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Author

Pitz, W. J.

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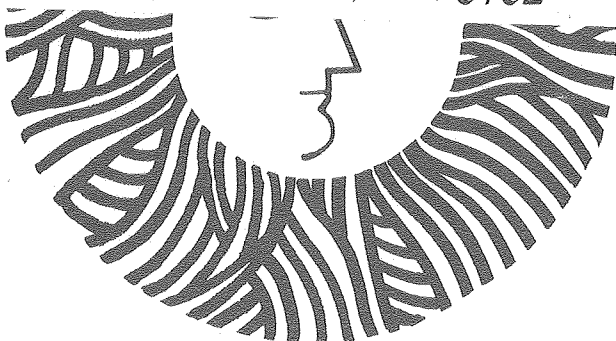
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W. J. Pitz, N. J. Brown, and R. F. Sawyer

October 1979

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FLAME STRUCTURE MEASUREMENT OF POLYMER DIFFUSION FLAMES*

W.J. Pitz
N.J. Brown
R.F. Sawyer

Department of Mechanical Engineering and
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

ABSTRACT

Flame structure measurements of polyethylene/opposed flow diffusion flames have been obtained under steady-state conditions as extinction was approached. The study indicates the following results. The CO/CO₂ ratio increases slightly as extinction is approached which is in agreement with the work of Maček (1975). Carbon monoxide significantly penetrates the luminous flame zone which is a departure from the flame sheet approximation. The composition profiles are similar within experimental error, thus justifying the use of the similarity assumption for modeling studies in the opposed flow configuration.

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1. INTRODUCTION

The increasing use of polymers has focused attention on the fire hazard arising from their inherent flammability. We anticipate that a better understanding of the mechanism of polymer combustion, particularly in diffusion controlled combustion characteristic of actual fires, will improve the ability to reduce polymer flammability. This study treats the polymer diffusion flame structure, particularly near extinction conditions.

Previous work on the structure of a polymethylmethacrylate (PMMA) opposed flow diffusion flame was reported by Seshadri and Williams (1977). The present work treats the combustion of polyethylene. Unlike PMMA, which depolymerizes almost entirely to its monomer upon vaporization, polyethylene yields only about 0.1% monomer upon vaporization (Wall, 1972). The work of Tsuiji and Yamaoka (1969) on the composition profiles of an opposed flow diffusion flame of gaseous propane and methane provide a valuable comparison for our investigation.

The idealization of a "collapsed flame zone" has provided a useful model of the diffusion flame (Williams, 1965). Beyond the one-step reaction of this flame model, the chemistry has not been well established. The chemistry in a diffusion flame becomes particularly apparent at the extinction condition where the heat and mass transfer rates become comparable to the chemical reaction rate. In this work, the chemical flame structure near extinction was studied. Maček (1975) proposed that the overall stoichiometry of the flame proceeds primarily to CO at extinction. After performing a series of equilibrium calculations assuming either CO or CO₂ stoichiometry, he found that the assumptions of CO stoichiometry gave better agreement between calculated and expected variation in the flame temperature along the flammability limit curve. Thus the CO/CO₂ ratio at extinction

is approached is of interest and was determined in this work.

Composition profile similarity for the opposed flow diffusion flame was determined in this study which, from the literature survey conducted, had not been checked previously. Since boundary layer theory predicts similar velocity profiles for the stagnation boundary layer flow, one might also expect to find similar composition profiles experimentally. The assumption of profile similarity greatly reduces the complexity of theoretical models for diffusion flames.

The second part of this paper describes the opposed flow diffusion flame apparatus, the molding of polymer samples, and the combustion gas analytical system. In the third part, the fluid dynamic characteristics of the nozzle, the reproducibility of the flame species profiles, and the composition profiles through the flame are presented. The salient characteristics of the composition profiles such as the CO/CO_2 ratios, CO and O_2 penetration of the flame, profile similarity, and extinction characteristics are discussed in the fourth part where the major conclusions of the study are also given.

2. EXPERIMENTAL SYSTEM

Opposed Flow Diffusion Flame Apparatus

Steady-state polymer combustion was studied in an opposed flow diffusion burner, which is illustrated schematically in Figure 1. The apparatus will be described briefly in subsequent paragraphs.

An oxidizing mixture of nitrogen and oxygen flowed in a downward direction through an aerodynamic nozzle to the surface of a polymer sample. The 12.7 mm diameter cylindrical polymer sample was driven upward by a stepping motor which was part of an automatic feed control designed to maintain the surface of the sample in a fixed position relative to the nozzle. The beam of a helium-neon laser was used to sense the position of the polymer surface. The laser beam skimmed over the polymer surface and impinged on a photo-diode detector when the polymer sample had regressed. When the sample was located the proper distance from the nozzle, the laser beam was blocked by the sample and no signal was detected by the photo-diode. A positive photo-diode response activated a stepping motor which advanced the sample to the proper position. The automatic feed control allowed the investigation of steady-state polymer combustion and the measurement of polymer regression rates.

The oxidizer flow metering system consisted of a rotameter and a Hasting electronic mass-flow meter. The meters were calibrated individually at conditions simulating operating conditions by wet test meters which were checked with a 2832 cm^3 (0.1 cu.ft.) standard bottle. The pressure of the gases flowing through the meters was monitored. After measuring, the oxidizer gases were mixed in a tee and by passage through stainless-steel wool packing in the upstream end of the nozzle.

