EFFECT OF CHAR LAYER ON TRANSIENT THERMAL OXIDATIVE DEGRADATION OF POLYETHYLENE

by

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A transient one dimensional model has been presented to simulate degradation and gasification of polyethylene, in early stage of fire growth. In the present model effect of oxygen on degradation and rate of polymer gasification while the sample is subjected to an external radiative heat source is numerically investigated. This model includes different mechanism, which affect the degradation process, such as in depth thermal oxidative decomposition, in depth absorption of radiation, heat transfer, volatiles advection in solid phase and convective heat transfer on surface. Also effects of radiative parameters, due to formation of char layer such as surface reflectivity and absorptivity on thermal degradation of polyethylene are investigated. The results for 40 kW/m² heat source are reported and yielded realistic results, comparing to the published experimental data. The results show that an increase in oxygen concentration leads to considerable increase in gasification rate and also leads to sharp increase of surface temperature.

Key words: *thermal oxidative degradation, polyethylene, reflectivity, absorptivity, absorption coefficient*

Introduction

Polymeric material are increasingly common as components of furnishing, they constitute a substantial fraction of the fuel load in typical modern residential dwellings and other buildings. It is important to understand why these materials behave as they do in fire environment. Of particular interests are factors which control the rate of polymer gasification under transient heating such as occurs in initial stage of fire growth. According to Esfahani's numerical study [1] the rate of gasification plays the major role in the ignition and combustion processes, thus any attempt for better understanding of gasification process and formulating the parameters affecting the rate of gasification would be of significant value in the development of fire detection and protection appliance. Although in the past decades, great advances in the field of thermal degradation of solid material have been made, only a few works have been published on the simultaneous thermal and oxidative degradation of solid material. For example the work of Hirata *et al.* [2] discusses various type of reaction mechanism in the polymer such as random scission initia-

tion and first order termination reaction in presence of oxygen. Kashiwagi and Ohlemiller [3] presented detailed experimental results of the oxygen effects on the rate of degradation of polyethylene (PE) and polymethyl methacrylate (PMMA). Bulter [4] proposed a mixed layer model to simulate surface regression, melting, and bubbling effects of degrading polypropylene sample, in her task [5], she presented a numerical model for combustion of bubbling thermoplastic in the microgravity atmosphere, however the oxygen effects on the rate of gasification were neglected in the mentioned model. The previous numerical works of Esfahani (1999-2004) [6] illustrated ignition of epoxy, in his other numerical task [7] the model of oxygen sensitive thermal degradation of PMMA was considered for first time, where the oxygen concentration on irradiated surface is assumed to be equal to the far ambient in order to skip the complexity of oxygen diffusive and volatile convective behavior. Esfahani and Kashani [8] presented a model for degradation and combustion of polymethyl methacrylate.

Numerical simulation of thermal oxidative degradation of PE and effect of oxygen concentration on gasification, absoptivity and reflectivity of PE is the purpose of the present work. One dimensional model was used to predict thermal behavior of PE. This model includes different mechanisms which affects degradation process such as, in depth thermal oxidative decomposition, surface absorption and reflection of radiation, in depth absorption of radiation, conductive heat transfer, volatiles advection in solid phase, radiation from surface, and convective heat transfer on surface. Specific feature of the present work compare to all previous studies is char layer process, while extends applicability of the previous work regarding oxidative degradation [7].

Physical model

Physical model is gotten from Kashiwagi experimental work [3]. A piece of solid fuel (PE) is placed vertically and exposed to high radiative heat source from it left side while both sides are cooled by natural convection flow, therefore two boundary layer formed adjacent to the walls of sample. Length, height, and width of sample are 15 mm, 100 mm, 100 mm, respectively. Height and width of sample are about 6 times bigger than the length, so effect of these two dimensions in comparison with length of sample is negligible. This effect is presented in Esfahani's work [9]. The specimen is assumed to be in thermal balance with the ambient air before onset of heat radiation. To circumvent the complexity of the problem, effect of volatiles advection on oxygen distribution on PE surface has not been considered so the oxygen concentration on sample surface is assumed to be constant. This is particularly true when the mass flux of volatiles is small. As it can be seen in Kashiwagi experimental work [3], maximum value of mass flux that leaves PE surface is 0.5 mg/scm^2, which is about 30% of PMMA mass flux.

