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# EFFECT OF WATER ON IGNITION OF CELLULOSIC MATERIALS

By

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# A DISSERTATION

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#### ABSTRACT

#### EFFECT OF WATER ON IGNITION OF CELLULOSIC MATERIALS

By

### Mahmood Abu-Zaid

This experimental study is an attempt to understand and quantify the effect of water on fire extinguishment, thermal decomposition, and piloted ignition of wood.

For the extinguishment part, the effect of water as externally applied on hot porous and non-porous ceramic solids was studied. Both solids were instrumented with several surface and in-depth thermocouples. Similarities and differences in their thermal behavior, and the heat transfer from the non-porous solid during the evaporation of a single droplet of pure water were investigated. This study will help determine the minimum water application, and the application strategy to cool as-yet-unburned objects.

Thermal decomposition of wood as a function of sample moisture content and externally applied radiation was investigated. Experiments with three different moisture contents were performed at different heat fluxes in a controlled atmosphere combustion wind tunnel. Simultaneous measurements of weight loss rate; surface, bottom, and in-depth temperatures;  $0_2$  depletion; production of  $CO_2$ , CO, total hydrocarbons, and water were made. It was found that the presence of adsorbed water

delayed the decomposition process and diluted the decomposition products.

For piloted ignition, experiments on Douglas fir with four different moisture contents were performed at different levels of externally applied radiation. In these experiments, in addition to the quantities measured in the decomposition experiments, the time for piloted ignition was also recorded. An absolute minimum mass flux of approximately  $0.22 \text{ mg/cm}^2$ s was found to be necessary for piloted igntion to occur. It was also found that the presence of moisture increases the ignition time, surface temperature and evolved mass flux at ignition, and the critical heat flux. A single equation was derived to correlate all of the ignition data. This correlation accounts for the moisture-dependent thermal properties and the heat loss from the sample. To my father; Zaal, mother; Nadwa, my wife; Ensherah, and children; Sawsan, Anas, Ala, Esra, whose patient made the work possible.

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# NOMENCLATURE

| а              | a constant  |
|----------------|---|
| В              | D/d   |
| C              | specific heat   |
| C <sub>v</sub> | specific heat at constant volume                              |
| с <sub>р</sub> | specific heat at constant pressure                            |
| d              | diameter of droplet before impact                             |
| D              | maximum diameter of droplet on surface                        |
| F              | incident heat flux  |
| f <sub>s</sub> | heat flux entering the solid                                  |
| Н              | release height, i.e., the distance between the needle tip and |
|                | the solid surface   |
| h              | heat transfer coefficient                                     |
| h <sub>o</sub> | overall heat transfer coefficient                             |
| k              | thermal conductivity  |
| L              | liter   |
| q              | heat flux   |
| Q              | rate of heat  |
| R              | radius of the disk  |
| r <sub>d</sub> | maximum radius of droplet on the surface                      |
| S              | solid   |
| Т              | temperature   |
| Te             | equilibrium temperature                                       |

T contact temperature

| T <sub>s</sub>   | initial solid surface temperature in droplet evaporation    |
|------------------|---|
|                  | analysis, solid surface temperature in piloted ignition     |
|                  | analysis  |
| T <sub>w</sub>   | ambient temperature   |
| <sup>T</sup> sat | saturation temperature                                      |
| т <sub>w</sub>   | droplet temperature before impact                           |
| Tws              | time averaged surface temperature                           |
| $\Delta T_{sat}$ | T <sub>WS</sub> - T <sub>sat</sub>                          |
| t                | time  |
| t <sup>*</sup>   | dimensionless time, t/r                                     |
| v                | initial volume of droplet                                   |
| v <sub>i</sub>   | volume of droplet influence in solid                        |
| (x,y,z)          | cartesian coordinates                                       |
| (ξ,η,φ)          | oblate spheroidal coordinates                               |
| w                | water   |
| α                | thermal diffusivity   |
| γ                | <i>√ρ</i> <b>ck</b>   |
| δ                | penetration depth   |
| δ*               | critical droplet thickness, i.e., droplet thickness on the  |
|                  | non-porous solid at the time the evaporation rate starts to |
|                  | increase sharply  |
| ε                | emissivity  |

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 $\theta = \frac{T-T_e}{T_s-T_e}$  in droplet evaporation analysis,  $T-T_{\infty}$  in piloted

ignition analysis

 $\theta_{s}$  T<sub>s</sub>-T<sub>∞</sub>

ρ density

- σ Stefan-Boltzmann constant
- *τ* evaporation time of droplet

## CHAPTER 1

#### INTRODUCTION

Cellulosic materials such as wood, cardboard, paper and cotton often constitute the bulk of the fuels in many fires. A common fire starts in a residential room by a source of ignition. Thermal radiation from the flames and the hot gases may heat and cause ignition of nearby combustible materials allowing the fire to grow. The process of fire spread is closely related to piloted ignition, and may be regarded as a rapid succession of piloted ignitions.

Today, in the most technologically advanced civilization, we still face the threat of unwanted fires. A recent estimate shows an annual loss of \$10 billion of property and forest in the United States. More important and not expressible in dollars, is the annual loss of thousands of lives, and millions of people sustaining serious burn and smoke inhalation injuries. A fundamental study to understand the chemical and physical process that occur during combustion of cellullosic materials will result in achieving effective control and prevention of unwanted fires.

In the burning of cellulosic materials, water plays an important role from ignition to extinguishment due to their porous and hygroscopic nature and the availability of a large area to absorb moisture from surrounding air. The moisture content of these materials

is variable, and is influenced by the climate, season and location. Also, water is the most common agent used to extinguish unwanted fires. Thus, a study of ignition and extinguishment of combustible cellulosic materials is of great importance from the point of view of fire safety because these materials constitute the bulk of fuels in many building fires.

This study is an attempt to understand and quantify the effect of water on piloted ignition and extinguishment of cellulosic materials. Piloted ignition has been chosen because it is closely related to fire spread and occurs at lower critical temperatures relative to spontaneous ignition. Consequently, it is more hazardous. This work will be useful in determining the onset of piloted ignition, the fire growth rate under different humidity conditions as well as the minimum water application rate and the application strategy to restrain the fire growth.

## 1.1 Description of the Problem

The problem of extinguishment by water may be divided into two parts; (i) extinguishment of the already burning objects and (ii) prevention from burning of as-yet-unburned objects [Atreya (1985)]. In the cooling of unburned solids, the physical process of water evaporation on hot porous and non-porous solids is different. Results from some early experiments conducted by Atreya (1985) indicate that a water droplet impinging on a hot surface of wood (surface temperatures in piloted ignition range from 300 - 400  $^{\circ}$  C ) behaves differently than

it does on a hot copper plate at the same temperature. On the copper plate, a thin vapor film is formed between the droplet and the hot surface. As a result of this layer, the heat transfer is considerably lower than in the case of direct contact (liquid has much better thermal transport properties than its associated vapor). On the other hand, in wood the droplet is quickly absorbed and re-evaporated. Thus, in-depth cooling of the solid occurs.

The investigation of ignition of cellulosic materials must be preceeded by the study of thermal decomposition of such materials. The physical aspects of decomposition are associated with the heat and mass transfer, processes which occur as the solid is heated. The external heat is primarily transferred to the surface of the solid by radiation. This radial energy will be transferred into the solid by conduction process. As the temperature at any point rises, hygroscopic water is first released, followed by decomposition of the solid in a complex fashion. About two hundred compounds have been identified as the products of decomposition of wood [Goos (1952)]. The rate at which these products are generated depends upon the response of the solid phase to the applied heat flux. These products are driven to the surface of the sample by high pressure generated inside the solid where they mix with the surrounding air.

A good quantitative understanding of the factors controlling the decomposition of the solid, and the composition of the evolved gases, is important for ignition and fire spread and for fire prevention in general. It is also important in the context of minimizing air pollution from wood burning stoves, Ohlemiller (1987).

The ignition mechanism is influenced by factors both external and internal to the sample. Factors external to the sample are the environmental variables, such as temperature, composition and velocity of the surrounding gases, etc. Factors internal to the sample are its thermophysical and thermochemical properties and its moisture content. Experimentally, all variables other than the thermochemical and thermophysical can be controlled.

The actual process of ignition is quite complicated. A simplified phenomenological picture of piloted ignition by radiation has been clarified by Atreya (1986) and Kashiwagi (1981). The solid must first chemically decompose to inject fuel gases into the boundary layer. These fuel gases must then mix with the surrounding air, and the local mixture ratio must be near or within the flammability limits. At this instant, a premixed flame, originating from the pilot flame, flashes across the surface of the solid through the fuel-air mixture formed in the boundary layer. Further heating of the solid results in evential ignition and finally the establishment of a diffusion flame in the boundary layer.

The adsorbed water in the wood matrix could effectively delay the wood decomposition process, and reduce the flammability of wood (i.e., increase the ignition time) due to the high heat of vaporization of water. In addition, water vapor could complicate decomposition reactions, such as the reaction between water vapor and wood char to produce carbon monoxide and hydrogen at high temperature.

In view of the above observations, the research problem is logically divided into two parts:

(i) Effect of water as externally applied in the form of droplets,

(ii) Effect of adsorbed water on wood decomposition process, and piloted ignition as a function of sample moisture content and externally applied radiation.

#### 1.2 Related Literature

In this section, only the general literature related to the problem is discussed. Literature related to the specific component of the problem is discussed in the appropriate chapters.

For droplet vaporization a number of studies on hot non-porous metallic solids have been reported in the literature. These studies show that the vaporization mode of a liquid droplet on a hot surface depends on many factors. These are: (i) initial surface temperature of the solid, (ii) isothermal or non-isothermal condition of the solid, (iii) thermal properties of the droplet and the solid, and (iv) the droplet momentum at impact. Few studies [diMarzo and Trehan (1986)] have focused on in-depth cooling of the solid (which is important for predicting the rate at which fuel gases are produced), and none have reported transient in-depth temperature measurements.

A substantial portion of the combustible building materials are porous with low thermal conductivity and thermal diffusivity. Vaporization of water droplets on such solids is expected to be different from that for the non-porous metallic solids. However, despite the need to address the cooling of hot porous chars and unburnt wood during fires, the author has been unable to find relevant literature for porous solids.

A considerable amount of work has been done on the thermal decomposition of wood and other cellulosic materials. These studies were performed using different materials and under various conditions. Martin (1965) investigated the decomposition of pure cellulose in helium, Atreya (1983) investigated the decomposition from different kinds of wood in air. The decomposition of PMMA and particle board subjected to variable heat flux under an inert atmosphere was investigated by Volvelle et al. (1984) Kashiwagi (1987) studied the products generated from wood decomposition in 3 atmospheres of different oxygen concentration (N<sub>2</sub>, 10% O<sub>2</sub> - 90% N<sub>2</sub>, and air).

Lee and Diehl (1981) investigated the effect of absorbed water on samples containing 50% of oven-dry weight as water. They indicated that moisture not only changes the solid-phase thermal properties but also substantially dilutes the decomposition products. Atreya (1983) from his recent work concluded that the desorption of moisture, which has been ignored by previous investigators, has a considerable effect on the energetics of the decomposition process.

Ignition of cellulosic materials (both spontaneous and piloted) has been an active area of research in the past. Several excellent reviews have been published on this subject [Welker (1970), Kanury (1972), and Steward (1974)]. Although numerous techniques have been developed to investigate piloted ignition phenomena, the experiments essentially consist of exposing a sample to a known external radiant flux and recording the time required to ignite it in the presence of a pilot flame. Of the proposed ignition criteria, critical fuel mass flux at ignition [Bamford et al. (1946)] seems to be the most correct

physically (since it can be related to flammability limits), whereas the critical surface temperature at ignition has proved to be the most useful [Atreya (1983)]. However, only a few investigators [Atreya (1983), Kashiwagi (1981), and Garden (1953)] have actually measured the surface temperature at ignition, and measurements of the critical fuel mass flux at ignition for cellulosic materials are not available due to the complications caused by the simultaneous evolution of absorbed moisture.

The importance of moisture content in relation to fire tests has been recognized. Researchers have carefully controlled the moisture content of their samples, but little attention has been devoted to the effect of sample moisture on decomposition and piloted ignition. This study is a systematic investigation of its effect.

## 1.3 Present Work

In this work, the effect of water on piloted ignition and extinguishment is investigated experimentally. The apparatus and procedure used, experimental errors incurred, and data reduction procedures are described in Chapter 2.

For the droplet experiments, the heated solid was cast from ceramic and instrumented by several surface and in-depth thermocouples. In order to compare the results of porous and non-porous solids under the same conditions two identical solids, porous and non-porous, were cast from the same ceramic powder. Using different droplet sizes, a systematic set of experiments (initial solid temperature ranged from

75-325  $\degree$ C) were performed. In these experiments, transient surface and in-depth temperatures, and the droplet evaporation process were recorded. The results of these experiments and a discussion is presented in chapter 3.

Having recognized the importance and need for understanding the phenomenon of thermal decomposition for the piloted ignition problem, a set of experiments with three different moisture contents were performed at different levels of externally applied radiation. In these experiments simultaneous measurements of several physical and chemical quantities were recorded. The results of these experiments are described in Chapter 4.

For piloted ignition, a set of experiments with four different moisture contents were performed at different levels of externally applied radiation. In these experiments, besides the quantities measured in the decomposition experiments, the time for ignition was also recorded. Critical surface temperature and critical fuel mass flux ignition criteria were discussed, and a correlation for ignition data was derived and discussed. Chapter 5 describes the results of these experiments.

Finally, conclusions for the various components of this research problem and some recommendations for future work are presented in Chapter 6.

#### CHAPTER 2

# EXPERIMENTAL APPARATUS AND PROCEDURE

The primary objective of the present work is to understand and obtain useful data for droplet evaporation on hot porous and non-porous ceramic solids, wood decomposition, and piloted ignition of wood. Experiments for these components were performed using different facilities. This chapter describes the apparatus and procedure used for each component, the data processing methods, and errors encountered in these experiments.