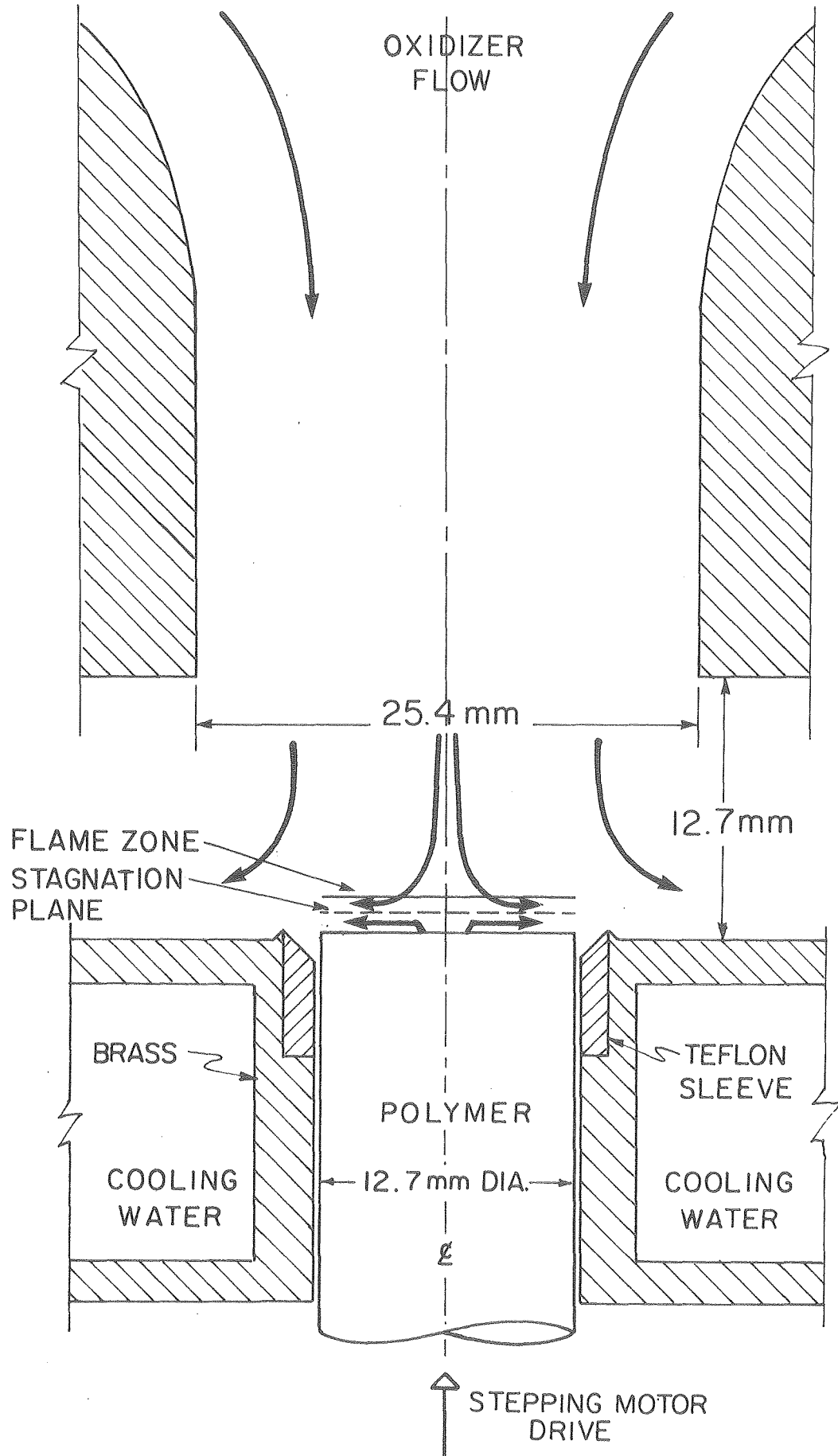


Figure 1. The opposed flow diffusion flame burner

The design of the aerodynamic nozzle was based upon wind tunnel design criteria (Morel, 1975). The nozzle had a large area contraction ratio (9.4) to reduce turbulence and nonuniformity in the exit velocity profile. The radius of curvature near the nozzle exit was large, thereby avoiding flow separation near the exit. An additional exit diameter of straightening section was located downstream of the contraction section to reduce the higher velocities expected near the walls. Velocity profile measurements at the nozzle exit were made with a hot wire anemometer which employed a 10 μ m diameter hot wire (10% rhodium, 90% platinum) connected to a TSI model 1010 heat flux system and a TSI model 1005 B linearizer.

Sample Preparation

Reproducibility of polymer properties such as density and chemical composition can not be assured in commercially obtained polymer rods. Since manufacturing details are proprietary, information regarding impurity identity and concentration, manufacturing conditions and quality control are lacking. The polymer samples used throughout were molded in the laboratory to obtain pure samples which burned reproducibly. Low density poly(ethylene) in pellet form was obtained from Aldrich Chemical. The polymer was placed in a stainless steel picture frame mold, heated in an oven at 180C for 2.7 hours, and immediately pressed with a hydraulic press to 3.2 MPa absolute. Mylar rather than silicon oil was used as a mold release since mylar did not penetrate into the polymer. The molded polymer was then cut into bars which were turned on a lathe to produce cylindrical samples 12.7 mm in diameter.

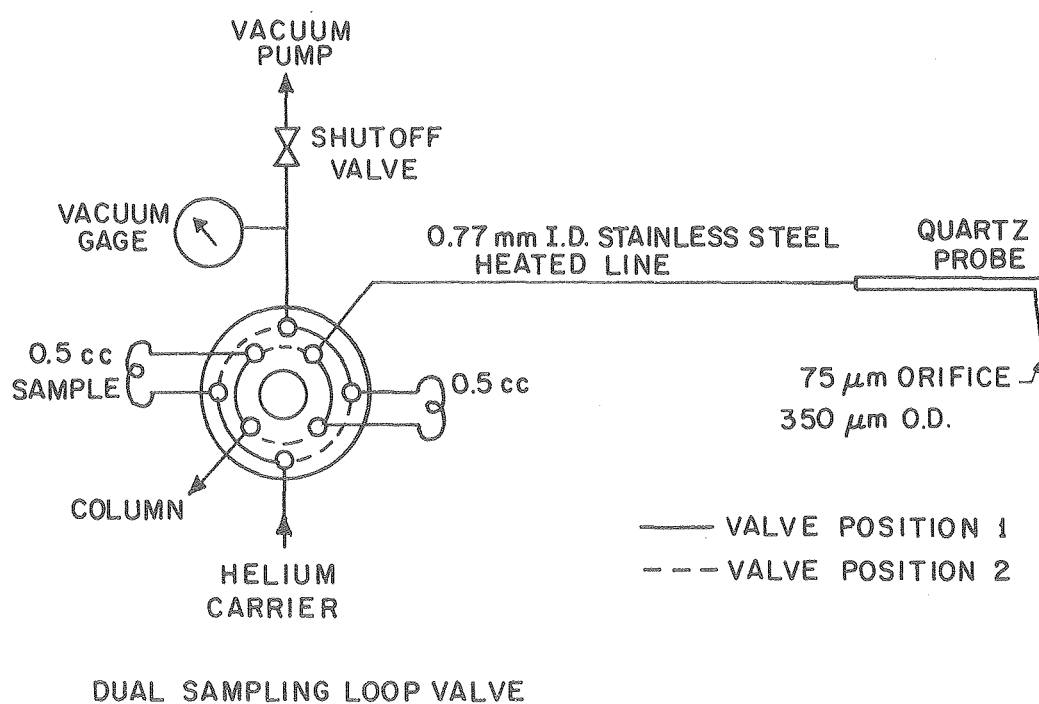
Gaseous Product Analysis

The analytical system used to determine flame composition consisted of

a quartz microprobe, sample manifold, a dual column gas chromatograph with dual detectors, and a microprocessor for peak integration. A more specific description of the components of the analytical system is given in subsequent paragraphs.