As the sample is being heated by external source, a portion of input energy is absorbed in depth by the sample and rest of it is reflected by PE surface. Besides, the degradation mechanism is activated through out the sample and release volatiles which form bubbles moving to the irradiated surface. With oxygen in the gas phase the sample behaves similarly to that in nitrogen environment until the first appearance of volatiles in

the solid phase. Shortly there after the color of the surface starts to turn brown and some bubbles are also observed. The surface color is darker with increasing oxygen content in the gas phase. A schematic of the problem geometry is depicted in fig. 1.
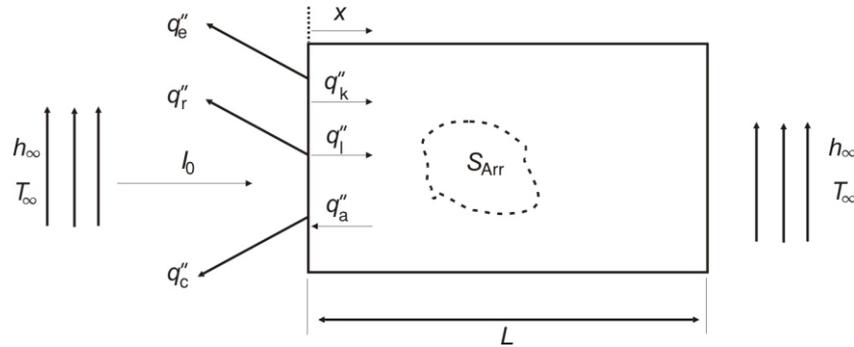

Figure 1. Physical configuration of the degradation problem

Assumption

Surface regression, storage of mass or energy, melting and dynamics of bubble movement are neglected in the present model. In addition all thermal properties of the fuel sample assumed to be fixed during the process. Oxygen concentration on sample surface is assumed to be constant. The length of the solid fuel is fixed and no thermal contraction or expansion is considered as time elapses in order to simplify the simulation. The virgin sample is assumed to be gray so absorptivity and emissivity are equal.

Mathematical model

The mathematical model follows the frame work of Esfahani [8] to simulate Kashiwagi and Ohlemiller's experimental studies [3] and is largely based on the model proposed by Ohlemiller [10].

According to Kashiwagi's finding [3] the rate of transient gasification can not be described simply as a function of surface temperature or proportional to energy input, in addition a model for predicting the gasification rate should include condensed phase oxidative chemical reactions. Despite this monitored significant findings, necessary improvements in modeling of gasification rate have not been taken for the last several decades. Brown and Kashiwagi [11] demonstrated that the greater amount of oxygen at the interface increases random scissions and generation of functional groups by which the transient gasification rate is enhanced. Therefore a proposed modified Arrhenius type

function has been utilized for PMMA in the previous work of Esfahani [7] which is extended for PE here. Similarly rate of gasification of PE is formulated as below:

$$m'' \quad A_s e^{\frac{E_s}{RT_s}} \rho_s (a \quad Y_{O_{surf}})^n \tag{1}$$

In the present work a and n are found as follow:

$$a = 1.5 \quad \text{and} \quad n = 0.8$$

The term in parenthesis in Arrhenius equation is the oxygen consideration factor. As the sample starts to degrade, released volatiles constitute bubbles, which grow and leave toward the direction imposing less resistance. The flux of these volatiles is calculated by integrating the gasification rate over solid region:

$$v_1 \quad \int_{l_s}^{x} m'' dx \tag{2}$$

Surface absorption term is considered as a source term in the boundary condition and formulated as below:

$$S = I_0 \alpha (1 - r) \tag{3}$$

Figure 2. Schematic of surface absorption and reflection

where I_0 and S are the intensity of radiative heat source and energy which is absorbed by char layer, on sample surface layer, respectively. Physical model of above formulation can be seen in fig. 2.

