PE, hydrazine-dihydrochloride in different proportions and under different heat sources depending on composition and pressure of ambient atmosphere. It has been found out, that the ambient atmosphere pressure has rather strong influence on intumescence character, geometrical size of coke char and on parameters of a coke sell structure.

In the study of the pyrolysis process of epoxy resin with additions of IA plus hydrazine-dihydro-chloride we found out that hydrazine-dihydro-chloride initiates the resin destruction shifting the temperature of destruction beginning into the region of lower temperatures. Using visual inspection it was established that an addition of small quantity of hydrazine-dihydro-chloride into the intumescence system produces earlier softening of the condensed phase, as well as melting of the deepening in the area of the front edge of flame under the condition of the limiting candle-like combustion. When hydrazine-dihydrochloride additions reach more than 10% of sample weight, spreading of vertically orientated sample takes place. From chromatographic analysis of destruction products it is known [1], that hydrazine-dihydrochloride promotes increasing of water outcome and decreasing of resulting quantity of carbon oxide. This leads to reduction of toxicity of the pyrolysis products. Besides methanol and benzol disappeared acetaldehyde, which can be an initiator of chain branched reactions in gas phase, is not found.

The dependence of coke char height, obtained by action of heating radiation source (of fixed power of the order 75 Wt) on vertically orientated sample with coating, on absolute pressure of oxidizing atmosphere is plotted on the Figure 4. It is shown, that for the composition CPh resin plus 30% IA and at the enough low pressure the cokes char height at first raises up, then reaches some maximum value, and then with further pressure increasing the height slowly decreases and at some value of pressure the foamed coke formation can be practically suppressed. An explanation of these results can be given with use of the fact that intumescence takes place at the stage of temperature increasing (and, hence, decreasing of the melt viscosity) and together

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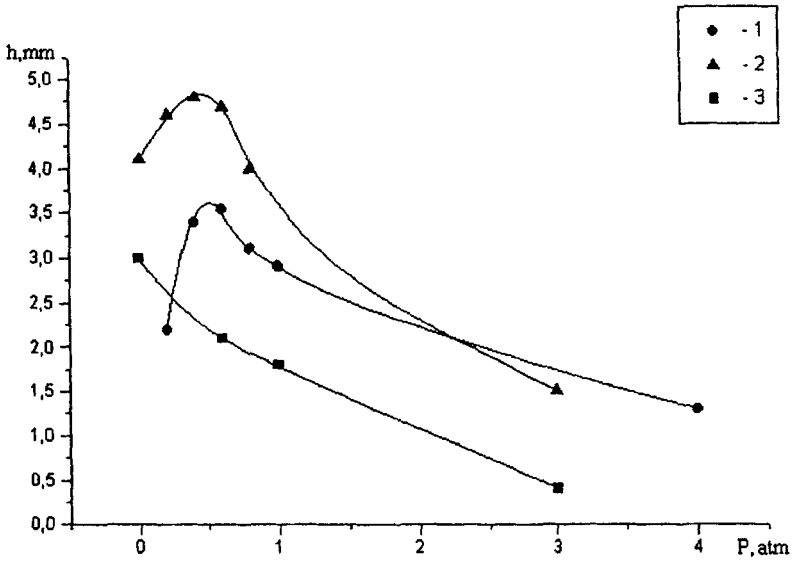

FIGURE 4 Dependence of coke layer height on absolute pressure in the chamber. 1-CPHhR + IA; 2-ED-20 + IA(40%) + 5%HDHCl; 3-ED-20 + IA(40%).

with increasing of system rigidity because of cross-linking processes occurring at the temperature increasing. The plot for the composition with additions of hydrazine-dihydro-chloride is upper than that without the additions, which lower the melt viscosity assisting in earlier foam formation.