Continuous sampling was used throughout the study. As illustrated in Figure 2, samples were extracted from the flame with a quartz microprobe with a small orifice of 75 μm diameter. This small probe was utilized to obtain maximum spatial resolution in the flame and to obtain aerodynamic quenching of the combustion gases. The probe position was controlled by a Brinkman MS-II-E three-axis micrometer positioner which was accurate to 0.01 mm. The probe to surface reference distance was determined optically. The error in determining the reference distance was estimated to be ± 0.05 mm. After extraction from the flame, the sample flowed through a heated sampling line to a sampling valve on the gas chromatograph. Dual sample loops permitted continuous sample purging of the system thus allowing maximum time for the sample to equilibrate with the tubing walls. Prior to injection, the sample was pressurized to atmospheric pressure. The loop temperature was measured by a thermocouple. No pressure gauges or tees were attached to the sampling line so that the line had no dead volume and thus rapid purging was possible.

A Hewlett Packard 5162 gas chromatograph (G C) was modified to include a column switching system which permitted one to separate a complex gas mixture with one injection at a significantly reduced analysis time. The sample was injected with a Poropak Q column and Molecular Sieve 5A (MS 5A) column in the "series" position (Figure 3). After the light gases N_2 , O_2 , CO and CH_4 had been convected onto the MS 5A column, the valve was switched into the "store" position. While the light gases were



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Figure 2. Gas sampling system

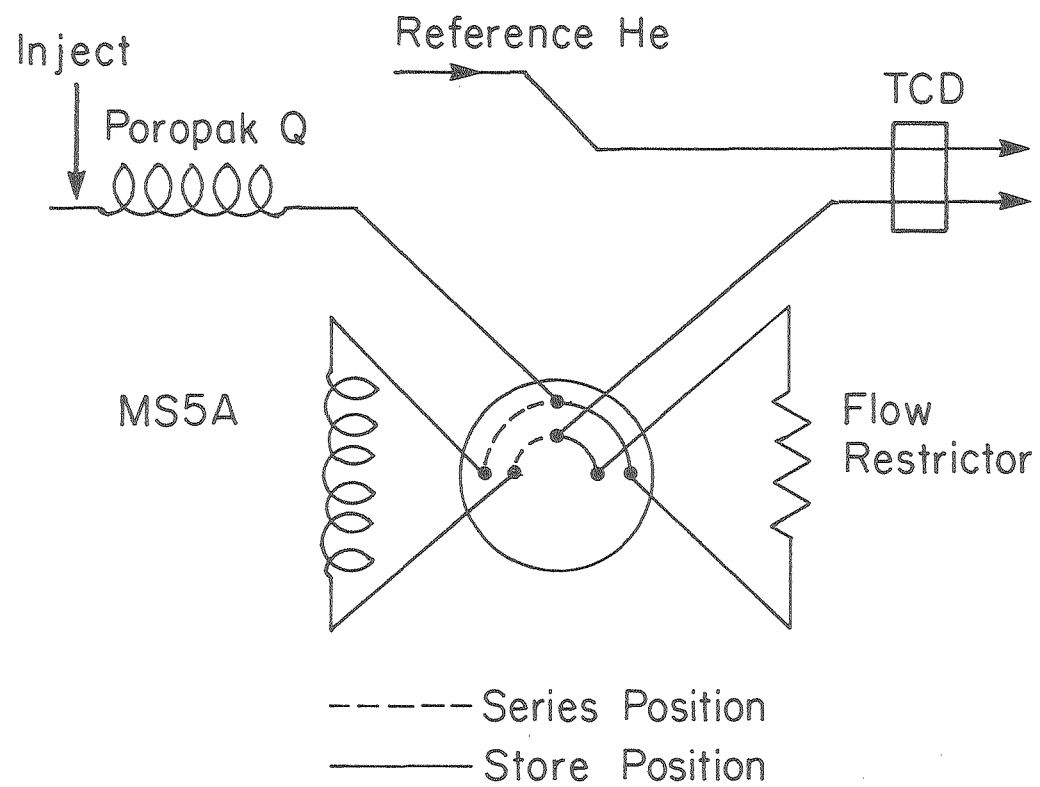


Figure 3. Column switching system

stored in the MS 5A, the CO₂, H₂O and hydrocarbons were eluted from the Poropak Q column into the TC and FID detectors. The flow restrictor replaced the flow resistance of the MS 5A, thereby reducing TC baseline shift attributed to valve switching and avoiding FID flameout. The valve was then switched back to the "series" position and the light gases were eluted from the MS 5A. The temperature of the GC lines and valves was monitored and maintained above the dew point temperature with heaters. This reduced the tailing of the water peak.

The peaks from both the TC and FID detectors were simultaneously integrated with an Autolab System IV integrator. The detector signals entered an analog to digital convertor contained in each detector channel module and were transmitted to the System IV microprocessor which computed the retention times and peak areas. The microprocessor also automatically updated the integration parameters and activated the column switching valve at preset times.

Reliable composition measurements are dependent upon the accuracy of the calibration procedures. Stainless steel, anodized vessels fitted with bellows valves were fabricated and used to store the calibration gases. Calibration gases were mixed in the laboratory using the partial pressure technique and the concentrations were verified with mass spectrometry. The GC was calibrated before and after each run.

3. RESULTS

Experimental results are presented in this section. The characteristics of the nozzle and the precision of the composition measurements are presented and discussed. The section is concluded with a presentation of the five composition profiles determined in the study.

Nozzle Flow

Velocity and turbulent intensity profiles were measured for a range of velocities at the nozzle exit. Figure 4 is a typical profile which gives velocity and turbulent intensity as a function of radial position for an exit velocity of 2.6 m/s. Radial position is measured from the centerline of the nozzle. The velocity profile is flat to within a 3% maximum deviation over the region encompassing the sample. The standard deviation of velocity over the sample region is 1.5%. This is an improvement over the screen nozzle used for preliminary work which gives a maximum deviation of 11% over the sample region. Since a single spray tube is used to introduce gas into the nozzle, slightly more gas is injected from the upstream than the downstream holes, and this results in the slightly skewed velocity profile. The average turbulence intensity in the region over the sample is 0.55% and thus indicates laminar-type flow. A uniform nozzle flow is important in the study of extinction. Non-uniformities lead to localized blow out and ill-defined extinction velocity.