Radiative heat flux which enters the depth of PE can be formulated as below:

$$I = I_0 (1 - r)(1 - \alpha) \tag{4}$$

The absorption of radiation in depth of solid is formulated by the Beer's law and has the following form:

$$\frac{dI_s}{dx} \quad I\beta_s e^{\beta_s x} \tag{5}$$

where I is the intensity of the radiative heat source, which enters the PE, and β_s is the absorption coefficient.
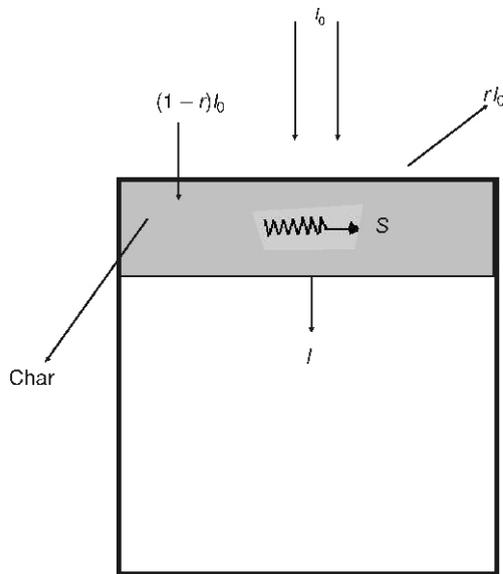
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Advection of volatiles, diffusion, and absorption of radiation and heat of degradation are mechanism dominating the energy conservation in the solid fuel and could be formulated in the following one dimensional form [7]:

$$c_s \ \rho_s \frac{\partial T_s}{\partial t} \ \upsilon_s \frac{\partial T_s}{\partial x} \ \kappa_s \frac{\partial}{\partial x} \frac{\partial T_s}{\partial x} \ \frac{dI_s}{dx} \ m \ L \tag{6}$$

Boundary conditions for above partial differential equation are:

$$x \ 0 \ \kappa_s \frac{\partial T}{\partial x} \ \varepsilon\sigma(T_{\text{surf}}^4 \ T_\infty^4) \ h_c(T_{\text{surf}} \ T_\infty) \ S \ 0 \tag{7}$$

$$x \ l_s \ \kappa_s \frac{\partial T}{\partial x} \ \varepsilon\sigma(T_{x \ l_s}^4 \ T_\infty^4) \ h_c(T_{x \ l_s} \ T_\infty) \tag{8}$$

$$t \ 0 \ T \ T_\infty \tag{9}$$

where h_c, T_s, and T_∞ are convective heat transfer coefficient on sample surface, surface temperature, and ambient temperature, respectively. Convective heat transfer coefficient is defined by Newton cooling law [12]. Using constant properties of air, convective heat transfer coefficient takes the following form:

$$h_c = 1.87(T_s - T_\infty)^{0.25} \tag{10}$$

As it was mentioned, color of sample surface turns brown, due to existence of oxygen. Change in color of surface leads to change in absorptivity and reflectivity of sample surface. Therefore, in the present work, absorptivity and reflectivity coefficient of PE surface are defined as a function of surface temperature and oxygen concentration, and are formulated as follow:

$$\alpha \ c_1 e^{\frac{c_2}{T_{\text{surf}} \ T_\infty}} (e^{c_3 Y_{O_{\text{surf}}}} \ 1) \tag{11}$$

$$\frac{r}{r_0} \ 1 \ c_4 e^{\frac{c_5}{T_{\text{surf}} \ T_\infty}} (e^{c_6 Y_{O_{\text{surf}}}} \ 1) \tag{12}$$

where α, r, $Y_{O_{\text{surf}}}$, and r_0 are absorptivity and reflectivity of sample surface, oxygen concentration adjacent to sample surface, and initial value of reflectivity, respectively. Value of constants in eqs. 11 and 12 are given in tab. 1. First exponent in eqs. 11 and 12 shows transient effect of char

Table 1. Constant values of eqs. (11) and (12)

c_1	c_2	c_3	c_4	c_5	c_6
810	3600	4	1000	2800	2

layer formation causes variation of reflectivity and absorptivity of PE surface, which depends on temperature variation. Term in parenthesis shows that, if oxygen concentration is equal to zero, darkening of surface layer will not occur.