Dispersion Role at the Combustion and Pyrolysis of Polymers

Detailed study of dispersion under the conditions natural for a combustion process gave values of coke and dispersed residues. From Figures 5a, 5b it is seen that both pressure and composition of oxidizing atmosphere have essential influence on values of both coke and dispersed residues of the material under investigation. It is found that in pure nitrogen the value of coke residue decreases with increasing of pressure considerably faster than in pure oxygen. Value of disperse residue increases a bit slower in pure oxygen in comparison with that in pure nitrogen. It seems, that an explanation to this fact can be

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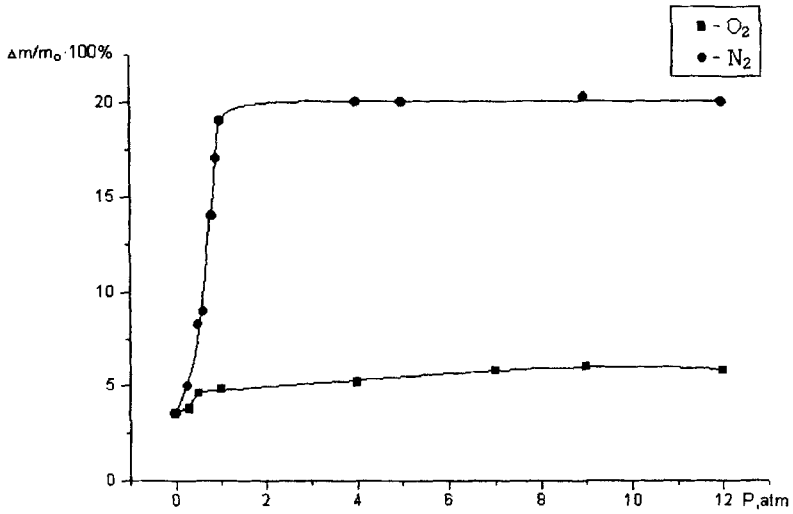

FIGURE 5a Dependence of disperse residue value on pressure. Composition ED-20 + PEPA.

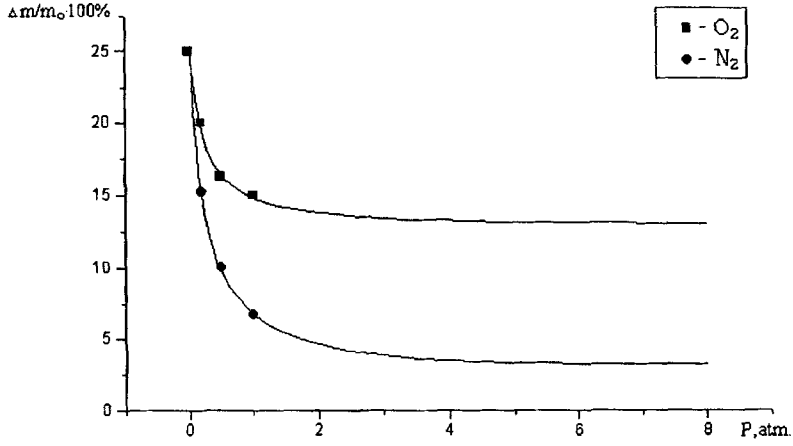

FIGURE 5b Dependence of value of coke residue in the crucible on pressure. Composition ED-20 + PEPA.

related to additional influence of atmospheric oxygen on cross-linking processes taking place at epoxy polymer pyrolysis.

The simplest estimation of critical for coke char local fragment tearing off [2] allows to make conclusion that external pressure increasing

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suppresses dispersion, this fact being in good agreement with the experimental results.

In the polymer destruction process some increasing of pressure in the chamber occurred, however it was insignificant in comparison with general pressure.

It is shown, that the absolute external pressure has essential influence on the process of foam formation at combustion and pyrolysis of coke formation polymers. Under variation of external pressure geometrical parameters of coke char are changed. It is established, that at pressure higher 1 atm pressure increasing suppresses foam formation. It is shown, that insignificant additions of HDHCl have crucial influence on the foam formation characteristics.

In stage by stage study of combustion process the dispersion role is revealed, especially noticeable at low pressure. It is found out that a composition of oxidizing atmosphere has influence on a pyrolysis process for cross-linked polymers.

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