Composition Measurements

There are two major sources of error in the composition profiles; one is associated with the reproducibility of the gas chromatography measurements and the other results from the uncertainties in the measurement of probe to surface distance. Calibration gases were mixed to simulate compositions in the flame. Multiple samples of each calibration gas were injected into the GC to determine the level of precision of individual species measurements. The errors in precision associated with each major species is indicated in Table 1. The reproducibility of N_2 and CO are within 1%, and CO_2 , CH_4 , and C_2H_6 are reproducible to within 3% of their concentrations.

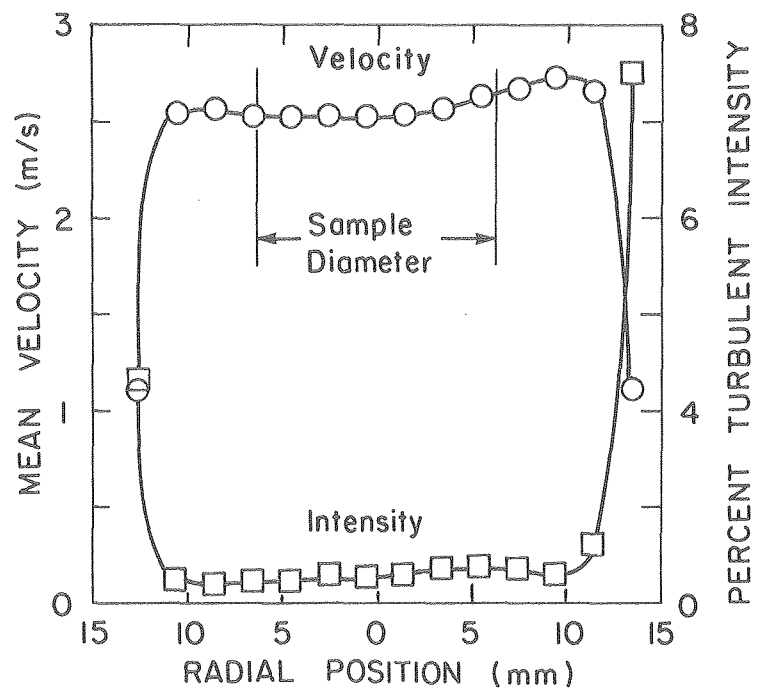


Figure 4. Velocity and turbulent intensity profiles at the nozzle exit

TABLE 1
GAS CHROMATOGRAPH REPRODUCIBILITY

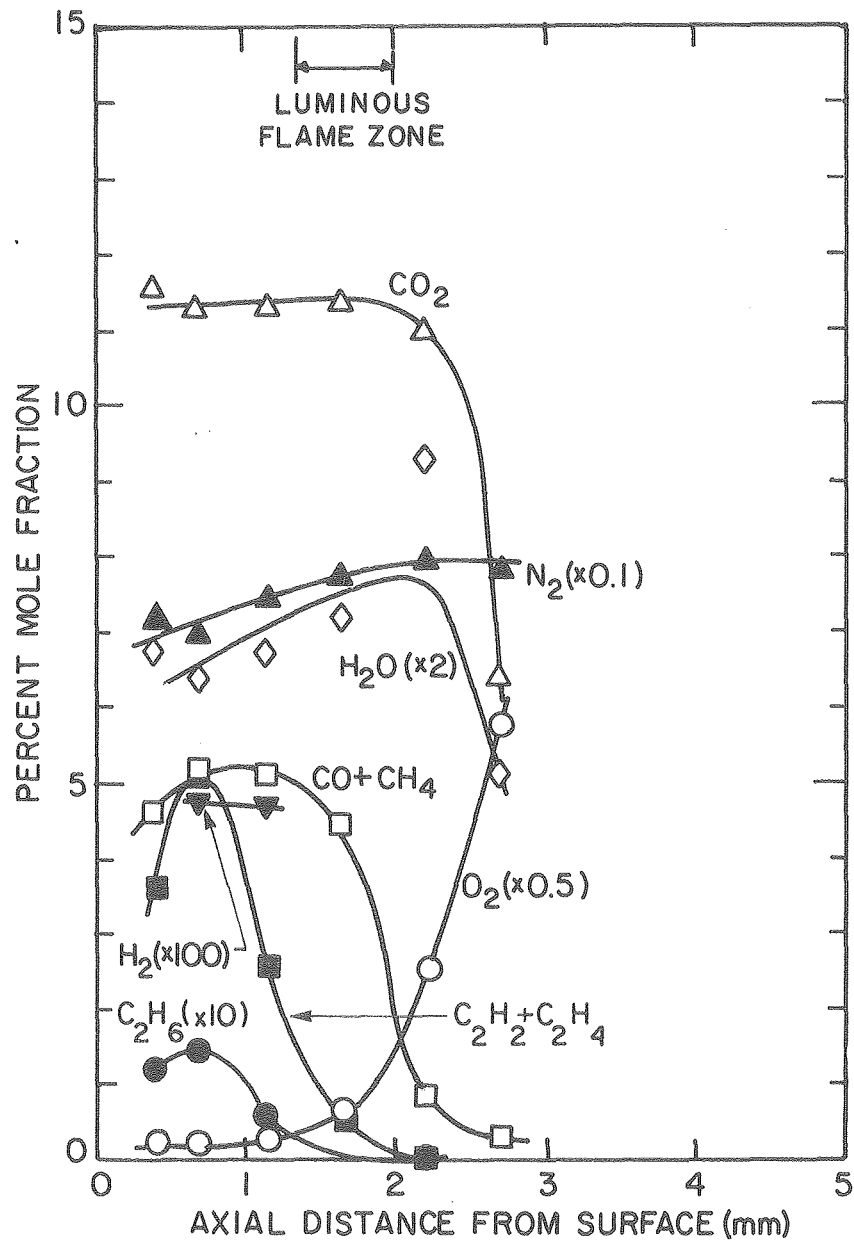
<u>Component</u>	<u>Percent Standard Deviation</u>	<u>Typical Concentration Percent Mole Fraction</u>
N ₂	0.8	80.
CO	0.9	6.
CO ₂	2.	8.
CH ₄	3.	0.5
C ₂ H ₄	2.	0.5
C ₂ H ₆	3.	0.2

The probe-surface distance of interest is the distance between the melt on the polymer surface and probe. Char nodules form on the melt and they, rather than the melt, are sensed by the helium laser/photo diode system. This improper designation of the polymer surface results in a distance measurement that is erroneously low.