Numerical model

The governing partial differential equations are discretized by utilizing the control volume formulation over the staggered grid region. A linearized equation is solved by using a fast iterative TDMA method [13]. In each time step, iteration will be done to achieve a proper convergence of (0.001 K) for energy equation. The mentioned computational process will be repeated in each time step and continues to the final stage of the problem. The number of allocated nodes in solid phase is 501 with time step of 0.1 s.

Properties of PE and other rated coefficient are selected from [3], [14], [15], and [16]. Data given in tab. 2, have been applied to the numerical model and assumed to be constant.

Table 2. Physical, thermal, and kinetics constants

Property	Value	Unit	Reference
ρ_s	801	kg/m^3	[14]
c_s	2300	J/kgK	[14]
κ_s	0.33	W/m^{2}K	[14]
L	414	kJ/mol	[14]
β_s	2300	l/m	[14]
ε	0.88	–	[15]
r_0	0.12	–	[15]
A_s	$3 \cdot 10^{16}$	l/s	[16]
E_s	2640	kJ/kg	[16]

Results

Figure 3 presents variation of mass flux at irradiation 40 kW/m^2. It can be seen that, degradation process for external source of 40 kW/m^2 begins at 85 s after irradiation of heat source. Mass flux suddenly increases at 96 s after irradiation for oxygen concentration of 40%. This sharp increase in mass flux occurs at 110 s after irradiation for 20% O_2 and coincides with the start time of the color change in the surface layer. As it can be seen in fig. 3, flux of volatiles leaving the interface is influenced by oxygen concentration. The lowest curve corresponds to

Figure 3. Mass flux for different oxygen concentration

0.0% O_2. This effect is explained in the context of modeling and expected regarding affiliation of modified Arrhenius function to oxygen content at interface. These results are harmony with Kashiwagi and Ohlemiller findings [3].

Variation of surface temperature for different oxygen concentration is depicted in fig. 4. As results show in fig. 4, surface temperature tends to higher plateau as oxygen concentration increases. According to the role of gasification, increase in oxygen concentration leads to higher rate of gasification, so more amount of absorbed heat is consumed for degradation. Beside of it, more increase in absorptivity of surface due to increase in oxygen concentration leads to more amount of heat absorption in the sample surface which correspond to experimental data. These two phenomena have opposite effect on surface temperature. But increase in surface absorptivity seems to be more effective. Therefore, as results show surface temperature increases due to increases in oxygen concentration. Also it can be found from the results in fig. 4, there is a sharp increase in surface temperature for oxygen concentration of 40 and 20%. For 40% O_2 surface temperature increases suddenly from 650 to 740 K at 96 s after irradiation, for 20% O_2 surface temperature increase suddenly from 650 to 715 K at 110 s after irradiation. Darkening of PE surface due to existence of oxygen in the ambient air is reason of this phenomenon. There is no sudden increase in surface temperature for 0.0 and 10% O_2.

Figure 4. Surface temperature for different oxygen concentration

Results of surface temperature, which are obtained from numerical model, are compared with experimental data [3] in tab. 3. As it can be seen in tab. 3, that predicted results are in the range of experimental values with the maximum deviation of 1.5% for surface temperature.
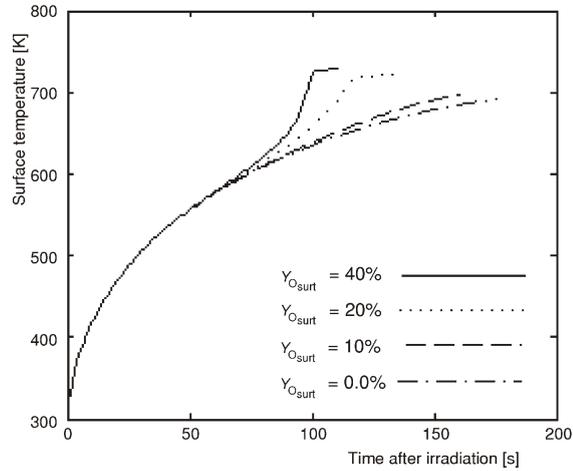
Table 3. Comparison of surface temperature at final stage which is obtained from numerical model and experimental data [3]