## 2.1 Droplet Evaporation Experiments.

## 2.1.1 Apparatus

Experimental study of cooling of a hot, low-thermal-diffusivity porous solid by water droplets requires an apparatus that satisfies the following conditions:

(1) The solid should be nearly isothermal, porous, and made of a low thermal conductivity and diffusivity material, and the temperature of the heated solid must be controllable.

(2) The heated solid must be instrumented with surface and in-depth thermocouples. From these transient temperature measurements during droplet evaporation the following information can then be determined:
(i) recovery time i.e. the time for the surface to recover to its

initial temperature, and (ii) the size of "surface and in-depth zones" affected by the droplet.

(3) A system capable of generating different size droplets is also required. This system must be capable of delivering droplets on the solid surface at a specified rate and at a specified release height.

An experimental apparatus was built to satisfy the above requirements, a schematic is shown in Figure 2.1.

## 2.1.1.1 Heated Ceramic Block

To obtain a nearly isothermal solid three different shapes of the ceramic blocks were considered. These were (i) Hemi-sphere: Here, to make the flat surface isothermal, a large variation of the heat flux applied to the hemispherical surface is necessary. (ii) Cylinder: It is possible to obtain a flat isothermal surface in this geometry if the cylinder is heated from below with a constant heat flux, and the cylindrical surface is kept well insulated. The main disadvantage is that a very small cylinder height is needed for the solid to be nearly isothermal. (iii) Oblate spheriodal: This geometry has been chosen because it combines the advantages of both cylindrical and spherical geometry. It also provides a natural co-ordinate system for this problem.

A castable porous ceramic (MgO) was used to cast a semi-oblate spheroidal solid. This solid was used to simulate an isothermal semiinfinite solid. The configuration of this block along with the calculated temperatures and heat flux is shown in figure 2.2. The block has a major semi-axis of 8.48 cm and a minor semi-axis of 7.22 cm. Theoretical calculations described below show that this ceramic block







convection and radiation is equal to the heat flux arriving at this surface from the heaters. The temperature of the heaters (109.01°C) was calculated Ceramic block configuration: The isotherms correspond to  $\xi = 0$ , 0.55, 0.85, 1.15. In the range (a-b), the heat flux lost from the exposed surface by to achieve this balance. The range (a-b) is the useable range for water droplet experiments. Figure 2.2

is nearly isothermal, despite the poor thermal conductivity (0.023 W/cm.k) of the porous ceramic. For the solid shown in Figure 2.2, let  $\xi$ ,  $\eta$ , and  $\phi$  describe the orthogonal oblate spheroidal co-ordinate system. Transformation of the cartesian to the oblate-spheroidal co-ordinate system is given by:  $\mathbf{x} = \mathbf{a} \cosh \xi \cos \eta \quad \cos \phi$ ,  $\mathbf{y} = \cosh \xi \cos \eta \quad \sin \phi$ , and  $\mathbf{z} = \mathbf{a} \sinh \xi \sin \eta$ , where  $\mathbf{a} = \text{constant}$ ,  $\xi \ge 0$ ,  $-\frac{\pi}{2} \le \eta \le \frac{\pi}{2}$ ,  $0 \le \phi \le 2\pi$ . As shown in Figure 2.2,  $\xi$ = constant, represents the isotherms and  $\eta$ = constant, represents the heat flux lines. As required by their definition, they are orthogonal at every point in the domain. Under steady-state conditions, the heat conduction equation reduces to the following Laplace equation

$$\nabla^{2}T = \left[\frac{1}{a^{2}(\sinh^{2} \xi + \sin^{2} \eta)^{1/2} \cos \xi}\right] \frac{\partial}{\partial \xi} \left(\cos \xi \frac{\partial T}{\partial \xi}\right)$$

$$+ \left[\frac{1}{a^{2}(\sinh^{2} \xi + \sin^{2} \eta)^{1/2} \cos \eta}\right] \frac{\partial}{\partial \eta} \left(\cos \eta \frac{\partial T}{\partial \eta}\right)$$

$$+ \left[\frac{1}{a^{2} \cosh^{2} \xi + \cos^{2} \eta}\right] \frac{\partial^{2}T}{\partial \phi^{2}} = 0 \qquad (2.1)$$

Assuming that temperature is not a function of  $\eta$  and  $\phi$ , solution of the above equation with the boundary conditions:  $T = T_1$  at  $\xi = 0$ ,  $T = T_2$  at  $\xi = 1.15$ , is given by:

$$T = 2.09(T_2 - T_1)tan^{-1}[exp(\xi)]+2.641 T_1 - 1.641 T_2$$
 for  $\xi \le 1.15$  (2.2)

and the heat flux is given by:

$$q = \frac{-E}{(\sinh^2 \xi + \sin^2 \eta)^{1/2}} \left[ \frac{\exp(\xi)}{1 + \exp(2\xi)} \right]$$
(2.3)

where  $E = \frac{2.09}{a} k (T_2 - T_1)$ .

As shown in Figure 2.2, the calculated heat flux on the exposed surface between a and b is nearly constant and it is matched to the radiative and convective heat losses to the environment. This yields a maximum temperature across the solid of 9.01  $^{\circ}$ C which is considered to be nearly isothermal relative to the dimensions of the droplet.

The above described block is instrumented with several surface and in-depth thermocouples. Chromel-Alumel thermocouples of 76  $\mu$ m diameter were used. The thermocouples were flattened to a film thickness of about 12  $\mu$ m. Seven surface and six in-depth thermocouples were placed inside the mold. The location of the thermocouples prior to casting was measured to within ± 0.1 mm. Due to the uncertainties introduced during the casting process and shrinkage, the thermocouple locations were evaluated by a least square fit to the steady state temperatures. Detailed description for evaluating the thermocouple locations is included in Appendix A.

In order to compare the results of porous and nonporous solids under the same conditions, two identical solids of the same material were casted. The non-porous solid was obtained by spreading a small amount of solution of very fine ceramic powder of the same material on the surface of the porous solid. This procedure closes the surface pores, rendering the surface nonporous.

As shown in figure 2.1, the ceramic block is heated by a heating mantle. In order to achieve a perfect contact, the heating mantle has the exact dimensions of the ceramic block. Furthermore, the heating mantle has been configured to deliver the heat flux to the ceramic solid according to equation (2.3). The temperature of the ceramic block is controlled by a variac.

## 2.1.1.2 Droplet Generating System

The droplet generating system consists of the following items (i) stainless steel needles (ii) high quality syringe (iii) 90 V DC gearmotor and (iv) speed controller.

The water droplets were generated by a high quality motor driven syringe. To obtain different sizes 10, 12, 15, 19, and 25 gauge stainless steel needles were used. These needles were filled to a smooth flat tip. Care was taken to keep the main body of the syringe which contains water at ambient temperature (25  $^{\circ}$  C).

The syringe plunger was driven by a gear-motor whose speed was controlled by a speed controller. As the syringe plunger slowly moved, a droplet was formed on the needle tip and increased in size, until its weight became sufficient to detach it from the tip.

The droplet generating system has been calibrated using electronic digital scale (Mettler AE 100) with an accuracy of  $\pm$  0.1 mg. All needles of different sizes were calibrated for the droplet size at ambient temperature by individually weighing 15 droplets. The mean deviation was

found to be  $\pm$  2% from the arithmatic mean. For example, a 30  $\mu$ L droplet was found to have a volume in the range 29.4 - 30.6  $\mu$ L.

The motor-driven syringe system was placed on an adjustable height platform to facilitate the adjustment of the needle tip height from the hot solid surface. The adjustable range is from (0-10cm).

## 2.1.1.3 Data Acquisition Equipment

An analog-to-digital converter which provides 22-bit resolution is linked to the HP data acquisition unit by IEEE interface bus. Signals from the thermocouples were fed into a HP3497A controller with 39 differential input channels. The data was collected in real time by using Digital Equipment corporation PDP11-73 microcomputer. Further, the data was transferred to VMS-VAX 750 for permanent storage on magnetic tapes.

#### 2.1.2 Experimental Procedures

The experimental procedure consists of setting the variac to a fixed value to heat the ceramic block to the desired temperature. It took between 10 to 12 hours for the solid block to reach steady state conditions.

Pure water was used for these experiments. This water was double distilled, deionized and degassed. Degassing process consists of boiling the water and cooling it under vacuum of 23 inch mercury. Before starting the experiment, some water in the needle was ejected to ensure that the droplets were at ambient temperature (~  $25 \, {}^{0}$ C).

Tests were performed with initial surface temperature ranging from 75-325 <sup>0</sup>C. The droplets were deposited gently on the heated surface from a release height of 6-9 mm (from the tip of the needle to the hot surface). This released height was varied to achieve a constant distance between the bottom of the droplet and the surface of the solid. During these tests transient measurements of temperature were collected by the data acquisition system.

A video camera was used to record the life time of the droplet on the hot surface. This provides a precise measurement of the evaporation time and droplet size. In addition, it shows the droplet behavior on the hot-surface.

# 2.2 Wood Decomposition and Piloted Ignition Experiments

## 2.2.1 Apparatus

The apparatus used in these experiments is schematically shown in figures 2.3. It consists of three main units:

(i) Combustion wind tunnel, to perform experiments in a well-controlled atmosphere and radiation environment.

(ii) Gas analysis equipment, to measure concentration of chemical species in the exhaust.

(iii) Data acquisition equipment, to collect and store data.

# 2.2.1.1 Combustion Wind Tunnel

This facility is the heart of the apparatus. It is capable of providing a flat-plate boundary layer flow on the sample surface at a
desired composition and free stream velocity. This tunnel consists of three main sections: the inlet section, the test section, and the exhaust section.

(i) Inlet Section: Gases are supplied to the inlet section by two sources; (a) pressure regulated laboratory air supply (up to 95 psig), and (b) high pressure gas cylinder ( $O_2$  or  $N_2$ ). These gases pass separately through high pressure tanks. In these tanks any oscillations in the flow would be damped out. To control the gas composition, these gases are metered by critical nozzles. Detailed description and calibration of these nozzles is presented in Appendix B. The resultant mixture enters the tunnel through eight symmetrically distributed turbulent jets. It then passes through a set of screens and a permeable cloth into a large settling chamber. In this settling chamber the gas velocities are small and the pressure is only slightly above atmospheric. It then passes through turbulance manipulators to suppress the turbulance in the flow.

(ii) <u>Test Section</u>: The test section consists of three modules: radiant heaters, tunnel top, and tunnel mainframe.

The external radiation was simulated by the use of nine radiant heaters. Three high temperature quartz heaters and six U-shaped chromalox coil heaters. The heaters are controlled by a 3-phase 440 volt variable transformer. These heaters have a maximum black body radiation temperature of 1200 <sup>°</sup>K and are therefore well suited to simulate external radiation in building fires. Reflective chromium plated steel sheets were configured to obtain a maximum radiant flux of 4 W/cm<sup>2</sup> at the sample surface.

The water-cooled tunnel top houses five pieces of the infrared optical glass (Kodak IRTRAN-II). The glasses were securely held in a copper frame lined with flexible silicon rubber to allow for expansion. The combination of these glasses make a 6" \* 30" infrared glass window. This glass window was cooled convectively by compressed air.

The tunnel mainframe is made of thick water-cooled brass walls. The mainframe is connected to the heater system to maintain the position of the heaters fixed relative to the sample surface. It consists of four observation windows for optical access and three slots for probe access. The latter slots were lined with high temperature silicon rubber to prevent leakage. Two non-combustible blocks (Kaowool insulating boards) mounted flush with the sample surface, are placed symmetrically on both sides of the sample. On top of the insulating boards, a  $\frac{1^n}{16}$  thick aluminum plate was placed in order to create a smooth boundary layer. Except for the sample space (6" \* 3") all tunnel base was covered with the combination of the ceramic boards and aluminum plate.

(iii) <u>Exhaust Section</u>: The primary purpose of the exhaust section is to mix the products of decomposition (or combustion) to obtain a representative gas sample for chemical analysis. This gas sample was used to determine the production or depletion rates of the species measured at the gas analyser. The gas sample is extracted through a cross-shaped probe, and carried by heated lines to the continuous gas analysers.

The mixing process takes place in a steel mixing chamber lined with the insulating boards. The mixing is accomplished by using heated electrical resistance tapes as louvers upstream in the mixing chamber.



Further downstream a net of electrical resistance tapes and wires were used for heating. This combination results in a hot mixing chamber. This heating is necessary to prevent condensation of heavy molecular weight hydrocarbons.

The suction introduced by the exhaust fan is adjusted to obtain the desired pressure above the sample surface. To reduce the turbulent fluctuations inside the tunnel, the fan is isolated from the tunnel by using 24" \* 24" \* 36" metal chamber.

### 2.2.1.2 Gas Analysis Equipment:

The gas sample was sucked from the exhaust section by a pump into the gas analysis equipment to continuously measure the depletion of  $O_2$ and production of  $CO_2$ , CO, total hydrocarbons [THC] and  $H_2O$ . A 1/4 inch outside diameter sampling line that was about 70 inches long was used to supply the gas sample to the analysis equipment. This resulted in transport delay times. The transport time for the gases from the sample location to the instrument was measured for each analyser. These times were 3.5 sec. for [THC], 6.5 sec. for  $O_2$ , 4.5 sec. for CO, and 5.0 sec. for  $H_2O$  and  $CO_2$ . A schematic of the system is shown in Figure 2.4, and a detailed description is given in Appendix C.

### 2.2.1.3 Data Acquisition Equipment:

In these experiments the same data acquisition equipment described in section 2.1.1.3 was used. In addition to the analog signal from the thermocouples, signals from the instruments, heat flux gauge, and the electronic balance were also collected and stored.



Figure 2.4 Gas Analysis Equipment.

# 2.2.2 Procedure:

#### 2.2.2.1 Sample Preparation:

Wood samples were cut to the desired size (6" \* 3" \* 1.5") from 12" \* 12" \* 1.5" boards of Douglas fir. This wood was obtained from the Forest Products Laboratory at Madison Wisconsin. The samples were carefully machined to smooth surfaces to eliminate any effect caused by surface roughness. The samples were instrumented by surface, bottom and in-depth thermocouples. (The thermocouples were made from fine chromel and alumel wires (0.003" dia). The method employed to install the thermocouples was developed and successfully used by Atreya (1983). For surface and bottom thermocouples, the thermocouple wires flattened to a film thickness, and electrically welded. The bead was polished by a fine emery cloth. A very fine layer of wood was skinned off from the sample surface using a sharp razor blade, and a portion of the thermocouple was secured underneath this layer using Tight-bond wood glue. The extra glue was wiped off and the assembly was allowed to dry under a heavy weight for 2-3 hours. The in-depth thermocouples were installed at different locations. Holes were drilled (1 mm dia, 1 inch length) at desired locations on the side of the wood sample. Ceramic insulating tubing with two holes were used to house the thermocouples inside the holes.