Five composition profiles have been determined and these are shown in Figures 5 through 9. The average nozzle exit velocity for all cases was 0.48 m/s. The abscissa is the distance measured perpendicular from the polymer surface. Each composition was measured at a constant reduced radial position $r^* = r/R$, where r is the radial position and R is the radius of the polymer sample. Two values of r^* were selected for study. The ordinate is concentration expressed as percent mole fraction. Individual species curves are expanded by the number indicated in parentheses on the profiles. The uncertainties in the species, H_2 and H_2O , have not been quantified. The composition profiles in Figures 6 and 7 were taken at the same conditions to check reproducibility. Comparison of Figures 7 and 8 provides information on the effect of varying r^* .

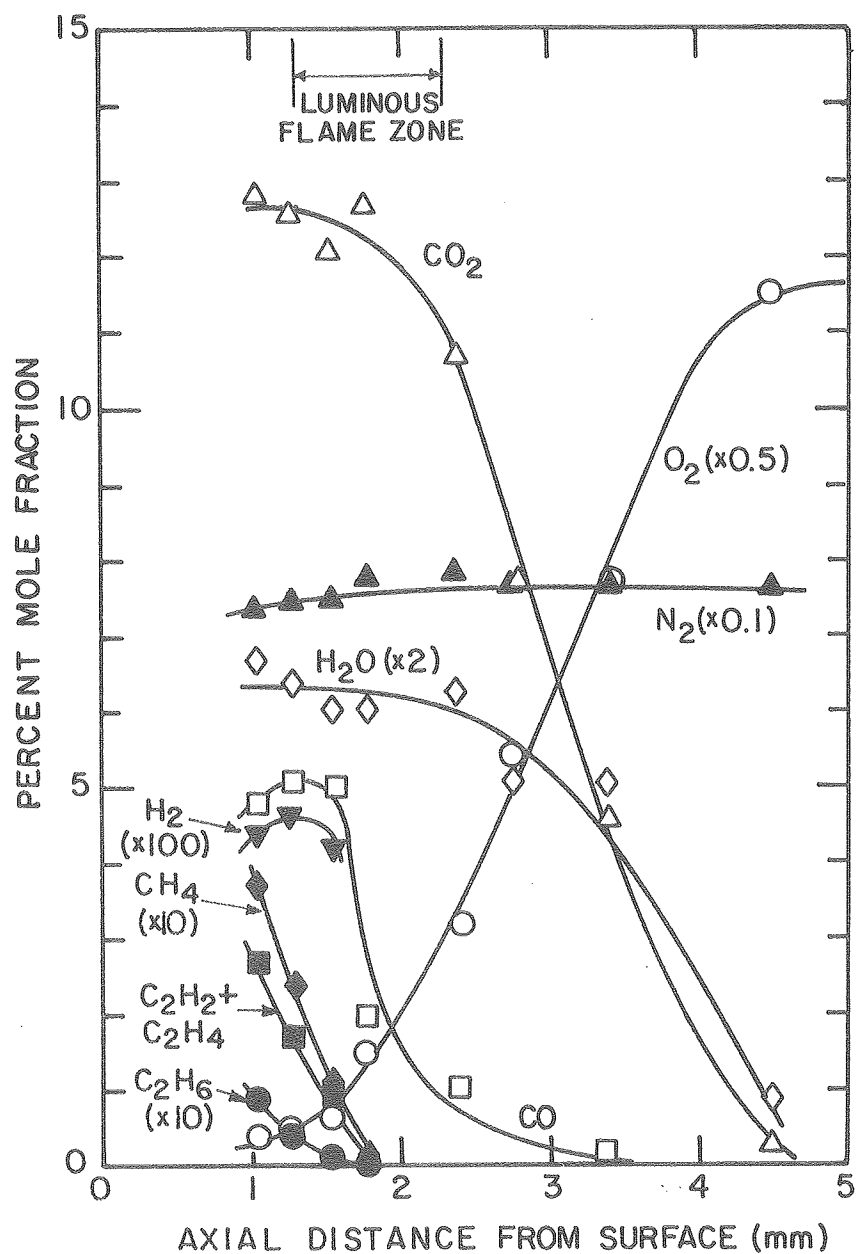
The location of the luminous flame zone was determined by visually identifying the region and by knowing the probe to surface distance. The luminous zone tends to increase with initial oxygen concentration since the yellow soot layer portion of this zone increases accordingly.

The profiles exhibit rather interesting features which are summarized in Table 2. The CO/CO_2 ratio of the peak maxima increases slightly as extinction is approached, that is, the ratio increases with decreasing initial oxygen concentration. To obtain the CO/CO_2 ratio for the $X_{O_2} = 0.212$ flame, it was necessary to subtract the CH_4 mole fraction from that of CO since the two peaks eluted as one due to low column efficiency. At the CO maximum, the value of the CH_4 peak of 0.3% associated with the $X_{O_2} = 0.253$ flame is used as the upper limit for the $X_{O_2} = 0.212$ case where fuel mole