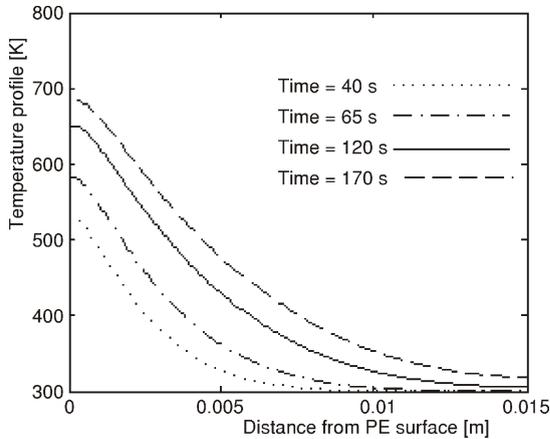| Oxygen concentration	Experimental [K], [3]	Numerical [K]	Difference
0.0%	680	685	0.7%
10%	680	690	1.5%
20%	705	715	1.4%
40%	730	740	1.3%

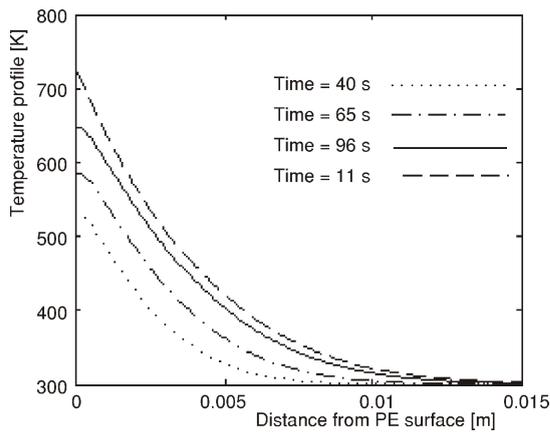**Figure 5. Temperature profile in PE for different time after irradiation at 0.0% oxygen**

Figure 6. Temperature profile in PE for different time after irradiation at 40% oxygen

In figs. 5 and 6 temperature profile within the solid fuel has been plotted for different time after irradiation. Figures 5 and 6 shows result for 0.0 and 40% oxygen concentration, respectively. It can be found by comparing these two figures that temperature distribution in PE does not vary with changing in oxygen concentration until 65 s after irradiation. This means that surface of PE has not had any change in color until that time.

Also it can be seen that, the back side of sample, has temperature of 330 K at 170 s after irradiation for 0.0% O_2 and 305 K at 110 s after irradiation for 40% O_2. Different exposure time, absorptivity, and reflectivity for 0.0 and 40% oxygen concentration are reasons of this difference. As it can be seen in experimental data [3], irradiation time for 40 and 0.0% oxygen concentration is 110 and 170 s, respectively. If irradiation continues, surface of PE is going to be molten. So test should be terminated before downward flow of molten surface. For 40% oxygen, melting of surface occurs more quickly than 0.0% oxygen concentration. According to fig. 4, surface temperature of PE for 40% oxygen is higher than surface temperature for 0.0% oxygen concentration, so melting of surface layer happens sooner for 40% oxygen, therefore test must be finished in advance.

Effect of absorption coefficient on surface temperature of PE is indicated in figs. 7 and 8. Figure 7 shows result for oxygen concentration of 40% and fig. 8 is for 0.0% oxygen concentration. It can be seen in fig. 7 that for β_s of 2300 m^{-1} surface temperature will be 740 K after 104 s of irradiation. By increasing β_s from 2300 to 4000 m^{-1}, energy which is absorbed by the layer near PE surface will increase, therefore surface temperature increases more quickly and it will be 740 K after 97 s of irradiation. Surface temperature

Figure 7. Variation of surface temperature via time for different values of absorption coefficient and oxygen concentration of 40%
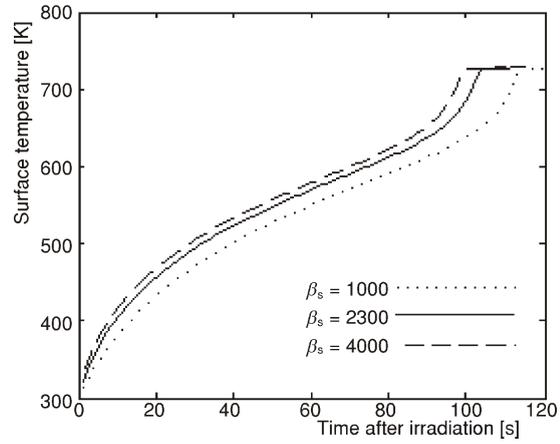