The samples were then dried in a temperature-controlled chamber at  $105^{\circ}$ C until a constant weight was achieved. This weight was recorded. A temperature-humidity-controlled chamber at a known relative humidity was

used to fix the moisture content in the samples to the desired value. This chamber (Model TH3, BMA Inc.) has controllable humidity range of 10 to 98% relative humidity for temperatures between 10  $^{\circ}$ C and 85  $^{\circ}$ C. Samples were conditioned for 5-6 weeks until a constant weight was obtained. The difference between this weight and the dry weight was used to determine the sample moisture content. Samples edges and bottom were taped with aluminum foil to avoid any edge effects.

## 2.2.2.2 Calibration:

The gas analysis instruments were first turned on and allowed to warm up until a steady value was reached. Zero gas  $(21.3 \ 0_2$  and rest  $N_2$ ) was allowed to flow through the gas analysers, and all the instruments were electrically adjusted to their reference value. These values were recorded. Calibration gas  $(0.201 \ methane, 1.97 \ CO_2, 0.201 \ CO$ CO and rest  $N_2$ ) was allowed to flow through the gas analysers. The output of all instruments was again recorded and a calibration factor was calculated and stored. The water meter was calibrated using room air. The mole fraction of this air was determined from the wet and dry bulb temperatures. Periodically, the meter was checked against a third point, by introducing an air stream at known condition from the humidity chamber. The calibration factors for some instruments, namely thermocouples and the radiometer, were constant, thus they did not require calibration each time.

### 2.2.2.3 Experiments:

The heaters were turned on at the desired radiant heat flux, and were allowed to warm up for about 15-20 minutes. The air valve was opened, and the pressure upstream the critical nozzle was set to the desired value by adjusting the air regulator. The air was allowed to flow through the tunnel for about 10 minutes to attain a steady state condition for all instruments. The pressure above the sample was adjusted by the exhaust fan to obtain atmospheric pressure. The leakages from the gap around the sample and from the top of the tunnel were then determined. A known volume of methane gas tracer was introduced in the main flow downstream of the critical nozzle, and leakages were determined from the difference in the methane concentration measurements upstream and downstream of the tunnel.

The cooling plates were opened, and the tunnel was allowed to warm up for about 20 minutes to drive out the absorbed moisture from the insulating boards. The tunnel was then allowed to cool to room temperature. The sample was placed on the weighing table with its top surface flat along the tunnel base. The sample was then exposed to a known external radiation by opening the cooling plates, and data collection was simultaneously started.

The heat flux sensor located upstream in the tunnel continuously monitored radiation from the heater during the experiment. A calibration between the heat flux at the sensor location and the heat flux at the sample location was used to determine the incident radiation on the sample surface.

For a given moisture content and external radiation, the following measurements were recorded as a function of time: (i) surface, bottom and in-depth temperatures, (ii) mass evolution rate of water vapor, (iii) production rate of combustible gases and (iv) weight loss rate of wood sample.

For piloted ignition experiments, in addition to the above measurements, the time required for ignition was also recorded. A video camera was focused on the sample to record the experiment. This provided a precise measurement of the time of ignition.

# 2.2.3 Data Reduction:

The output of the instruments were stored by microcomputer in real time. These data represent the mole fraction of the species measured. It must be processed because not all instruments register an event at the same time due to time lag and response time of the instruments. The procedure used for data reduction is the same as developed and used by Atreya (1983). The final result of this procedure is that all measured quantities are corrected and brought to the same time base. This data consists of instantaneous mole fraction of all the species at 5 sec. intervals. These values were then converted to percentages on mass basis. Finally, knowing mass flow rate of air, surface area and weight loss rate of the wood sample, the mass flux  $(mg/cm^2.s)$  for each specie was determined.

# 2.2.4 Experimental Error:

The difference between the true value of a quantity and the measured value is known as the measurement error. These errors may be systematic or random. Systematic errors must be eliminated or corrected, whereas, it is difficult to predict and correct random errors.

In these experiments, a systematic error introduced by the leakages in the tunnel was corrected by the tracer method discussed in Section 2.2.2.3. Random errors from electrical noise, limitation in accuracy and response time of measuring instruments, condensation of heavy hydrocarbons in the tunnel and sample lines, and oscillations in weight loss measurements were difficult to estimate since they vary with time due to the transient nature of the experiments. Therefore no corrections were made for those errors.

### CHAPTER 3

## DROPLET EVAPORATION ON HOT POROUS AND NON-POROUS CERAMIC SOLIDS

# 3.1 Introduction

The study of the behavior of impinging individual droplets on a hot surface is essential in the analysis of problems involving liquid sprays. Knowledge of how the droplet impacts and evaporates will help in understanding and prediction of the effect of sprays on cooling of hot surfaces. Such problems are encountered in a number of engineering areas; fire extinguishment, nuclear reactor safety, cooling of turbine blades, and cooling of continuously casted steel.

The evaporation of a liquid droplet on a hot solid surface, occurs by extracting heat from the region directly below it. In quasi-steady evaporation, the evaporation rate is the heat transfer rate divided by the heat of evaporation [Renksizbulut and Yuen (1983)]. For such a case, the heat of evaporation is equal to the heat transferred through the droplet and also equal to the heat transferred from the solid to the bottom of the droplet.

Water sprays are the most common method used in fire extinguishment. Different types of solids with different surface characteristics are involved in a fire. Natural solids such as wood are porous and produce low thermal conductivity porous chars, whereas, plastics such as PMMA are non-porous and remain non-porous during burning. Hence, the study

of thermal behavior of water droplet varporization on a hot and lowthermal-conductivity solids is important for fire extinguishment. Atreya (1985), divided fire extinguishment problem into two parts (i) extinguishment of the already burning objects (ii) prevention from burning of the as-yet-unburned objects. The present work primarily addresses the latter part of the problem. However, it may be applicable to the former part as well.

The outline of this chapter is briefly given below. Section 3.2 describes the factors effecting droplet evaporation, and the different regions of droplet vaporization process. Section 3.3 reviews the literature on the subject. Section 3.4 describes similarities and differences in the thermal behavior of the porous and the non-porous solids, and the heat transfer from the non-porous solid during droplet evaporation.

### 3.2 Background

Vaporization mode of a liquid droplet on a hot solid surface depends on many factors. These factors would have a significant effect on the evaporation time, the heat transfer rate from the solid surface, and the droplet behavior during evaporation. Such factors are (i) initial surface temperature of the solid (ii) isothermal or non-isothermal condition of the solid (iii) porosity of the solid (iv) thermal properties of the droplet and the solid, and (v) the droplet momentum at impact.

The droplet vaporization process on a hot solid surface could be divided into 4 regions; evaporation, nucleate boiling, transition

boiling, and film boiling (Leidenfrost phenomena). In the evaporation region, a direct contact between the droplet and the hot surface exists. Heat is transfered to the droplet by free convection, and evaporation takes place from the top and sides of the droplet. In the nucleate boiling region, vapor bubbles begin to form on the hot surface. As the initial surface temperature  $(T_c)$  increases, the rate of heat transfer from the hot solid increases. This is because the bubbles formation become more dynamically which cause an increase in fluid mixing near the hot surface. In the transition boiling, a vapor film start to form between the droplet and the hot surface due to the extensive formation of bubbles. As T increases, the rate of heat transfer decreases. This is because the direct contact between the liquid and the surface is decreasing, and the vapor has a lower thermal conductivity than of the liquid. In film boiling, the droplet set on a vapor layer. As T increases the rate of heat transfer increases, because thermal radiation is the major heat transfer mode from the hot surface to the liquid.

#### 3.3 Literature Review

Numerous studies of droplet vaporization process on hot metalic solids have been reported in the literature. These studies have focussed mainly on evaporation time and heat transfer from the hot solid during droplet evaporation. Few studies [di Marzo and Trehan (1986)] have focused on indepth cooling of the solid (which is important for predicting the rate at which fuel gases are produced), and none have reported transient in-depth temperature measurements.

Seki et al. (1978), reported surface temperature measurement of a hot wall cooled by a single droplet. They gave no description of the cooling effect produced by evaporating droplets. They demonstrated good agreement between the theoretical computation of solid-liquid contact temperature and experimental measurements. According to the solution for a semi-infinite body with a sudden change in surface temperature [Carslaw and Jaeger (1959)], the contact temperature is given by

$$T_{c} = \frac{T_{w} \gamma_{w} + T_{s} \gamma_{s}}{\gamma_{w} + \gamma_{s}}$$
(3.1),

where  $\gamma = \sqrt{\rho ck}$ ;  $\rho$ , c, k represents density, specific heat, and thermal conductivity respectively, and  $T_s$ ,  $T_w$  are the initial solid and droplet temperatures.

Makino and Michiyoshi (1979, 1978), studied the effect of initial size of the water droplet on it's evaporation time. They also determined the time-averaged heat flux from the time-dependent measurements of the projected droplet base area, the solid surface temperature just below the droplet and the liquid temperature within the droplet. Since their work includes nucleate boiling, transition boiling, and film boiling regions, they present a wide range of the so called the boiling curve (q vs  $\Delta T_{sat}$ ). Thus this work establishes the thermal behavior of droplets during the vaporization process.

Bonancina et al. (1975), studied the heat transfer characteristics of atomized liquids on hot surfaces in a high pressure environment. Rizza's (1981) theoretical work on a non-isothermal wall was very useful for a spray evaporators. However, the work of [Bonancina (1975), Rizza (1981)] is of limited significance in fire extinguishment studies.

Gottfried and Bell (1961), determined the evaporation rate of the droplet from the measured evaporation time. The disadvantage of this method is that it assumes the evaporation rate to be constant which actually increases as the droplet becomes smaller. This non-constancy of droplet evaporation was noticed by Abu-Zaid and Atreya (1988a). Wachters et al. (1966), determined the evaporation rate by photographing the droplet from the side at small intervals. They obtain the volume and the rate of change of volume as a function of time. The disadvantages of this method is that very accurate volume measurements are necessary because their rate of change needs to be determined and they are multiplied by a large heat of evaporation. Also, the errors become worse as the volume decreases.

Gottfried et al. (1966), investigated the Leidenfrost phenomenon of some common fluids. They used droplets of volume < 0.1 mL on stainless steel plate at surface temperature ranging from 150-500°C. They determined the Leidenfrost point for water, carbon tetrachloride, ethanol, benzene, and n-octane. Furthermore they found that the Leidenfrost point was independent of droplet size for the range of droplet volumes studied.

Avedisian and Koplik (1987), studied Leidenfrost boiling of methanol droplets on hot porous ceramic surfaces. Their work was motivated by the recent interest in ceramic combustion engines. They measured evaporation times in the wetting and film boiling regions on a polished stainless steel surface and three ceramic/alumina surfaces of 10%, 25%, and 40% porosity. They found that Leidenfrost temperatures increased as

surface porosity increased. They also indicated that in the Leidenfrost regime, droplets evaporated faster on the porous surfaces than on the stainless surface, and the evaporation time decreased with increasing surface porosity at the same surface temperature. They explained this reduction in evaporation time by the decrease of the vapor film separating the droplet from the ceramic surface due to vapor absorption and flow within the ceramic material.

The work most directly related to the present work is that of di Marzo et al. (1987). They investigated experimentally the thermal behavior of droplets on hot aluminum and macor solids. In continuation of the above work, di Marzo and Trehan (1986), theoretically investigated the cooling effect of a hot semi-infinite aluminum block and obtained isotherms and envelope of the droplet influence. Evans and di Marzo (1986), presented a mode for the evaporation of a droplet in contact with high thermal conductivity and diffusivity solids. They determined that the evaporation time is in reasonable agreement with the experimental data. The model also describes the spatial and temporal behavior of the evaporation heat flux.

# 3.4 Results and Discussion

The data discussed here were collected during the large number of systematic experiments performed on porous and non-porous ceramic solids at various initial surface temperatures. Droplet sizes of 10-51  $\mu$ L were used, because they are representative of the droplets delivered by the fire suppression equipment [Evans and di Marzo (1986)]. Section 3.4.1 describes the transient cooling of hot porous and non-porous solids.

The similarities and differences in the thermal behavior of both solids during droplet evaporation is discussed. This work was presented in a paper [Abu-Zaid and Atreya (1988a)] submitted to the Journal of Heat Transfer. Section 3.4.2 describes the heat transfer during droplet evaporation on the non-porous solid. An extended abstract of this work [Abu-Zaid and Atreya (1988b)] was accepted for presentation at the Eastern Session of the Combustion Institute (Florida, December 1988).

# 3.4.1 Transient Cooling of Hot Porous and Non-Porous Solids

Results of a typical test composed of 3 droplets are shown in Figures 3.1-3.4. Transient surface and indepth temperatures measurements for a 30  $\mu$ L droplet are shown in Figures 3.1 and 3.2 for a non-porous solid, and in Figures 3.3 and 3.4 for a porous solid. These figures show the repeatability of the experimental measurements during the droplet evaporation process. Figures 3.1 and 3.3 also show the uniformity of surface temperature prior to droplet impact.

Individual traces of surface and indepth temperatures for different droplet sizes and for different initial surface temperatures are shown in Figures 3.5-3.13. In these figures a non-dimensional temperature  $\theta = \left(\frac{T-T_e}{T_s-T_e}\right)$  was plotted against a non-dimensional time t<sup>\*</sup> (t/ $\tau$ ). The non-

dimensional temperature  $\theta$  is chosen because  $\theta$  versus t<sup>\*</sup> curves for each case of the porous and the non-porous solids, for all initial solid surface temperatures (T<sub>s</sub>) and droplet sizes that were investigated, fall approximately on top of each other.

