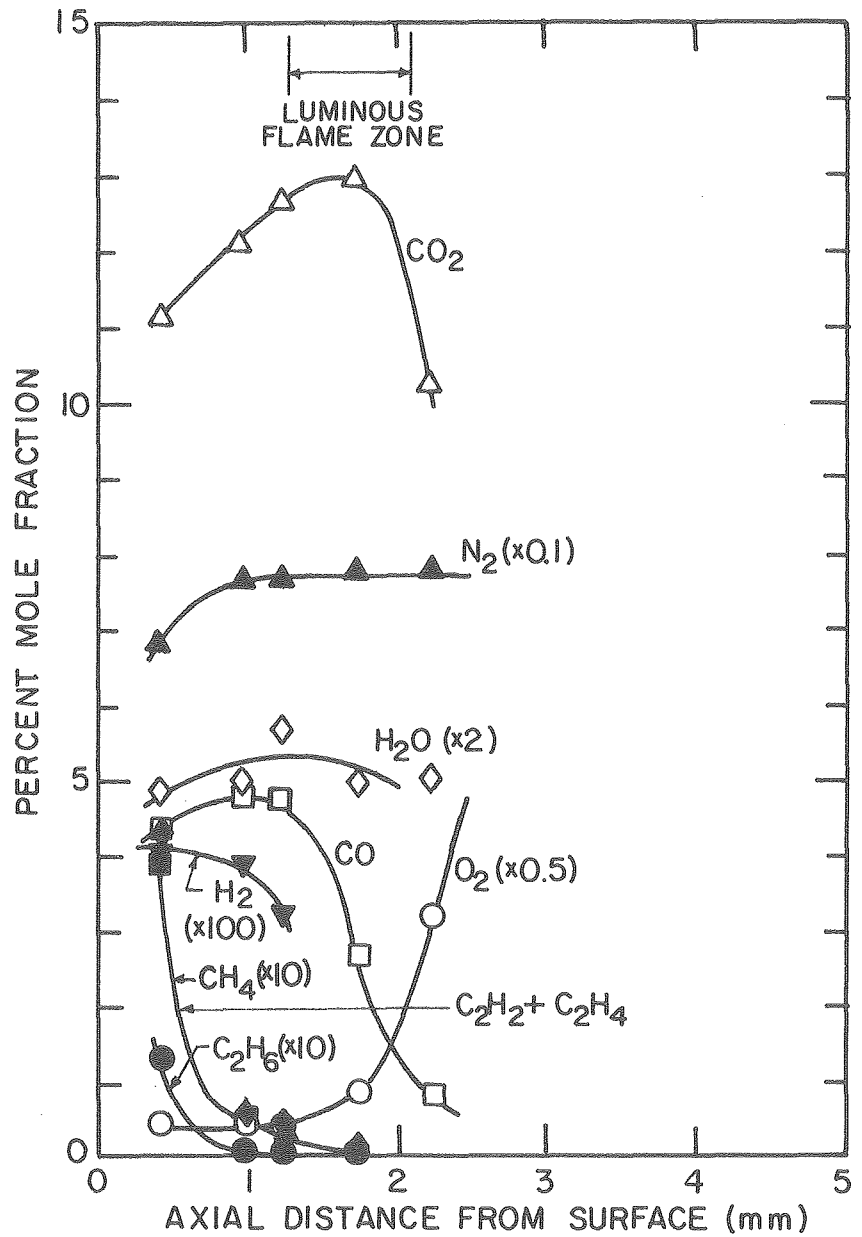
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Figure 5. Composition profiles for low oxygen concentration in the oxidizer ($X_{O_2} = 0.212$ and $r^* = 0.31$)



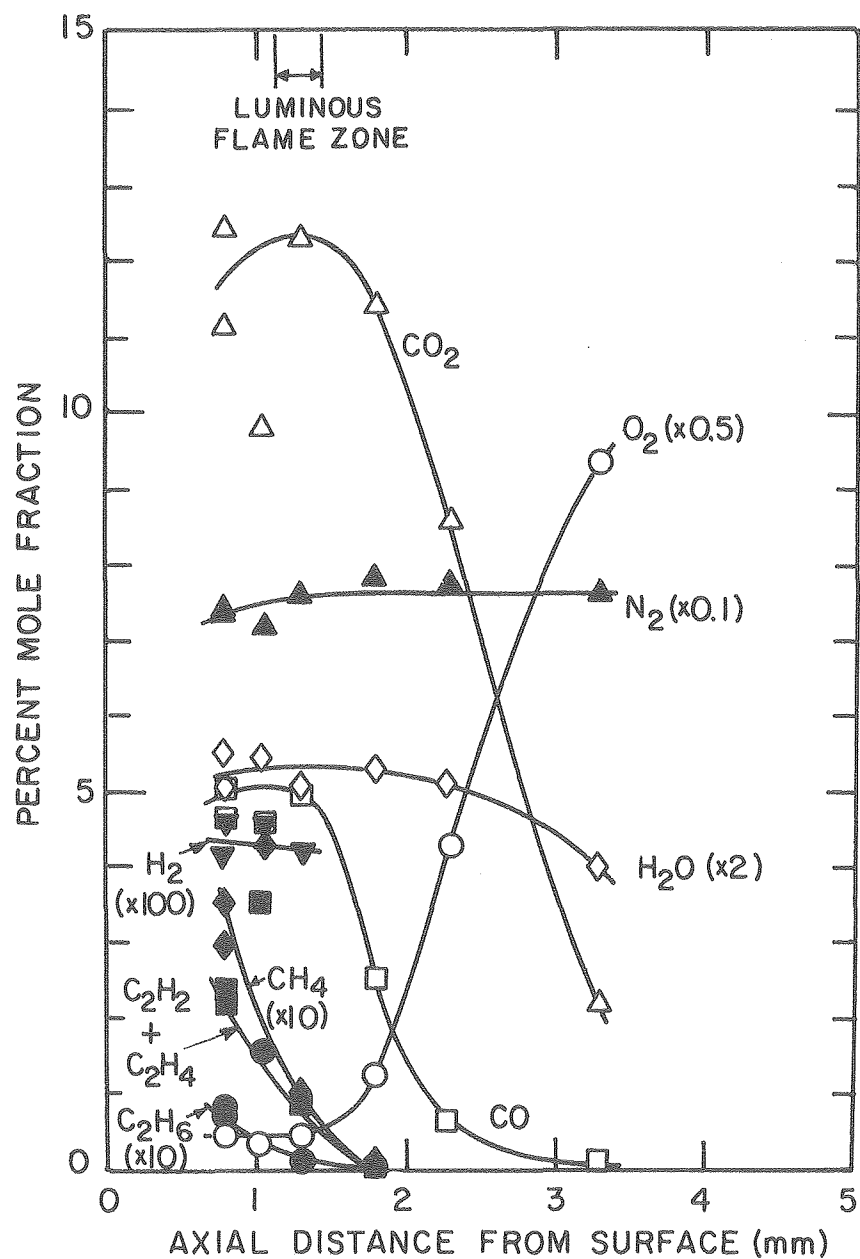
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Figure 6. Composition profiles for an intermediate oxygen concentration in the oxidizer ($X_{O_2} = 0.232$ and $r^* = 0.31$)