Figure 8. Variation of surface temperature via time for different values of absorption coefficient and oxygen concentration of 0.0%
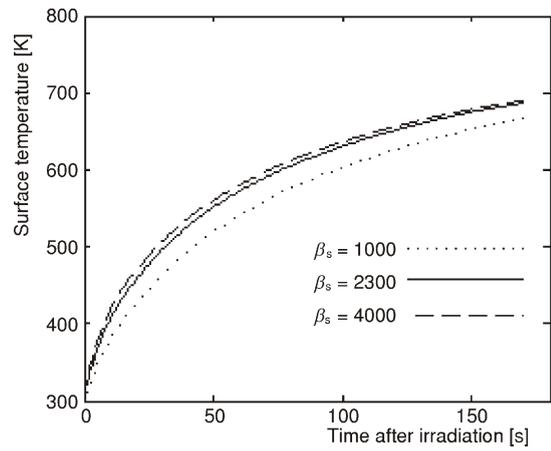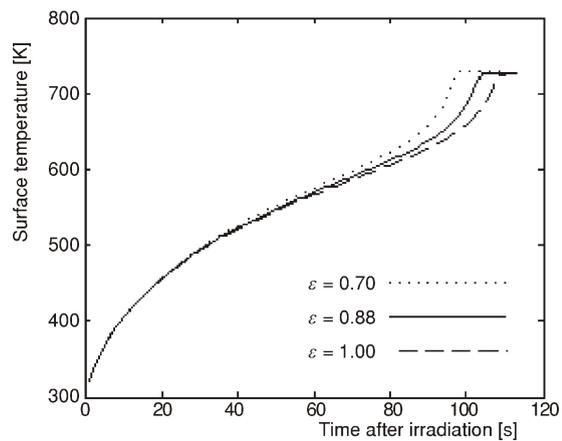

Figure 9. Variation of surface temperature via time for different values of surface emissivity and oxygen concentration of 40%

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reaches 740 K after 112 s for β_s of 1000 m^{-1}. The same results are yielded for oxygen concentration of 0.0% in fig. 8. Surface temperature after 170 s irradiation is 650, 675, and 680 K for β_s of 1000, 2300, and 4000 m^{-1}, respectively. When β_s increases, most of energy is absorbed in the layer near surface, therefore temperature of surface increases, but when β_s decreases, amount of energy which enter PE, diffuse to the deeper layer and it is distributed more in depth of PE, so surface temperature decreases when β_s decreases.
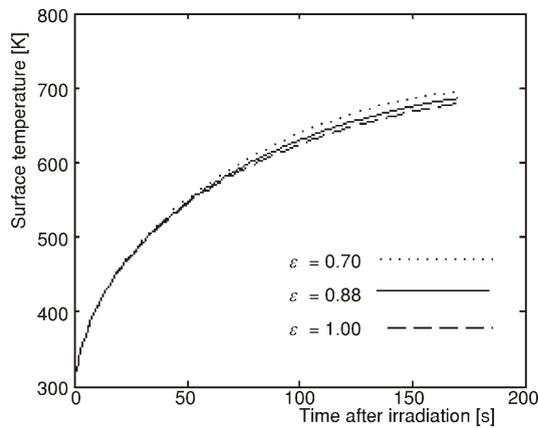

Figure 10. Variation of surface temperature via time for different values of surface emissivity and oxygen concentration of 0.0%
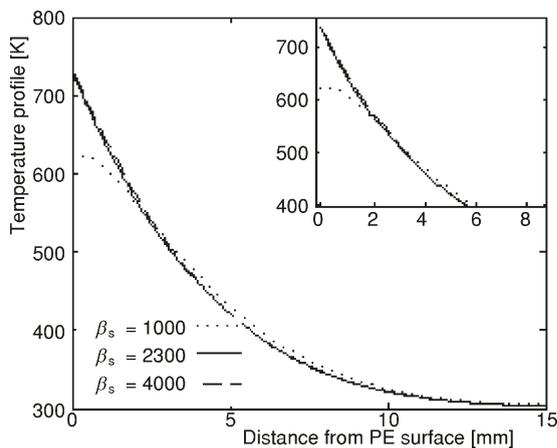

Figure 11. Temperature profile for different values of absorption coefficient and oxygen concentration of 40%

Figures 9 and 10 show surface temperature variation via time of radiation for different values of surface emissivity. Figure 9 shows results for 40% oxygen and fig. 10 for 0.0% oxygen concentration. When emissivity increases, amount of heat which leave PE surface by means of radiation, increases, therefore surface temperature decreases and *vice versa*.