For both the porous and the non-porous solids at an initial surface temperature  $(T_S) \leq 164$ °C, the hot surface immediately cooled to the theoretical contact temperature upon contact with the droplet. For  $T_S >$ 164°C, the surface temperature, upon contact with the droplet falls considerably below the theoretical contact temperature. Seki et al. (1978) found that on stainless steel, the measured contact temperature increases with increase in the surface temperature up to 180°C, and was in agreement with the theoretical contact temperature. For  $200 \leq T_S \leq$ 300°C, they found that the measured contact temperature was less than the theoretical contact temperature and was approximately constant. The present results for the ceramic are in qualitative agreement with Seki's data except that the present data shows a deviation from the theoretical value at 164°C rather than at 180°C.

After achieving the contact temperature the solid surface approaches the "equilibrium temperature" (interface temperature during evaporation). During the droplet evaporation period the thermal behavior of the porous and non-porous solids are different. These differences are described below:

### 3.4.1.1 Non-porous solid

Measurements show that for  $T_S = 75^{\circ}C$ , the surface and indepth temperatures remains nearly constant during the droplet evaporation process. This implies that the heat flux during the evaporation time is also nearly constant. For  $100 \le T_S \le 175^{\circ}C$ , the interface temperature remains nearly constant until a critical droplet thickness ( $\delta^*$ ) is reached on the hot surface. Then, the temperature starts to decrease sharply until the droplet has completely evaporated as shown in Figure 3.5. After the droplet has completely evaporated, the solid recovers <u>quickly</u> to its initial surface temperature. This sharp decrease in the surface temperature implies an increase in the evaporation rate. This will be discussed in more details in section 3.4.2.

For  $T_S = 200$ °C, the interface temperature drops to a value few degrees above the saturation temperature of water (see Figure 3.10). Then the surface recover to its initial value. The evaporation times at this temperature were very short, since the droplet diameter after impact on the surface was very large (see Figure 3.19). This large droplet diameter is due to lower surface tension.

## 3.4.1.2 Porous solid

For porous materials the interface temperature never clearly attains an equilibrium value. Both the surface and the indepth temperatures continue to decrease until the droplet vanishes from the surface (as can be seen from Figures 3.8 and 3.9). The time taken for the droplet to vanish from the surface is defined as the evaporation time. By this time a part of the droplet has already been evaporated, and the rest has penetrated in both the axial and the radial directions. The effect of the droplet penetration is clear from following two observations: i) A thermocouple in the porous matrix at the same location as the non-porous matrix cools faster under otherwise indentical conditions. This implies that, for the porous matrix, there must be an energy sink in the neighborhood of the thermocouple. Thus confirming the indepth



Non-Dimensional Surface Temperatures 0



Non-Dimensional Indepth Temperatures 0



Non-Dimensional Indepth Temperatures 0



Non-Dimensional Surface Temperatures 0







Non-Dimensional Surface Temperatures 0



Non-Dimensional Indepth Temperatures 0





Non-Dimensional Surface Temperatures 0



Non-Dimensional Indepth Temperatures 0

penetration of moisture. (ii) The evaporation time for the porous case is lower than the non-porous for the same droplet size and under the same condition. After the droplet vanishes from the surface, the porous solid recovers <u>slowly</u>, as opposed to the quick, pure conduction, recovery for the non-porous solid. The porous indepth temperature profiles during recovery are not as smooth as the non-porous solid, due to the migration of moisture inside the matrix (as can be seen from Figures 3.4 and 3.9). The presense of the moisture changes the thermal properties of the solid. This change makes the recovery behavior not only different but also longer than the non-porous solid.

The evaporation time for various droplet sizes versus intial solid surface temperature is shown in Figure 3.14. This time is as defined previously, the time taken for the droplet to vanish from the surface. Figure 3.14 shown that this time is a function of the droplet size and the intial surface temperature. As expected, lower the initial surface temperature and larger the droplet volume, more time is required to evaporate the droplet. Figure 3.14 shows also that for  $T_S \leq 164^{\circ}C$ , the evaporation time of the non-porous is a larger than the porous. For  $T_S > 164^{\circ}C$  the evaporation time for the porous is larger. For this range the parameter B for the non-porous solid was larger than the porous solid (see Figure 3.19). Generally, the evaporation time on the non-porous solid is larger than the porous solid for the same droplet under the same conditions. This indicates that in the process of cooling hot porous and non-porous solids, the porous solid needs droplets at higher frequency.

The recovery time is shown in Figure 3.15. This time is defined as the time for the surface to recover to its initial temperature. This time was determined by evaluating the time it took the surface temperature to recover to 20 percent of the maximum temperature drop. The maximum temperature drop is the difference between the initial surface temperature and the equilibrium temperature of the thermocouple underneath the evaporating droplet. The non-porous solid took slightly less time to recover than the porous solid despite the fact that for the non-porous solid, the evaporation process is steady and the evaporation time is larger.

Figures 3.16 and 3.17 show the non-dimensional maximum radial  $(x/r_d)$ and axial  $(z/r_d)$  influence distances of a single droplet plotted against the initial surface temperature. The influence distance encloses all locations where the temperature drop due to the droplet evaporation is at lest 20 percent of the maximum temperature drop i.e., the nondimensional temperature  $\theta = 0.8$ . Figures 3.16 and 3.17 show that the influence distance decreases at higher solid temperatures as more energy is available to evaporate the droplet. Also these influence distances are larger for the porous solid.

The non-dimensional maximum volume of influence  $(V_i/V)$  is shown in Figure 3.18. This quantifies the cooling effect of a single droplet. The volume of influence is defined as the volume of the semi-oblate spheroid formed by the axial and radial influence distances. Due to the low thermal conductivity/diffusivity of the ceramic, the droplet will produce intensive local cooling, because heat recovery from other portions of the solid matrix is limited. Thus, the influence zone is






on-Dimensional Maxim. Radial Influence



Non-Dimensional Maxim. Axial Influence



Non-Dimensional Max. Volume of Influence



expected to be relatively high. The influence zone of the porous solid is larger than the non-porous solid. This is due to the enetration of the droplet into the solid matrix, thus causing more cooling in both the radial and the axial directions. Figure 3.18 shows that the non-porous solid at  $T_S < 100$ °C cools more with a 10 µL droplet than with larger droplets. This is because the evaporation process for larger droplets attains a steady state and the extra fluid in the larger droplets just increases the evaporation time. This phenomena was not observed in porous solids because the evaporation process never quite attains a steady state.

Figure 3.19 shows the parameter B plotted against the initial solid surface temperature. This parameter is defined as the ratio of maximum droplet diameter during evaporation to the droplet diameter before impact. The diameter of the droplet on the surface was determined from the measured maximum wetted area. The droplets after impact had a circular shape during the evaporation process except for the non-porous solid at temperatures greater than 164°C. At these temperatures the droplets explode due to the intensive vapor formation between the droplet and the solid surface. As the surface temperature increases, B tends to increase due to the decrease in surface tension. B for the non-porous solid was larger than that for the porous solid due to migration of a part of the droplet into the porous matrix.

## 3.4.2. Heat Transfer During Droplet Evaporation on the Non-Porous Solid

During the experiments, the evaporation phenomena was recorded by a video camera. The droplet evaporation behavior changed with the initial

solid surface temperatures  $(T_g)$ . For  $75 \le T_g \le 125^{\circ}C$ , upon impact, the droplet have a disk configuration, and direct contact between the droplet and the solid exists. The evaporation takes place from the top and the sides of the droplet. The droplet diameter on the surface remains constant for up to approximately 80 percent of the evaporation time, and then starts to shrink. At  $T_g = 125^{\circ}C$ , after the initial contact, nucleate boiling was visually observed (see Figure 3.20; note that the equilibrium temperature is above the boiling point of water). As  $T_g$  increases (i.e.  $150 \le T_g \le 175^{\circ}C$ ), the boiling became more dynamic, and the extensive formation of vapor sometimes scatters the droplet. Seki et al. (1978) reported similar droplet behavior at these temperatures. For  $T_g > 200^{\circ}C$ , the droplets spread very fast on the hot surface, forming a thin water film. The evaporation time at this temperatures was very short.

Individual traces of the surface and indepth temperatures for a 51  $\mu$ L droplet impacting on the solid at  $T_g = 125$ °C are shown in Figures 3.20 and 3.21. As discussed in section 3.4.1, the hot surface immediately cooled to the contact temperature upon contact with the droplet. The contact temperature was evaluated at the temperatures studies using equation 3.1. This temperature was compared to the experimental values. For  $T_g < 164$ °C, both temperatures were in excellent agreement. For  $T_g > 164$ °C, the measured contact temperature is approximately constant (slightly above the boiling point) and is lower than the theoretical contact temperature. The theoretical and experimental contact temperatures for the range studied are shown in









Figure 3.22. Seki et al. (1978) found that on stainless steel, the measured contact temperature was in agreement with the theoretical contact temperature up to 180°C. For higher temperatures the measured contact temperature was lower than the theoretical temperature.

Figure 3.20 shows that after achieving the contact temperature the solid surface approaches the "equilibrium temperature," (i.e. interface temperature during evaporation). This equilibrium temperature remains nearly constant until a critical droplet thickness ( $\delta^{\star}$ ) is reached on the hot surface. Then, the surface temperature starts to decrease sharply until the droplet has completely evaporated. This sharp decrease in the surface temperature implies an increase in the evaporation rate. As the droplet thickness reduces, the heat transfer through the droplet increases allowing more evaporation to take place from the top surface of the droplet. The time for evaporation after the droplet has achieved the critical thickness is longer for larger droplet at a specified initial surface temperature. This is because larger droplets spread more on the surface, and this large volume require longer time to evaporate. As the initial surface temperature increases this time decreases due to higher heat flux. After the droplet has completely evaporated, the solid recovers smoothly to its initial surface temperature. For  $T_{c} \ge 200$  °C, the interface temperature (which is equal to the contact temperature) was a few degrees above the boiling temperature of water. Since, the droplet diameter after impact on the surface for this temperature range was very large (see Figure 3.29) the evaporation times were therefore very short. After evaporation, the surface recovers to its initial temperature.

Prior to droplet impact, the measured temperatures were compared with steady state calculations. The agreement was within 2°C. This temperature distribution was used to calculate the heat flux inside the solid which was then evaluated at the solid surface. From the surface heat flux, the overall heat transfer coefficient was determined according to equation (3.2), the convective heat transfer was determined according to equation (3.3)

$$q'' = h_0 (T_s - T_m)$$
 (3.2)

$$q'' - h (T_s - T_{\infty}) + \epsilon \sigma (T_s^4 - T_{\infty}^4)$$
(3.3)

Figure 3.23 shows the heat transfer coefficients at the surface of the ceramic block versus surface temperature.

Surface and indepth temperature measurements during droplet evaporation show that the isotherms and the heat flux lines inside the solid matrix may be approximated by oblate spheriodal coordinate system. In such a coordinate system a temperature distribution [adapted from [Keltner (1973)] is given by:

$$T(\zeta, t^*) = \frac{2(T_e - T_s)}{\pi} (1 - 2 \frac{\zeta}{\delta} + \frac{\zeta^2}{\delta^2}) \cot^{-1} \zeta + T_s ,$$
 (3.4)

where  $\zeta$  represents the isothermal lines,  $t^* = \frac{\alpha t}{R^2}$  is the dimensionless time,  $\alpha$  is the thermal diffusivity, R is the radius of the disk, T<sub>s</sub> and











Average Evaporative Heat Flux (KW/m²)











 $T_e$  respectively are the solid surface temperatures before and after the step change, and  $\delta$  is the penetration depth. This is the solution for a step change in temperature over a disk-shaped area on the surface of a half-space. Using this equation, temperatures were evaluated at various locations and at different times. The results were in fair agreement with the measured values. From equation (3.4) it can be shown that the rate of heat arriving at the surface (Q), is given by the following equation

$$Q = 4 R K (T_{a} - T_{a}) (1 + \frac{\pi}{5})$$
 (3.5)

The instantaneous average evaporative heat flux is then determined by dividing by the instantaneous wetted area. The instantaneous evaporation rate may also be determined from Q by dividing it by the heat of evaporation.

The average evaporative heat flux and the average evaporative mass flux were correlated with the initial solid surface temperature as shown in Figures 3.24 and 3.25. Both quantities follow the same pattern; the higher the initial surface temperature and the smaller the droplet, the higher is the average evaporative heat flux and the average evaporative mass flux. This is because at higher temperatures, more energy is available for droplet evaporation, and small droplets have larger conductance due to their smaller thickness on the hot surface. Figures 3.24 and 3.25 also show a slight decrease in both quantities for T<sub>s</sub> > 300°C. The evaporation time and the recovery time curves (Figures 3.26 and 3.27) also show a slight increase at the same temperature. Since, this temperature corresponds to the maximum average evaporative heat flux and the minimum evaporation time, it is likely the beginning of the transition regime. Unfortunately, the experiments could not be continued for higher temperatures, (i.e. in the transition regime), due to the burn out of the heating mantle.

The non-dimensional maximum volume of influence (Vi/V) is shown in Figure 3.28. This quantifies the cooling effect of a single droplet. Figure 3.28 shows that the solid at  $T_g \leq 100$ °C cools more with a 10  $\mu$ L droplet than with larger droplets. This is because the evaporation process for larger droplets attains a steady state and the extra fluid in the larger droplets just increases the evaporation time. Thus, the smaller droplets are more efficient for cooling materials at this range of temperatures.

Figure 3.29 shows the parameter B plotted against the initial solid surface temperature. This parameter is defined as the ratio of maximum droplet diameter during evaporation to the droplet diameter before impact. For  $T_g \ge 200$  °C, B is quite large, since the droplets spread fast and form a thin water film on the hot surface. For this range B decreases as  $T_g$  increases. This is because the droplet is evaporating very fast, and does not have enough time to spread on the hot surface.