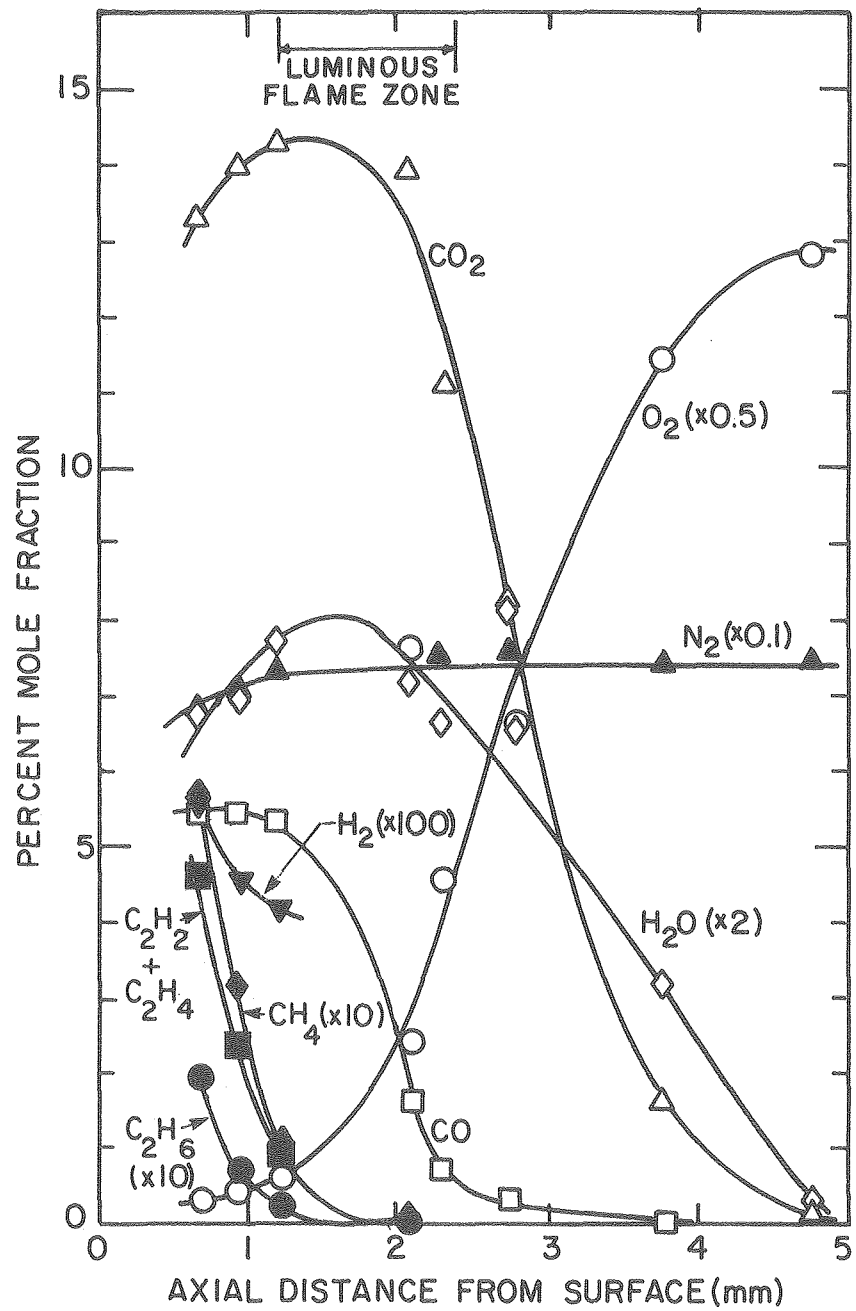
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Figure 7. Composition profiles for an intermediate oxygen concentration in the oxidizer ($X_{O_2} = 0.232$ and $r^* = 0.31$)



XBL 798-6764

Figure 8. Composition profiles for a large radial position ($X_{O_2} = 0.232$, $r^* = 0.67$)



XBL798-6731

Figure 9. Composition profiles for a high oxygen concentration in the oxidizer flow ($X_{O_2} = 0.253$, $r^* = 0.31$)

TABLE 2
CASES CONSIDERED AND PROFILE CHARACTERISTICS

<u>Oxidizer Oxygen Mole Fraction</u>	<u>Radial Location (r/R)</u>	<u>X_{O₂} at Surface</u>	<u>X_{O₂} at Luminous Flame Zone</u>	<u>CO/CO₂ Peak Ratio</u>	
0.212	0.31	0.3%	1%	0.43	
0.232	0.31	0.6%	3%	0.40	} 0.39 Average
0.232	0.31	0.8%	2%	0.37	
0.232	0.67	0.9%	1%	0.41	
0.253	0.31	0.5%	4%	0.38	

fractions are observed to be slightly less than the "0.253 case." The corrected value of CO is used to determine the CO/CO₂ ratio given in Table 2.

For the cases considered, CO penetrates the luminous flame zone; however, the remaining fuel species (H₂ being inconclusive) decrease rapidly to near zero concentration at the luminous flame zone. Less than 4% O₂ penetrates the luminous flame zone from the oxidizer side and less than 0.8% is found near the polymer surface. The gradient of oxygen concentration is small in all cases. For those cases where peaks could be discerned, the CO peak maximum is closer to the polymer surface than the maximum of the CO₂ peak.

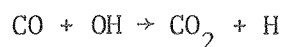
4. DISCUSSION

The CO/CO₂ ratio increases on the average for decreasing nozzle oxygen concentration as conditions closer to the extinction limit are approached (Table 2). The CO/CO₂ ratio increase is substantiated further by the shape of the nitrogen profile which decreases most rapidly from the luminous flame zone to the surface for the case closest to extinction (Figure 5). The decrease in nitrogen mole fraction results from the net mole change from reactant to product. A net increase in the number of moles in going from reactant to product results with the assumption of CO stoichiometry. If proportionally more CO is produced, the number of moles of product increases affecting a corresponding decrease in the N₂ mole fraction.

The same increasing trend in CO/CO₂ ratios as extinction is approached is seen in data from the opposed flow diffusion flame work of Seshadri

and Williams (1978), and Tsuji and Yamoaka (1969) (Table 3). The CO/CO₂ increase provides some support of Maček's (1975) proposal that gas-phase reactions in flames proceed primarily toward CO stoichiometry at extinction. Maček performed a series of equilibrium calculations assuming either CO or CO₂ stoichiometry, and the assumption of CO stoichiometry gave better agreement between calculated and expected variation in flame temperature along the flammability-limit curve. The decreased production of CO₂ in a flame near extinction is a result of insufficient reaction time for conversion of CO to CO₂. If theoretical models are to better predict extinction, an intermediate reaction describing the conversion of CO to CO₂ should be included.