It can be seen in fig. 9 that surface temperature reaches 740 K after 96, 104, and 108 s for surface emissivity coefficient of 0.7, 0.88, and 1.00, respectively. The Major effect of increase in emissivity is delay in time of reaching maximum surface temperature. For oxygen concentration of 0.0% variation of surface temperature via time for different values of emissivity is yielded in fig. 10. It is found that, change in emissivity from 0.88 to1.00 decrease maximum surface temperature from 675 to 670 K and reduction of it from 0.88 to 0.7 shifts maximum surface temperature from 675 to 685 K.

Temperature distribution in PE sample at final stage 110 s after irradiation is depicted in fig. 11 for different values of absorption coefficient and oxygen concentration of 40%. It can be seen that by decreasing β_s from 2300 to 1000 m^{-1} heat absorption in layers adjacent to surface decreases, therefore temperature profile from

surface to the depth of 2.5 mm falls to the lower values as compared to temperature profile of $\beta_s = 2300$ m^{-1} and it shifts up to the higher temperature from depth of 2.5 mm to the back side of sample. Increase in β_s from 2300 to 4000 m^{-1} leads to minor change of temperature in depth of PE sample.

Figure 12 shows temperature profile in PE sample at final stage 170 s after irradiation for different values of absorption coefficient and oxygen concentration of 0.0%. By increasing β_s from 2300 to 4000 m^{-1}, heat absorption coefficient at PE surface increases, therefore maximum temperature occurs at surface. By decreasing absorption coefficient, radiation can diffuse to the deeper layer of PE and maximum temperature shifts to the depth of sample. Also it can be seen in fig. 12 that decrease in β_s from 2300 to 1000 temperature profile from surface to depth of 1 mm falls to the lower values as compared to temperature profile of $\beta_s = 2300$ m^{-1} and it shifts up from depth of 1 to 15 mm to higher temperature.
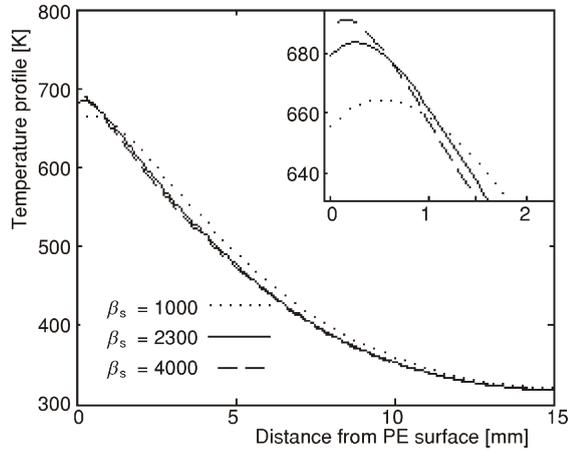

Figure 12. Temperature profile for different values of absorption coefficient and oxygen concentration of 0.0%

Figure 13 shows variation of surface absorptivity via time of irradiation for different values of absorption coefficient and oxygen concentration of 40%. For β_s of 4000, 2300, and 1000 m^{-1} surface absorptivity varies from 0 to 0.7, 0.68, and 0.22, respectively.
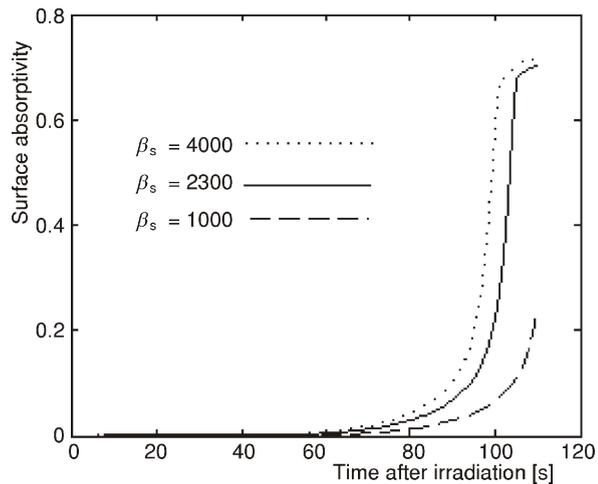