Non-Dimensional Max. Volume of Influence





### CHAPTER 4

## DECOMPOSITION OF WOOD

In Chapter 2 it was noted that, prior to ignition investigation, it is important to understand the response of the solid phase to heat, and the resulting production of decomposition products. In this work, products generated when a wood sample heated under controlled conditions are examined in a systematic set of experiments. The set includes 12 experiments, performed at 4 different heat fluxes (approximately 1,2,3, and  $4 \text{ w/cm}^2$ ), and at 3 different moisture contents (dry, 9%, and 17%). Conditions for these experiments were suggested by the specific application of interest. In room fires, the wood burning process may be treated as transient heating and decomposition of a semi-infinite wood sample. The moisture content of wood varies and is influenced by the climate, season and location. The heat flux incident on the wood samples also varies with time and their location relative to an already existing flame in a room fire.

The results of the decomposition experiments are presented in this chapter. Section 4.1 describes the physical aspects of the problem and discusses the importance of the moisture content and its relation to the overall decomposition problem. Section 4.2 reviews the previous

literature. Section 4.3 outlines the procedure used to analyze all the measured species. Section 4.4 includes results and discussion.

#### 4.1 Background

Decomposition of wood refers to the process that produces various chemical species and a residual charred solid upon heating. The physical properties of wood are highly dependent upon its microscopic structure. Wood is predominantly made up of a lignocellulosic structure with various infiltrated substances. It is composed of three major constituents: (i) cellulose (50% by wt), (ii) hemicellulose (25%), and lignin (25%) [Stamm (1964)].

The physical aspects of wood decomposition are associated with heat and mass transfer. Consider a semi-infinite wood sample initially at ambient temperature, subjected to a prescribed heat flux. The sample heats up by pure transient conduction, in which moisture starts to evaporate and an evaporation zone begins to travel into the solid. When the sample surface layer becomes sufficiently hot, it starts to decompose. This decomposition zone moves into the interior of the solid. Consequently, the products of decomposition are driven to the surface of the sample, and flow upward due to buoyancy and mix with the surrounding air. As the interior of the sample becomes hotter the decomposition zone penetrates deeper into the virgin solid, leaving behind a thermally insulating layer of char.

The rate at which the decomposition products and water vapor are generated depends on the response of the solid-phase to the applied heat flux. As pointed out by Simms and Law (1967) and Lee et al.

(1974), the presence of moisture in the solid phase changes its thermophysical properties. This will influence the heat and mass transfer process because of its large heat of vaporization. This heat of vaporization depends upon how the water is held inside the solid matrix. There are four ways by which water can be held in wood: (i) Free or absorbed water: this water is mechanically held in the capillary structure as a result of surface tension forces. Its quantity is limited by the porosity of the wood. The energy required to evaporate this water is only slightly greater than its latent heat. (ii) Bound or adsorbed water: this water is held by hydrogen bonds to the cell walls. Its quantity is limited to 30 percent of the oven-dry weight of the wood. The energy required to evaporate this water increases as moisture content decreases. At the fiber saturation point (~30 percent moisture content) the energy required is the same as for absorbed water. (iii) Water vapor: this is present in the air filling the cell cavities. The quantity of this vapor is normally a very small fraction of the total moisture content. (iv) Water of constitution: this is a part of the molecular structure of cell walls and it is firmly held by chemical bonds. In reality it is not water at all, and is only released upon thermal degradation of wood.

# 4.2 Literature Review

Many investigators have studied decomposition of wood and determined the various products. Schwenker and Beck (1963) detected 37 volatile compounds, and they identified only 18 of them. Goos (1952)

listed 213 compounds which have been identified as products of wood decomposition.

Martin (1965), investigated the decomposition of pure cellulose in helium. He reported a time-dependent analysis of permanent gases and tar. He indicated that there are at least two fundamentally different ways in which cellulose thermally decomposes; one supplies the bulk of the volatile fuel which supports the flaming combustion of the material, the other produces mainly water and the oxides of carbon. Vovelle et al. (1986) studied the decomposition from PMMA and particle board subjected to a variable radiant heat flux under an inert atmosphere. They indicated that the mass loss rate is directly proportional to the instantaneous value of the heat flux absorbed by the surface of the sample.

Lee et al. (1976), investigated wood decomposition in air at two heat fluxes (3.2 and 8.4  $w/cm^2$ ). They indicated that the decomposition process is dependent upon the external heating rate, the total time of heating and the anisotropic properties of wood. Lee and Diehl (1981) investigated the effect of absorbed water (samples with 50% of oven-dry weight) on the decomposition of wood. They indicated that moisture not only changes the solid-phase thermal properties and delays the decomposition process, but also substantially dilutes the decomposition products.

Atreya (1983), investigated wood decomposition experimentally and theoretically. He performed numerous experiments for 10 different kinds of wood in air, and reported transient measurements of surface temperature and evolved mass flux. In his theoretical investigation he

included energetics and kinetics of moisture desorption in his model. He indicated that the desorption of moisture, which has been ignored by previous investigators, has a considerable effect on the energetics of the decomposition process.

Kashiwagi et al. (1987), studied the products generated from wood decomposition in 3 atmospheres of different oxygen concentrations ( $N_2$ , 10%  $O_2$  - 90%  $N_2$ , and air). They indicated that ambient oxygen significantly increases the desorption mass flux and surface temperature. They showed that decomposition rates in air were nearly double those in nitrogen, and surface temperature in air increased as much as 200°C over that in nitrogen at 4 w/cm<sup>2</sup>, due to oxidation of char.

# 4.3 Products Analysis

It is important to know the production and the depletion of major species during decomposition. The equipment used for continuous measurements of  $CO_2$ , CO,  $O_2$ ,  $H_2O$  and total hydrocarbons (THC), as well as the necessary time lag and response time corrections, are described in Chapter 2. As a result, the mole fractions of all measured species were obtained as a function of time. Since the instrument used to measure CO require a dry sample stream, the mole fraction of CO was on a dry basis. This value was converted to a wet basis using the measured mole fraction of  $H_2O$ . The following equation was used:

mole fraction on wet basis - measured mole fraction on dry basis (4.1) x (1-mole fraction of water removed by the drier)

The evolved mass fluxes of all the measured species were then determined using the total mass flow rate of air. The total mass flow rate of air includes the air flow through the critical nozzles (equation B.1 in Appendix B), and leakages from the gap around the sample and top of the tunnel. As explained in Section 2.2.2.3, these leakages were determined by the methane trace method, and they had been calculated from the difference in the methane concentration measurements upstream and downstream of the tunnel. The instantaneous  $0_2$  depletion was determined by subtracting the initial steady state value of  $0_2$  from the measured value at any time.

The sample mass flux was calculated by dividing the time derivative of the measured transient weight-loss by the initial front surface area of the sample. Although it was not possible to measure the organic condensibles (tar), its instantaneous value was determined by difference [(sample mass flux +  $O_2$  depletion) - (sum of the mass evolution rates of  $CO_2$ , CO, H<sub>2</sub>O and THC)]. Finally, the mass fluxes of all the measured evolved species were normalized by the sum of sample mass flux and  $O_2$ depletion to obtain the percent mass flux.

#### 4.4 Results and Discussion

This section describes the results obtained from the set of systematic experiments done on Douglas fir. In these experiments as described in Chapter 2, simultaneous measurements of weight loss, surface, bottom, and indepth temperatures, oxygen depletion, production of  $CO_2$ , CO, total hydrocarbons (THC), and water were made. The

experiments were parameterized with the sample moisture content and the external radiation flux. All experiments were performed in air under non-flaming conditions. A duration of 30 minutes was assumed to be long enough to reveal enough information about the decomposition process. For all three moisture contents, spontaneous ignition occurred at 4  $W/cm^2$ , and data was continuously recorded even after ignition.

# 4.4.1 Sample Mass Flux

Mass flux histories for dry, 9%, and 17% moisture contents at 1,2,3, and 4  $W/cm^2$  are shown in Figures 4.1, 4.3, and 4.5 respectively. These figures show that higher evolved mass flux corresponds to higher heat flux. The general trend for mass flux at high heat fluxes (4, 3, and 2  $W/cm^2$ ) is rise to a peak, fall-off and then attaining a nearly quasisteady state value.

Wichman and Atreya (1987) have developed a simplified model for the decomposition of charring materials in an inert atmosphere. They predict a fall-off in mass flux proportional to the negative one-half power of time. Figures 4.2, 4.4, and 4.6 show the mass flux histories plotted on a log-log basis. The fall-off rule agrees only for some cases and for only a short period of time. For example, the curve for the dry case at  $4 \text{ W/cm}^2$  follows such a time-dependence from 100-240 seconds. This disagreement may occur because of the effect of ambient oxygen concentration on the decomposition process. The behavior of evolved mass flux in the presence of oxygen is expected to be different than in an inert atmosphere, because oxygen not only causes a strongly exothermic reaction at the sample surface, but it could also diffuse



Figure 4.1 Effect of Incident Heat Flux on Time-Dependent Mass Flux for Dry Condition.



Figure 4.2 Effect of Incident Heat Flux on Time-Dependent Mass Flux for Dry Condition



Figure 4.3 Effect of Incident Heat Flux on Time-Dependent Mass Flux for 112 Moisture Content Condition



Figure 4.4 Effect of Incident Heat Flux on Time-Dependent Mass Flux for 11% Moisture Content Condition



Figure 4.6 Effect of Incident Heat Flux on Time-Dependent Mass Flux for 17% Moisture Content Condition

TIME (s)

500

1000

100

50

0.06

0.04

0.02

10

through the porous char to the decomposition front of virgin wood. Thus, to theoretically investigate wood decomposition in air, it is important to include the heat released by char oxidation in such a model.

From Figures 4.1-4.6, we note that the mass flux curves do not start at the origin. This is because the mass flux is an average quantity, determined from the time derivative of the measured mass loss. Since the initial mass evolution from the samples is essentially pure water, the starting point is higher for higher moisture content. The desorption of water from the solid matrix is quickly followed by the evolution of a large amount of tar. This increases the mass flux, and it continues to increase until a thin char layer forms. In this heating stage, the mass flux attains its maximum value. As the sample heating rate increases at higher heat fluxes, the heat-up time is decreased. Thus, for higher heat fluxes the mass flux peak occurs earlier. Vovelle et al. (1984), observed that this peak increases linearly with heat flux.

The production of volatile gases, which escape from the solid, leaves behind a layer of low thermal conductivity char. Formation of the char increases both the absorptivity and emissivity of the surface. But since the thermal conductivity of the char is much lower than that of wood, the net heat transfer through the char is reduced but the net heat loss by radiation is increased. The oxygen in the ambient air also reacts with the char in a highly exothermic reaction and supplies additional heat to the solid. This heat compensates for the reduction of the net heat transfer through the solid due to the formation of char. Furthermore, the char cracks permitting a larger quantity of volatiles

to escape. A combination of these factors is believed to produce the quasi-steady value attained by the decomposition rate after attaining the peak.

## 4.4.2 Sample Temperatures

Measurements of surface, bottom, and indepth temperature of the sample were also made during the experiments. These measurements contain the history of heating of the solid matrix, and they reflect the thermal response of the solid to the irradative heat flux. For example, surface temperature measurement is very important with regard to the heat generated by char oxidation. These reactions take place primarily in a thin surface layer, because their rates strongly depend on temperature and the presence of oxygen. Thus, surface temperature is very useful in determining when the heat of char oxidation is being released as well as at the rate that this heat is transferred to the surroundings.

Surface temperature profiles for dry and 17% moisture content at  $1,2,3 \text{ W/cm}^2$  are shown in Figure 4.7. In this figure higher surface temperature corresponds to higher heat flux. The rate of rise for these temperatures is determined by the moisture content and the irradiative heat flux. This rate increases with higher heat flux and lower moisture content. As an example for the dry case, it took 240 seconds exposure at 1 W/cm<sup>2</sup> for the surface temperature to reach 300°C and only 16 seconds exposure at 3 W/cm<sup>2</sup>, whereas for 17% moisture content it took 1600 sec to reach the same temperature at 1 W/cm<sup>2</sup> (see Figure 4.7).

Char oxidation occurs when the surface temperature is greater than 400°C [Kashiwagi (1987)]. This reaction is strongly exothermic, and is expected to rapidly accelerate the local temperature rise. Figures 4.7 and 4.13 show that, when surface temperature suddenly increases, a significant increase in oxygen depletion is noticed at the same time, as expected. Figure 4.7 shows a sharp rise in surface temperature occurs for the 17% moisture content case at 245 seconds. Simultaneously oxygen depletion increases significantly at the same time (see Figure 4.13). Thus an inflection point in the surface temperature at a value greater than 400°C indicates the start of char oxidation.

Figure 4.7 shows that the start of the oxidation time increases as incident heat flux decreases, and decreases as moisture content decreases at the same incident heat flux. For example, char oxidation occurs for the dry case at 550 seconds when exposed to 2  $W/cm^2$ , and at 215 seconds for 3  $W/cm^2$ . For 17% moisture content the oxidation occurs at 1050 seconds for 2  $W/cm^2$  and at 245 seconds at 3  $W/cm^2$ . This is because the char develops more quickly at higher heat fluxes and lower moisture contents. The effect of moisture is expected to be less significant near the heated surface after char oxidation starts, because for this region the temperature profile is primarily determined by the exothermic heat generated from oxidation of carbon.

Temperature profiles at various locations in the sample corresponding to 9% moisture content at 2 W/cm<sup>2</sup> and 17% moisture content at 3 W/cm<sup>2</sup> are shown in Figures 4.8 and 4.9, respectively. The indepth temperature rose slightly above the boiling point of water, and remained constant for some time before continuing its rise. This is due to the






endothermic vaporization of the adsorbed water. This plateau in temperature-time curves became longer at greater depths in the sample and at higher moisture contents. The upward inflection of the indepth temperatures at 600 seconds in Figure 4.8, is caused by the heat generated from char oxidation (notice the sudden increase in the surface temperature at this time). For different moisture contents it is difficult to compare local indepth temperatures, since thermocouples are not at the same locations for all cases.

#### 4.4.3 Decomposition Products

In this study, oxygen depletion, production of  $CO_2$ , CO, total hydrocarbons (THC), and water are closely examined in order to quantify the effect of the adsorbed water on the decomposition process. The data are presented as mass flux of the permanent gases, and as percent of mass flux. Plots for different moisture contents at different heat fluxes are shown in Figures 4.10-4.17.