Some distinctive characteristics of the composition profile are discussed in the subsequent paragraphs. The CO₂ concentration maximum lies to the oxidizer side of the CO concentration maximum (Figures 5, 7-9). The conversion of CO to CO₂ occurs principally via the following reaction (Glassman, 1977).



Since the CO maximum is on the oxidizer side of the stagnation plane, the CO must diffuse against the oxidizer flow to the region of the CO₂ maximum where the CO is subsequently converted to CO₂. The location of the CO oxidation region is in agreement with reaction rate profiles determined for an opposed flow diffusion flame with methane fuel (Tsuji and Yamoaka, 1971).

Another interesting characteristic of the CO profile is that CO penetrates the luminous flame zone in significant concentrations. The penetration is a striking departure from the flame sheet approximation and further emphasizes that a reaction describing the oxidation of CO to CO₂ should be included in models for diffusion flames. Many different

TABLE 3

<u>TRENDS IN CO/CO₂ RATIOS</u>			
Researchers	<u>CO/CO₂</u>	<u>Ratio</u>	Comments
	Far away from extinction	Closer to extinction	
Current study	0.38	0.43	-----
Tsuji and Yamoaka (propane fuel, $-f_w$ = 0.8)	0.72	0.79	Oxidizer is air and propane flow rate is constant
Seshadri and Williams	0.55	0.60	Nozzle velocity constant.

fuel species are found on the fuel side of the flame: CH_4 , C_2H_2 + C_2H_4 , C_2H_6 , H_2 , and CO (Figures 5-9).

According to the literature survey conducted, similarity of the composition profiles of a flame in a stagnation-point flow has not been verified experimentally. Since the velocity profiles of a stagnation-point boundary layer are similar, one might also expect similarity of the composition profiles. Composition profiles were taken at a small radial position (Figures 6 and 7) and a large radial position (Figure 8). The profiles are similar within experimental error thus justifying the use of the similarity assumption for modeling studies in the opposed flow configuration.

One method of approaching extinction is to decrease the oxygen concentration in the nozzle flow at constant nozzle exit velocity. This method is used throughout this study. A second method of approaching extinction is to increase the nozzle velocity for a fixed oxygen concentration. It is of interest to compare the two methods. The nondimensional Damköhler number which is the ratio of flow time to reaction time is especially useful for this purpose. When the flow time and reaction time become comparable, the flame extinguishes.

In this study where the first method was used, the luminous flame zone has a lower oxygen concentration for flame conditions approaching extinction, and this behavior is shown in Table 2. This is in agreement with the results of Tsuji and Yamoaka (1969). These results suggest that this method of approaching extinction increases reaction time by decreasing the oxygen concentration in the flame zone. Using this method, Seshadri and Williams found the opposite trend, namely that the O_2 concentration in the luminous flame zone increased when extinction is

approached. As extinction is approached via method two, an increase in the oxygen concentration at the luminous flame zone is indicated (Tsuji and Yamoaka, 1969). Although the oxygen concentration is sufficient for reaction, the flow time is too small to allow adequate reaction time. Although the two approaches to extinction are different, extinction occurs for both methods due to insufficient time for reaction.

A crucial question in polymer combustion is whether oxygen diffuses to the surface and reacts heterogenously to provide significant energy release for polymer pyrolysis. Stuetz et al. (1975) have proposed that polymer surface oxidation reactions provide significant energy for pyrolysis. The oxygen concentration gradient and oxygen concentration near the surface are examined in the current study to address this question. The difference between the oxygen diffusion rate to the surface and the rate at which oxygen is transported away from the surface by the pyrolyzing polymer is calculated. If the assumption is made that no O_2 diffuses into the solid polymer, any excess of diffusion inflow over convection outflow is the reaction rate of oxygen at the surface interface. In addition, Fick's first law and the mixture rule

$$D_{O_2m} = \frac{1 - X_{O_2}}{\sum_{i=2}^n \left[X_i / D_{O_2i} \right]}$$

$i = 2$

are assumed where D is the diffusion coefficient, i is a species in a mixture of n components, and X is the mole fraction. Diffusion coefficients are calculated using formulas based on the Lennard-Jones potential. The concentration gradient and the concentration of O_2 are evaluated at

the experimental point nearest the surface. The assumption is made that oxygen at the surface reacts with ethylene to form CO_2 and H_2O , and that energy from the surface reaction goes exclusively for depolymerization. Also, the polymer is assumed to contain no oxygen initially.

The motivation behind the model calculation is to establish an upper bound on the contribution of surface reaction energy release to depolymerization. The set of composition profiles that gave the greatest net transfer of oxygen to the surface is considered (Figure 9). The following values are used: fuel mass flux of $6.61 \text{ gm/m}^2\text{s}$ (the polymer burning rate), gradient in oxygen mole fraction of 7.4 m^{-1} , oxygen mole fraction of 5.4×10^{-3} , surface temperature of 900 K, and enthalpy of depolymerization of $3.85 \frac{\text{kJ}}{\text{gm}}$.

The result of this maximum energy release calculation indicates that heterogeneous surface oxidation could provide 20% of the energy required for depolymerization; however, the contribution could be less. Some of the energy from the oxygen surface reactions could be lost through heat transfer to the surroundings. The heat release could be less than the maximum amount assumed if the products of the surface reactions differ from CO_2 and H_2O . Since the calculation is made at a point 0.7 mm from the surface, the oxygen could also be reacting in the gas phase in the region between 0.7 mm and the surface, and thus the heterogeneous reaction would be providing less heat for pyrolysis. The 20% value could also be altered due to small errors in the composition profile which would, in turn, affect the value of the concentration gradient.

The uncertainty in the determination of oxygen diffusion to the surface from species concentration profiles results from experimental

difficulties. The evaluation of the gradient requires a very accurate composition profile in the surface region. The uncertainty of the probe-surface distance resulting from surface irregularities contributes to errors in the gradient. Also, the probe resolution alters the concentration gradient obtained.

Conclusions

1. The composition profiles are similar to within experimental error.
2. The CO/CO₂ ratio increases slightly near extinction.
3. CO significantly penetrates the luminous flame zone, which is a departure from the flame sheet approximation.
4. Two modes of approaching extinction are identified. However, extinction is achieved in both modes due to insufficient time for reaction.
5. It is extremely difficult to ascertain the contribution of heterogeneous oxidation heat release to the energy required for depolymerization.

Acknowledgements

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