Figure 13. Variation of surface absorptivity for different values of absorption coefficient and oxygen concentration of 40%

Considerable variation of surface absorptivity (gradient = 0.01 s^{-1}) begins at 79, 90, and 107 s for β_s of 4000, 2300, and 1000, respectively.

Conclusions

At the present work an oxidative-thermal degradation model is developed, that highlights the effect of oxygen concentration and radiative parameters such as emissivity, absorptivity, and reflectivity on rate of gasification. Also variations of reflectivity and absorptivity of sample surface were presented in order to fulfill the surface darkening effect.

The following conclusion can be drawn based on the model:
– a modified Arrhenius type function was presented which comprise oxidative degradation PE sample exposed to the radiative source of 40 kW/m^2,
– an increase in oxygen concentration substantially increases the rate of gasification,
– an increase in the oxygen concentration leads to increase in absorptivity due to darkening of PE surface,
– increase in mass flux due to increase in oxygen concentration and increase in absorptivity of PE surface are two factors which are in competition with each other, The first one reduce the temperature and the second one increase the temperature, but increase in absorptivity is more effective, therefore temperature profile shift up due to increase in oxygen concentration,
– sharp increase of surface temperature was found for oxygen concentration of 40 and 20%. But this sharp increase is more considerable in 40% oxygen. Darkening of PE surface due to existence of oxygen in the ambient air is reason of this phenomenon,
– the predicted results are in the range of experimental values with the maximum deviation of 1.5% for surface temperature,
– when emissivity increases, amount of heat which leave PE surface by means of radiation, increases, therefore surface temperature decreases and *vice versa*, and
– by increasing β_s, heat absorption in layers adjacent to surface increases, therefore temperature profile from surface to the depth of 1.5 mm shifts up to the higher values and it falls to the lower temperature from depth of 1.5 mm to depth of 15 mm. Reduction of β_s leads to increase of temperature in depth of PE sample.

Despite of complexity of PE degradation process, simplified model which is used in the present work to simulate this process seems to be successful. The model shows deviation from the experimental results and needs further improvement in the future, however it can still describe the general trend of the physical processes and has the advantage of being very simple and results could be achieved with reasonable accuracy and quickness.

Nomenclature

A_s – pre-exponential factor of reaction, [s^{-1}]
c_s – specific heat of solid-phase, [Jkg^{-1}K^{-1}]

E_s – activation energy, [Jkg^{-1}]
h_c – convection heat transfer coefficient, [Wm^{-2}K^{-1}]
I_0 – intensity of radiative heat source, [Wm^{-2}]
κ_s – thermal conductivity of solid-phase, [Wm^{-2}K^{-1}]
L – heat of degradation, [kJmol^{-1}]
l_s – length of solid fuel, [m]
m'' – rate of gas production in each cell, [kgm^{-2}s^{-1}]
q'' – heat flux, [Wm^{-2}]
R – gas constant, [kJkg^{-1}K^{-1}]
r – reflectivity, [–]
r_0 – reflectivity of virgin material, [–]
T – temperature, [K]
t – time, [s]
v – local mass flux of volatiles, [kgm^{-2}s^{-1}]
x – coordinate [m]
$Y_{O_{surf}}$ – oxygen mass fraction on surface, [–]

Greek letters

α – absorptivity of PE, [–]
β_s – absorption coefficient of solid PE, [m^{-1}]
ε – emissivity, [–]
ρ_s – density of solid, [kgm^{-2}]
σ – Stefan-Boltzmann constant, [–]

Subscripts

c   – conducted
e   – emitted
l   – local
r   – reflected
s   – solid
O   – oxygen
surf   – surface

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