The water has a similar trend at 2 and 3  $W/cm^2$ . An initial mass flux peak arises from the desorption water in the solid matrix, followed by a rise to another peak, and eventually a gradual fall-off in the water production. This latter water may include the water of constitution. The water curve at 1  $W/cm^2$  shows only one peak (see Figures 4.16 and 4.17). The quantity of water produced is generally larger for larger heat fluxes and higher moisture contents.

The water production is quickly followed by a large amount of tar whose quantity was determined by difference, as mentioned previously. This organic condensate has an extremely complex in composition.



Figure 4.11 Time-Dependent Product as Percent of Mass Flux at 2 W/cm<sup>2</sup> for 9% Moisture Content Condition.



Figure 4.12 Time-Dependent Mass Flux of Evolved Products at 3 W/cm<sup>2</sup> for 9% Moisture Content Condition.



Figure 4.13 Time-Dependent Mass Flux of Evolved Products at 3 W/cm<sup>2</sup> for 17% Moisture Content Condition.



Figure 4.14 Time-Dependent Mass Flux of Evolved Products at 4 W/cm<sup>2</sup> for Dry Condition.



Figure 4.15 Time-Dependent Mass Flux of Evolved Products at 4 W/cm<sup>2</sup> for 9% Moisture Content Condition.



Figure 4.16 Time-Dependent Mass Flux of Evolved Products at 1 W/cm<sup>2</sup> for Dry Condition



Figure 4.17 Time-Dependent Mass Flux of Evolved Products at 1 W/cm<sup>2</sup> for 17% Moisture Content Condition.

Kashiwagi et al. (1987) investigated its composition by means of capillary gas chromatography coupled with mass spectroscopy. They were able to pass only 20% by weight through the gas chromatograph. They identify more then 40 different species.

The production of combustible gases (CO and THC) at 2 and 3 W/cm<sup>2</sup> shows similar trends; gradual increase until attaining a steady value. For both gases this steady state value is slightly higher for higher heat fluxes and lower moisture contents. But a major difference is the time it took to attain this value. For example, for the 9% moisture content case the steady state value was attained after 950 seconds at 2W/cm<sup>2</sup> (see Figure 4.10), 200 seconds at 3 W/cm<sup>2</sup> (see Figure 4.12), whereas for the case with 17% moisture content it took 400 seconds at 3 W/cm<sup>2</sup> (see Figure 4.13). At 1 W/cm<sup>2</sup>, there were only a small amount of combustible gases produced for the dry case (see Fig. 4.16), and a not noticabe amount for the 17% moisture content (see Figure 4.17). Thus the presence of moisture significantly affects the production of combustible gases by both delaying and decreasing their production.

The oxygen depletion is the amount of oxygen used to react with carbon to form carbon oxides. As mentioned earlier in Section 4.4.2, the time when a significant increase in oxygen depletion occurs corresponds to the time of an inflection point in surface temperature. Generally, the higher the heat flux and lower the moisture content, the larger the oxygen depletion and  $CO_2$  production.

The mass flux of all the measured species was time-integrated for all three moisture contents and for 1, 2, and 3  $W/cm^2$ . Since

spontaneous ignition occurs at  $4 \text{ W/cm}^2$  for all cases, this set of experiments will be discussed separately later in this section.

The effect of the heat flux and the moisture content on the integrated mass products are shown in Figures 4.18 - 4.21. These Figures show that a larger amount of water production corresponds to higher heat flux and higher moisture content. Also larger oxygen depletion and  $CO_2$  production correspond to higher heat flux and lower moisture content. Finally, the production of combustible gases is larger for high heat fluxes and lower moisture contents, as expected. From these results, it is clear that the presence of adsorbed water in the solid matrix reduces the average fuel rate evolved during the decomposition process, by delaying their production.

At 4 W/cm<sup>2</sup>, and for all moisture content cases (dry, 9% and 17% moisture content), spontaneous ignition occurred. In reality, these experiments belong to the discussion on ignition, but since they were performed in the absence of a pilot flame like the rest of the decomposition experiments, discussion of these result is presented here.

The general trend of the measured species in the three cases is very similar (see Figures 4.14 and 4.15). At time of ignition there is a sudden increase to a sharp peak, then fall-off, and finally attainment of a steady state value. The sharp peak is caused by the large amount of fuel available in the gas phase at time of ignition. From visual observation, the flame then reduces in size, because it is now fed by the fuel evolved from the solid phase. As noticed previously in the decomposition experiments, the fuel produced attained a steady state value even with the char formation. This fuel with a constant value of



Figure 4.18 Time-Integrated Product Mass and Composition (shown as percentages next to each bar) as Function of Incident Heat Flux for 9% Moisture Content Condition.



Figure 4.19 Time-Inetegrated Product Mass and Composition (shown as percentages next to each bar) as Function of Moisture Content at 2 W/cm<sup>2</sup>.



Figure 4.20 Time-Integrated Product Mass and Composition (shown as percentages next to each bar) as Function of Incident Heat Flux for 17% Moisture Content Condition.



Figure 4.21 Time-Integrated Product Mass and Composition (shown as percentages next to each bar) as Function of Moisture Content at A Heat Flux of 3 W/cm<sup>Z</sup>.

the oxygen concentration in ambient air produces nearly constant values of  $CO_2$  and  $H_2O$ .

The rate of water release in all three cases is very close. This because the adsorbed water has been evolved prior to the time of ignition and the combustion water is much greater than adsorbed water. The  $CO_2$  production was larger for the dry case, because more fuel evolved in this case is expected to be more. Oxygen depletion was larger for the dry case, which was needed to produce a large amount of  $CO_2$  and  $H_2O$ .

The time of ignition for the dry, 9%, and 17% cases was 88, 130, and 158 seconds respectively. Thus the presence of moisture increase the time required for spontaneous ignition by diluting and delaying the production of fuel. The average surface temperature at spontaneous ignition was approximately 530°C. This temperature is expected to be lower for piloted ignition, which is the subject of the next chapter.

#### Chapter 5

# Piloted Ignition

In the previous chapter, it was shown that the presence of moisture in the wood matrix delayed the decomposition process and diluted the decomposition products. These factors are expected to have a significant effect on the ignition process. The work presented in this chapter attempts to experimentally quantify the effect of adsorbed water on the piloted ignition of wood. The experiments were performed for four moisture contents (dry, 11%, 17% and 27%) and at various incident heat fluxes ranging from 1.8 to  $3.7 \text{ W/cm}^2$ .

This chapter is divided into four major sections. Section 5.1 describes the physical aspects of the problem, factors influencing the ignition process and the relation of moisture to ignition. Section 5.2 reviews the previous literature. Section 5.3 describes the experimental apparatus and procedure. Section 5.4 describes the results of the experiments; a model to correlate the ignition data is also derived and discussed.

# 5.1 Background

Ignition refers to the appearance of flame in the volatile gas stream evolved from a solid exposed to external heating (usually

radiative). Depending upon whether the ignition occurs with or without the aid of an external ignition source, the result is accordingly classified as spontaneous (auto-) or piloted (forced) [Welker (1970)]. For piloted ignition the actual process, as noted in Chapter 1, is quite complicated. The solid must first decompose to produce combustible gases. The combustible gases then mix with the surrounding air to produce a mixture within the flammability limits of composition. The mixture needs an external source of heat, such as a pilot flame, to initiate combustion. Spontaneous ignition will occur only when two conditions are satisfied at some point in the proximity of the exposed surface. First the air-fuel mixture must attain flammability limits, and second, this mixture must achieve a thermal condition that enables it to automatically react in an exothermic manner to yield a flame without the aid of any external source of heat. It is clear that the attainment of spontaneous ignition is more difficult than piloted ignition.

The factors influencing ignition of a sample exposed to a radiant heat flux may be separated into two general categories namely, those external and internal to the sample. Factors external to the sample are environmental variables such as temperature, composition and velocity of the surrounding gases, and the magnitude and spectral quantity of the external radiant flux. Factors internal to the sample are its thermophysical and thermochemical properties, its moisture content, and whether the sample is thermally thick or with negligble temperature gradient (thin materials). Experimentally, except for the sample properties, all other variables can be controlled.

A common feature of cellulosic materials is that they are hygroscopic and porous. Thus, a large area is available for them to adsorb moisture from the surrounding air. Moisture content of these materials is variable, and is influenced by the climate, season and location. Normally, wood at room temperature and humidity contains about 10 to 15% water by weight, and may contain up to 30% (fiber saturation point) if it is in equilibrium with air saturated by water. As discussed in Chapter 4, this water is held by hydrogen bonds to the cell walls, and is called adsorbed water. Its presence in the wood matrix is expected to have a significant effect on the ignition process. The obvious effect is to increase the time required for ignition by changing the heat transfer and hence the rate of temperature rise. This is because the presence of moisture increases the values of the thermal properties such as the thermal conductivity and the volumetric specific heat. Furthermore, since the specific heat of water vapor is twice that of nitrogen, additional fuel is required to attain the same limit flame temperature.

# 5.2 Previous Literature

The ignition of cellulosic material (both auto- and piloted) has been an active area of research, and several excellent reviews have been published on the subject [Welker (1970), Kanury (1972), and Steward (1974)].

Numerous techniques have been developed to investigate the ignition phenomenon. Various heat sources has been used, namely gas-fired radiant panels [Simms and Co-workers (1960,1963,1967)], high tungsten

filament lamps [Smith et al. (1970)], carbon arc [Martin (1969)], CO<sub>2</sub> laser [Kashiwagi (1981)], electrical heating elements [Atreya (1983)], etc. The experiment essentially constituts of exposing a sample to a known external radiant flux and recording the timee required to ignite the sample in the presence of a pilot flame. The ignition times measured by various investigators have not always been in good agreement. For example, the time for ignition of wood irradiated by a tungsten lamp is about four times larger than by diffusion flames at the same incident irradiance [Wesson et al. (1971)]. The primary reason for this disagreement was found to be due to the difference in the spectral quality of the incident radiation relative to the spectral absorptance of wood. Thus the spectral nature of the heat source to be used in the experiments must closely match the intended application. For building fires this implies that the heat source should have an effective blackbody radiation temperature of about 1200°K.

Several ignition criteria have been proposed such as: critical surface temperature at ignition [Simms (1963)], critical mass flux [Bamford et al. (1946)], critical char depth [Sauer and Interim (1956)], critical mean solid temperature [Martin (1965)], etc. Of these, critical fuel mass flux at ignition seems to be physically the most correct since it can be related to flammability limits. Surface temperature has proved to be the most useful ignition criterion since it can be conveniently related to fire spread [Atreya (1983)]. However, only a few investigators [Atreya (1983)], Kashiwagi (1981), Garden (1953)] have directly measured the surface temperature at ignition; others have estimated it by extrapolating measured indepth

temperatures [Martin (1965)], or by using a linear heat conduction theory [Simms (1960,1963,1967)]. The reported surface temperature at piloted ignition for wood ranges from 300-540°C. Atreya's recent work (1983) has shown that with the correct interpretation of measured surface temperatures a somewhat narrower range [375  $\pm$  30°C] is obtained. In this regard, it is important to note that all of the above criteria are based on an indirect quantity which is assumed to be closely related to ignition.

The importance of moisture content in relation to fire tests has long been recognized. Researchers have carefully controlled the moisture content of their samples. However, few investigators have addressed its effect on ignition. Martin et al. (1958), studied autoignition of  $\alpha$ -cellulose conditioned at different relative humidities using carbon arc as a radiant source. Simms and Law (1967) performed both auto- and piloted ignition tests on samples with different moisture contents using gas-fired radiant panels. In both these studies moisture was found to significantly affect the ignition process.

Atreya (1983) investigated (experimentally) piloted ignition for different types of wood. He showed that critical surface temperature is a reasonable and convenient criterion for ignition. He indicates that this temperature increases for low heat flux experiments due to the build up of char. Later Atreya et al. (1987) investigated the effect of sample orientation on piloted ignition and opposed-wind flame spread. They used two types of wood (red oak and mahogany) to investigate two orientations (horizontal and vertical). They reduced the experimental data according to the thermal flame spread theory

using the measured surface temperature. They indicated that as long as the temperatures are defined consistently with the thermal theory, the results are orientation independent within the measurement error.

The mathematical description of the heat transfer for the ignition problem has been widely investigated. The problem is usually considered to be one dimensional with all heat transfer occurring normal to the sample surface. The cases investigated produce correlations of different forms, because different boundary conditions and various assumptions were used in each case.

Butler et al. (1956), assumed that the sample is inert, undergoes no decomposition, front surface is opaque to the incident radiation, no heat lossess, and the rear surface is insulated. They used the solution of this problem as the basis for correlating their ignition data. They omitted the surface temperature rise at ignition because of the difficulties they encountered in attaching physical significance to it. They also indicated that the temperature rise at ignition was relatively constant. Thus, they claimed no significance error was introduced by omitting it.

Simms and Law (1967), included convective cooling losses at the front surface in the boundary condition. However, in order to correlate their ignition data for wet and dry woods, they had to fix two unmeasured parameters, the convective heat transfer coefficient (h) and surface temperature at ignition ( $T_s$ ). For piloted ignition they used  $T_s = 360$ °C and  $h = 8.6 \times 10^{-4} \text{ cal/cm}^2 \cdot \text{sec}^\circ\text{C}$ ; for spontaneous ignition they used  $T_s = 525$ °C and  $h = 14 \times 10^{-4} \text{ cal/cm}^2 \cdot \text{sec}^\circ\text{C}$ . The

convective coefficients used in their work are larger than those used by other investigators. For example, Alvares et al. (1969) used h =  $2.8 \times 10^{-4} \text{ cal/cm}^2 \text{ sec}^\circ \text{C}$  based on free convection heat transfer theory. In the opinion of Welker (1970), the reason for a large value of h (required to obtain a correlation) in Simms work may be that the heat losses by reflection of incident radiation and emission of radiation from the surface were ignored.

Atreya and Wichman (1987) developed an approximate analytical model for the piloted ignition process. They solved for time, surface temperature and mass flux at ignition, and showed that the predictions were in good agreement with the experimental measurements. Tzeng et al. (1988) developed a theoretical model for the piloted ignition process. They solved the equations numerically, and examined the location of the ignition source, fuel mass evolution rate from the surface, and the surface temperature of the solid. They showed that the (1) model adequately explains the pre-ignition flashes that are often observed experimentally, (ii) provides a rational criterion for positioning the pilot flame, (iii) indicates that the heat losses to the surface play an important role, and shows that the fuel flow rate by itself is insufficient for predicting the onset of piloted ignition.

Recently, Mikkola and Wichman (1988) suggested a correlation that relates the incident heat flux (F) to the negative one-half power of ignition time (t). They deduced this relation from the solution of the one-dimensional heat conduction equation for a semi-infinite solid with constant external heat flux, linearized surface heat loss, and a constant initial sample temperature. They used wood ignition data

available in the literature and plotted  $t^{-0.5}$ vs F, and showed that the data collapsed onto separate straight lines whose common abscissal intercept is approximately constant (12.5 KW/m<sup>2</sup>). They explained this constancy for the different types of wood by the similarity in the chemical structure of wood. They indicated that this value (12.5 W/m<sup>2</sup>) is the minimum heat flux for ignition, and provided additional support for their correlation, by showing that the data for polypropylene and polyurethane, have different slopes and intercepts. They explained that this is because their chemical structure are different from wood.

# 5.3 Apparatus and Procedure

A schematic of the experimental arrangement used for these tests is shown in Figure 5.1. Since the experimental apparatus used is described in detail in Chapter 2, only the relevant aspects are considered here. The external radiation was provided by electrical radiant heaters. These heaters have a maximum backbody temperature of 1200°K and are therefore well suited to simulate external radiation in building fires. A small natural-gas flame was located in the mixing layer, pointing downward. The pilot flame was off the sample edge at a height of 0.5" from the sample surface (see Figure 5.1). The configuration and the position of the pilot were chosen in order to avoid heating the sample.

The samples were prepared and instrumented with thermocouples. The samples were then dried by a standard procedure in a temperature-





controlled chamber at 105°C until a constant weight was achieved. Then they were conditioned in a temperature-humidity-controlled chamber for 5-6 weeks until a constant weight was obtained. This procedure of fixing the moisture content in all samples used for a particular set of experiments minimized the changes in the results due to variations in moisture content. The sample edges and the bottom were wrapped with aluminum foil to minimize any edge effects.

All the experiments were performed under identical conditions of air flow rate and atmospheric pressure above the sample. This is because for piloted ignition the mixture of the products of decomposition generated by the solid, and the surrounding air, should be within flammability limits. The flow rate of air above the sample may considerably affect the results.

The procedure consisted of placing the instrumented sample flush with the Kaowool insulating boards on a load cell to continuously monitor its weight loss. The sample was then exposed to a known external radiation. This external radiation was also continuously monitored by a heat flux sensor located upstream the sample (see Figure 5.1). A calibration between the heat flux at the sensor location and the heat flux at the sample location was used to determine the incident radiation on the sample surface. A video camera was focused on the sample, and recorded the entire experiment; this provided information about ignition behavior and a precise measurement of ignition time.

For a given moisture content and external radiation, in addition to the time required for ignition, the following measurements were also recorded as a function of time: (i) front and back surface temperatures, (ii) mass evolution rate of water vapor, (iii) production

rate of combustible gases, and (iv) weight loss rate of the wood sample.

## 5.4 Results and Discussion

This section describes the results obtained from the piloted ignition experiments. These experiments were parameterized with the sample moisture content, and the external radiant flux. Thus, except for these two parameters, all the experiments were performed under identical conditions.

# 5.4.1 Ignition delay time

Ignition delay times were measured for the samples with different moisture contents under various external radiant fluxes. This value was provided from recorded tapes for all the experiments. A summary of all the ignition data is shown in Figure 5.2. This figure shows that the ignition time is a function of moisture content and the incident heat flux. Generally as incident heat flux decreases, the time required for ignition increases, and as the moisture content increases, this time increases at the same incident heat flux. For example, a dry sample ignited at 1.5 minutes at  $1.9 \text{ W/cm}^2$ , whereas a sample with 27% moisture content ignited at 16.5 minutes, for the same incident heat flux. Thus, the presence of moisture in a sample would increase the total energy needed to ignite the sample by increasing its ignition time.

The higher heat flux data (more useful for fire spread purposes)



Figure 5.2 Effect of Moisture on Ignition Delay Time.

shows also a large difference in ignition time. For example, a dry sample ignited at 24 seconds at  $3.5 \text{ W/cm}^2$ , whereas a sample with 27% moisture content ignited at 90 seconds at the same incident heat flux, suggesting that fire spread on wood containing moisture would be slower than on dry wood.

### 5.4.2 Correlation of results

Ignition time data were correlated on the basis of a relationship suggested by the solution of the one-dimensional heat diffusion equation in the solid.

Consider a semi-infinite solid initially at a constant temperature  $(T_{\infty})$ . At t > 0 the solid is exposed to a prescribed heat flux (F). Assuming that the solid is inert and undergoes no thermal decomposition. Since the heat transfer through an inert solid is by conduction only, the following form of the one-dimensional heat diffusion equation applies

$$\rho C \frac{\partial^2 \theta}{\partial t} - K \frac{\partial^2 \theta}{\partial x^2} \quad \text{for } x > 0, \ t > 0 \tag{5.1}$$

where  $\theta = T - T_{\infty}$ . The solid is assumed to be at the temperature of its surroundings prior to the experiment, therefore

$$\theta = 0$$
 for  $t \le 0, x > 0$ 

The boundary condition is obtained from the energy balance at the solid surface (i.e., heat diffused into the solid - incident heat flux convective heat loss - radiative heat loss). Thus, the boundary condition at the front surface becomes

$$-K \frac{\partial \theta}{\partial x} \Big|_{x=0} = f_{s}(\theta_{s}, T_{\omega}, t) = \epsilon \left[F - h \theta_{s} - \sigma((\theta_{s} + T_{\omega})^{4} - T_{\omega}^{4})\right] \quad (5.2)$$

where  $\epsilon$  = the emissivity (or absorptivity) of the surface,

- h = heat transfer coefficient/ $\epsilon$ ,
- $\sigma$  = Stefan-Boltzmann constant,
- F the prescribed incident heat flux,
- $\theta_{\rm g}$  T<sub>g</sub> T<sub>w</sub> where T<sub>g</sub> is the surface temperature,
- f = heat flux going into the semi-infinite solid at the front
  surface,

and  $\rho$ ,C, and K are, respectively the density, heat capacity, and thermal conductivity of the solid.

Atreya (1983), obtained an analytical solution for the above problem by the use of approximate integral methods. He solved for the ignition time and gave it in the following form;

$$t = M \left[ \frac{\theta_{s}^{2}}{2f_{s}^{*2}} + \frac{r}{\beta^{3/2}} \ln \left[ \frac{(2s\theta_{s} + r - \sqrt{\beta})(r + \sqrt{\beta})}{(2s\theta_{s} + r + \sqrt{\beta})(r - \sqrt{\beta})} \right] - \frac{\theta_{s}(r + 2s\theta_{s})}{\beta f_{s}^{*}} \right], \quad (5.3)$$

where  $M = \frac{2}{3} \frac{\rho CK}{\epsilon^2}$ ,  $r = -(h + 4 \sigma T_{\infty}^3)$ ,  $s = -\frac{25}{3} \sigma T_{\infty}^2$ ,  $\beta = (r^2 - 4Fs)$ , and  $f_s^* = \frac{f_s}{\epsilon} = F + r \theta_s + s \theta_s^2$  This equation was simplified by approximating the logorithm term, and substituting the value of  $f_s^*$  as the net heat flux diffused into the solid [i.e.  $f_s^* = (F - L)$ ], where L, is the total heat loss by convection and radiation. The final form of equation 5.3 is

$$t = \frac{M}{2} \frac{\theta_{s}^{2}}{f_{s}^{*2}} \left[ 1 + \frac{10r (1 - L/F)^{2}}{16s \theta_{s} (1 - r^{2}/4Fs)^{2}} - \frac{2(r/\theta_{s} + 2s) (1 - L/F)}{4s(1 - r^{2}/4Fs)} \right]$$
(5.4)

The terms inside the bracket of equation 5.4, were examined closely, and they were evaluated for a wide range of heat fluxes (F). Let

$$e_{1} = \frac{10r (1-L/F)^{2}}{16s \theta_{1}(1 - r^{2}/4Fs)^{2}}, e_{2} = \frac{2(r/\theta_{s} + 2s) (1 - L/F)}{4s(1 - r^{2}/4Fs)}$$

 $e_3 - e_1 + e_2$  and  $e_4 - e_3 + 1$ . Figure 5.3 shows the value of each term plotted against the incident heat flux. Note that at piloted ignition the surface temperature  $\theta_s$  is approximately constant. Thus, the only

variable is F. For the range of heat fluxes studied  $(1.8 - 6 \text{ W/cm}^2)$  which are representative for igntion), two conclusions may be drawn from Figure 5.3: (i) the variation in each of the terms  $e_1$  and  $e_2$  is small compared to the dominant term which has a value of 1.0 (ii) the value of all the terms in the bracket  $(e_4)$  can be approximated by a constant with an error of less than  $\pm 6$ % in the range of interest.



TERMS OF EQUATION 5.4

By substituting the value of M given under Eq. (5.3), equation (5.4) may be written as

$$t \simeq \frac{e_4 \rho C K \theta_s^2}{3 \epsilon^2 f_s^{*2}}$$
(5.5)

Assuming that the front surface temperature at the time of ignition is constant, so that it is no longer a correlating parameter, the heat flux into the solid at the instant of ignition is given by

$$f_s^* = C^* t^{-0.5}$$
, (5.6)

where, 
$$C^* = \sqrt{\frac{e_4 \rho C K}{3\epsilon^2}} \theta_s$$
. (5.7)

Substitute the value of  $f_s^*$  back into equation 5.2, the following relation between incident heat flux and time for ignition is obtained

$$F = C^* t^{-0.5} + L$$
, (5.8)

where  $C^{\star}$  is given by equation 5.7, and L represents the heat loss by convection and radiation. L, is given by the following equation

$$L = h \theta_{g} + \sigma((\theta_{g} + T_{\omega})^{4} - T_{\omega}^{4}) \quad . \tag{5.9}$$

Equation 5.8 is the basic relationship for correlating the data of ignition time as a function of incident heat flux. The ignition time to the negative half power was plotted versus incident heat flux. The data for the four moisture contents are shown in Figure 5.4; the measured values were shown by points and the least square fit by the straight lines. As shown in the figure each moisture data collapsed onto a separate straight line, the intercept corresponds to L, and the slope corresponds to  $C^*$  in equation 5.7.

The parameters L and C<sup>\*</sup>, were calculated for each value of the moisture contents; L was determined from equation 5.9. For  $\theta_{\rm g}$ , the value of the measured surface temperature at ignition was used. The value of C<sup>\*</sup> was calculated from equation 5.7. The values for the thermophysical properties ( $\rho$ CK) were obtained from the literature [Parker (1988), Simms (1967)]. The oven-dry properties were given in Parker (1988), who used the same type of wood. The properties for the rest of the moisture contents were determined by using the formulas of Simms (1967). A value of 1.75 was used for  $\mathbf{e}_4$ . This is the average value of all the terms in the bracket of equation 5.4 from Figure 5.3.

A comparison of the derived and the calculated values of L and  $C^*$ are presented in Table 5.1. Because surface temperature at ignition was found to increase with moisture content (this will be discussed in the next section) it would be expected that the heat loss increases. The derived and the calculated values of L show an increase, as the moisture content increases. The reason for a low value in the experimental heat loss (L) at 27% moisture content is not known to the





author; more data is needed to confirm this value. Because the thermophysical properties of wood increase as moisture content increase, it is expected that the value of  $C^*$  increases. The experimental values of  $C^*$  have an average deviation of 10% from the calculated values; this may be because of the error introduced by approximating all terms in the bracket of equation 5.4 by a constant. Such an approximation is expected to introduce ~ 12% error as discussed earlier in this section. The sixth column in Table 5.1. was obtained using a computer program (NLINA), provided by Professor J. Beck (1977). This program can be used to estimate parameters for both linear and non-linear cases. The calculated heat loss values along with the ignition data were used to estimate  $C^*$ .

The ignition correlation reveals the strong influence of the heat loss and moisture content upon ignition characteristics. It shows that, as the time required for ignition tends to infinity, the heat loss tends to the incident heat flux (see equation 5.8). Hence the critical incident heat flux, below which ignition is impossible, is given by L. This critical flux is, in effect, the rate at which heat is lost from the surface at ignition temperature. Because surface temperature has been found to increase with moisture content, it would be expected that the critical incident heat flux would increase (see Table 5.1). Thus the moisture has a significant effect on piloted ignition; it increases the ignition delay time and more importantly increases the critical incident heat flux.

Piloted ignition is closely related to fire spread. In fire spread, the volatiles produced from the heated surface are ignited

either by the source of the heat itself or by the aid of external sources. Thus, fire spread may be regarded as a rapid succession of piloted ignitions. This suggests that the critical heat flux for piloted ignition is a very important concept for fire spread. Using this value in building regulations will result in a safe separation distances between housings, and this will reduce the risk of fire spread to neighboring properties.

# TABLE 5.1

| Moisture<br>Content | L          |                   | c*         |                   |  |
|---------------------|------------|-------------------|------------|-------------------|--|
|                     | Calculated | Experi-<br>mental | Calculated | Experi-<br>mental | Experimental Using<br>Calculated Heat Loss |
| Dry                 | 1.01       | 1.13              | 8.6        | 9.78              | 10.73                                      |
| 118                 | 1.06       | 1.20              | 13.2       | 15.53             | 14.84                                      |
| 17%                 | 1.10       | 1.32              | 15.54      | 13.3              | 15.02                                      |
| 27%                 | 1.30       | 1.14              | 20.86      | 22.85             | 20.20                                      |

Comparison of Calculated and Experimental Values of C<sup>\*</sup> and L

# 5.4.3 Surface temperature

Surface temperature profiles for dry and 11% moisture content at 2,3, and 4  $W/cm^2$  are shown in Figure 5.5 and 5.6 respectively. These Figures show that the rate of rise of surface temperature increases as the incident heat flux increases. Also the rate of rise increases as moisture content decreases at the same incident heat flux as shown in Figure 5.7.
At the time of ignition the flame appears in the gas phase, and this causes a sudden increase in the surface temperature. The arrows in Figures 5.5-5.7 indicate the time of ignition. These figures show that ignition time increases as heat flux decreases (see Figures 5.5 and 5.6), and increases as moisture content increases at the same incident heat flux (see Figure 5.7). Data for surface temperature at ignition are summarized in Figure 5.8. There is some scatter in the data, but the trends are clear. At any incident heat flux, the surface temperature at ignition is higher for higher moisture content, and at any moisture content, it increases as heat flux decreases. This increase in ignition temperature is due to the slow decomposition of the solid at the surface, and the resulting build up of the low thermal conductivity char layer prior to ignition.

The sample moisture content also had a significant effect on surface temperature at auto-ignition. In the decomposition experiments discussed in Chapter 4, it was pointed out the auto-ignition occurs at 4  $W/cm^2$  for the 3 moisture cases studied. The surface temperature of the sample at ignition in these experiments were 510, 525, 550°C for dry, 9%, and 17% moisture content, respectively. These temperatures are much higher than the piloted ignition temperatures under the same conditions. This is because in the case of piloted ignition, the sample surface temperature must rise only high enough to decompose the sample, provided that decomposition is rapid enough to produce enough combustible gases to form a combustible mixture with the surrounding air at the pilot location. On the other hand auto-ignition, must be initiated by heating either the sample surface to the point where it ignites the gases, or by



Effect of Incident Heat Flux on Surface Temperature for Dry Condition Pigure 5.5



Effect of Incident Heat Flux on Surface Temperature for 11% Moisture Content Condition



Effect of Moisture Content on Surface Temperature at an Incident Heat Flux of  $2.65 \text{ W/cm}^2$ . Figure 5.7







heating the gases themselves to the ignition point. In either case, this would require a higher surface temperature.

Sample surface and bottom temperatures during a typical test of piloted ignition are shown in Figure 5.9. This Figure shows that the bottom temperature increased only by 2-3°C before the time of ignition. Thus the assumption that the samples were thermally thick used in section 5.4.2 is valid.

#### 5.4.4 Sample mass flux

Mass flux histories for dry and 17% moisture content are shown in Figures 5.10 and 5.11 respectively. The arrows in these Figures indicate the time of ignition. These Figures show that the mass flux increased dramatically at the time of ignition and continue to increase. Meanwhile a char layer on the sample surface forms. This char layer is not completely inert but it is much more resistant to the evolved gases than original wood. This is because char has lower thermal conductivity, and the heat penetrating through this thick char would decrease. This consequently decreases the volatile mass flux, which continues to decrease until attaining a nearly steady state condition.

Numerical values of the mass flux at the time of ignition for the different moisture contents at various incident heat fluxes were determined from the mass flux histories. These values are presented in Table 5.2. This table shows that the mass flux increases as the incident heat flux increases, and at the same incident heat flux, it increases as moisture content increases. This is because the initial mass evolution from the samples is essentially desorption of water followed by a large amount of tar. These quantities are evolved at





higher rates as moisture content and incident heat flux increases.

Table 5.2 shows that a mass flux of about 0.22 mg/cm<sup>2</sup>s is necessary for piloted ignition to occur. Bamford et al. (1945) proposed that piloted igntion of vertical slab of wood is possible when the mass flux of the decomposition products exceeds a critical value. For deal, oak, and pine wood, they gave this critical mass flux as approximatley 0.25 mg/cm<sup>2</sup>.s.

### Table 5.2

Comparison of Mass Flux at Ignition For Different Moisture Content At Various Heat Fluxes (F)

| Moisture<br>Content | F<br>W/cm <sup>2</sup> | Mass Flux<br>mg/cm <sup>2</sup> s | F<br>S/cm <sup>2</sup> | Mass Flux<br>mg/cm <sup>2</sup> s | F<br>W/cm <sup>2</sup> | Mass Flux<br>Mg/cm <sup>2</sup> s |  |
|---------------------|------------------------|-----------------------------------|------------------------|-----------------------------------|------------------------|-----------------------------------|--|
| Dry                 | 1.85                   | 0.22                              | 2.6                    | 0.28                              | 3.48                   | 0.48                              |  |
| 11%                 | 1.80                   | 0.28                              | 2.76                   | 0.37                              | 3.53                   | 0.50                              |  |
| 17%                 | 1.84                   | 0.30                              | 2.36                   | 0.44                              | 3.21                   | 0.42                              |  |
| 278                 | 1.92                   | 0.37                              | 2.77                   | 0.47                              | 3.62                   | 0.58                              |  |

### 5.4.5 Products evolved

In this study oxygen depletion, production of  $CO_2$ , CO, total

hydrocarbons [THC], and water were measured before and after the time of ignition. The procedure used for the analysis of these products is the same as used for decomposition experiments, as described in section 4.3. The data is presented as mass flux of permanent gases, and as percent of mass flux. Plots for different moisture contents at various heat fluxes are shown in Figures 5.12 - 5.21.



Figure 5.12 Mass Flux of Evolved Products at Incident Heat Flux 3.5 W/cm<sup>2</sup> for Dry Condition



Figure 5.13 Products as Percent of Mass Flux at Incident Heat Flux 3.5 W/cm<sup>2</sup> for Dry Condition



Figure 5.14 Mass Flux of Evolved Products at Incident Heat Flux 2.6 W/cm<sup>2</sup> for Dry Condition



Figure 5.15 Products as Percent of Mass Flux at Incident Heat Flux 2.6 W/cm<sup>2</sup> for dry Condition



Figure 5.16 Mass Flux of Evolved Products at Incident Heat Flux 2.75 W/cm<sup>2</sup> for 11% Moisture Content Condition



Figure 5.17 Proudcts as Percent of Mass Flux at Incident Heat Flux 2.75 W/cm<sup>2</sup> for 11% Moisture Content Condition



Figure 5.18 Mass Flux of Evolved Products at Incident Heat Flux 2.77 W/cm<sup>2</sup> for 27% Moisture Content Condition



Figure 5.19 Products as Percent of Mass Flux at Incident Heat Flux 2.77 W/cm<sup>2</sup> for 27% Moisture Content Condition



Figure 5.20 Mass Flux of Evolved Products at Incident Heat Flux 1.85 W/cm<sup>2</sup> for Dry Condition



Figure 5.21 Mass Flux of Evolved Products at Incident Heat Flux 3.21 W/cm<sup>2</sup> for 17Z Moisture Content Condition.

Before ignition, the gases evolved are products of decomposition. This was the subject of discussion of Chapter 4. These Figures show that water is a major component of the evolved gases. The higher the incident eat flux and moisture content, the higher is the water production rate. As pointed out in Chapter 4, the presence of moisture in the samples delayed and diluted the combustible gases. This is the reason why ignition occurs at later times for samples with higher moisture content.

After the time of ignition, the general trend of the measured species is very similar for all moisture content cases at the various incident heat fluxes; a sudden increase to a sharp peak, fall-off, and then attainment of a steady state value. The sharp peak is caused by the burning of the large amount of fuel available in the gas phase at the time of ignition. Meanwhile a char layer starts to form which reduces the rate of the evolved gases including the combustible gases. This results in a reduction of the product rates, which eventually attain a steady state.

Figures 5.12 - 5.21 show that the production of water after ignition increases as the incident heat flux increases, and at the same incident heat flux, the water production does not change significantly with the increase in moisture content. This is because the combustion water is much larger than the adsorbed water, and a large quantity of the adsorbed water was evolved prior to the time of igntion. This is supported by the fact that water production prior to ignition is higher for higher moisture contents. These Figures also show that the production of  $CO_2$  decreases as moisture content increases at the same

incident heat flux, and decreases as incident heat flux decreases at the same moisture content. Oxygen depletion follows exactly the same trend as  $\rm CO_2$ , as expected. This is because, as pointed out in Chapter 4, the production rate of combustible gases decreases as the heat flux decreases, and are diluted by the presence of moisture in the sample. Figures 5.12-5.21 also show that the production of combustible gases is almost zero, since these gases burn to produce  $\rm CO_2$  and water.

Figure 5.18 shows that at about 475 sec, as the flame gradually died out,  $CO_2$  production and  $O_2$  depletion gradually decreased, while the production of combustible gases (CO and THC), started to gradually increase. After the flame died-out completely a steady state was attained. They may now be considered as decomposition products.

The tar in some experiments shows a negative value (see Figure 5.12) because it was determined by differences (as discussed in Section 4.3), and any measurement error will show in the tar value.

### CHAPTER 6

### CONCLUSIONS

This chapter presents a summary of the important results for different components of this work. Section 6.1 summarizes the conclusions derived from the results of the droplet evaporation on the hot porous and non-porous solids. Section 6.2 summarizes the conclusions obtained from a set of systematic experiments performed to investigate the effect of water on both thermal decomposition and ignition. Some recommendations for future work are presented in Section 6.3.

### 6.1 Droplet Evaporation Experiments

The transient surface and indepth temperature data obtained during droplet evaporation reveal the thermal behavior of both the porous and the non-porous solids. The following conclusions are apparent from this investigation:

1. The only similarity in the thermal behavior of both porous and nonporous solids is the agreement in the experimental contact temperature. This is because it occurs during the initial contact and the droplet sees both solids as a semi-infinite body.

2. The theoretical and experimental contact temperatures are in good agreement up to the boiling point of water and then diverage with the experimental contact temperature becoming roughly constant at a value slightly greater than the boiling point.

3. During the droplet evaporation process, surface and indepth temperatures for the non-porous solid remain nearly constant whereas for the porous solid there was a continuous decrease in these temperatures.

4. A thermocouple in the porous matrix at the same location as that of the non-porous matrix cools faster under identical conditions.

5. Both, the recovery time and the envelope of droplet influence were larger for the porous solid. These results confirm the indepth cooling of the porous solid due to water penetration.

6. The evaporation time is longer for the non-porous solid than for the porous solid for the same droplet diameter and under identical conditions. This is because part of the droplet diffuses into the porous solid matrix.

7. Smaller droplets are more efficient for cooling non-porous solid at or below 100°C. This is because the evaporation process for larger droplets attains a steady state and the extra fluid in the larger droplets just increase the evaporation time. This phenomenon was not observed in the porous solid because evaporation process never quite attains a steady value.

8. For the non-porous solid, the instantaneous evaporation rate, and the instantaneous average evaporative heat fluxes were determined from the transient measurements of surface and indepth solid temperatures, and the droplet diameter on the solid surface. Although the total heat transfer is more for large droplets the average evaporative heat flux is higher for smaller droplets. This is because small droplets have larger conductance due to their smaller thickness on the hot surface.

### 6.2 Thermal Decomposition and Piloted Ignition Experiments

The following conclusions are drawn from this investigation:

1. The presence of moisture in the wood sample has a significant effect on the surface temperature and char oxidation. As the moisture content increases, the rate of increase of surface temperature decreases. Thus, the char oxidation occurs at later times for the same incident heat flux.

2. The presence of moisture in the wood matrix delays the decomposition process and dilutes the decomposition products.

3. The mass flux at the time of ignition increases as the moisture content increases. This is because the initial mass evolution from the sample is essentially desorbed water, and this component evolves at higher rates as the moisture content increases. An absolute minimum mass flux of about  $0.22 \text{ mg/cm}^2$ .s is necessary for piloted ignition to occur.

4. The presence of moisture in the wood matrix increases the total energy needed to ignite the sample by increasing the ignition time. It also increases the critical incident heat flux by increasing the heat loss from the sample surface.

5. A single correlation was derived for all ignition data. This correlation accounts for the moisture dependent thermal properties and the heat loss from the sample.

6. The surface temperature at ignition increases as moisture content increases. This is due to slow decomposition of the solid at the surface and the resulting buildup of the low thermal conductivity char layer prior to ignition.

### 6.3 Recommendations for Future Work

Based on this research, it seems that the following areas need further investigation in the future.

A study of transient cooling of the porous solid by droplet
evaporation of fluids other than pure water. This will hopefully yield
.
the optimum cooling agent.

2. A study of the effect of droplet release height on the cooling process will yield the optimum strategy for delivering the coolant agent.

3. A study of the droplet evaporation of common fuels such as methanol and benzene at higher temperatures on solids with different porosity. This may have direct application in the design of combustion chambers for internal combustion engines.

4. Investigation of the effect of oxygen concentration on the ignition process. This will allow determination the minimum oxygen concentration necessary for ignition.

5. Investigation of the effect of air velocity on the ignition process. Air velocity may significantly affect the ignition process, for ignition to occur, the mixture of the products of decomposition generated by the solid and the surrounding air should be within flammability limits.

6. Develop a theoretical model for thermal decomposition of wood and compare with the present experimental data.

APPENDIX A

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#### APPENDIX A

### METHOD OF ESTIMATING LOCATIONS OF IN-DEPTH THERMOCOUPLES AND THERMOPHYSICAL PROPERTIES OF THE CERAMIC

In this appendix, the procedures used to determine the location of in-depth thermocouples in the ceramic blocks and the thermophysical properties of the ceramic are described. Some of the properties were supplied from the Sandia National Laboratories [Taylor and Groot (1985)]. However, since the properties of castable materials change according to the procedure used in casting and drying them, it was necessary to re-evaluate these properties. A program provided by Professor J.V. Beck (1977) was used to estimate the thermal conductivity and the density-specific heat product using transient temperature and heat flux measurements.

### A.1 In-depth Thermocouples Locations

The locations of thermocouples, prior to casting, were measured to within  $\pm$  0.1 mm. Due to the uncertainties introduced during the casting process and shrinkage, thermocouple locations were evaluated by a least square fit to the measured steady state temperatures. This criterion yields a unique "best fit" line for the given set of data. Figure 1A shows the least square fit to the steady state temperatures. The evaluated thermocouple locations from the least square fit were close

to the measured values before casting. Table 1A shows the comparison of the two values. Attempts to determine thermocouple locations radiographically by using X-rays were not successful.

### A.2 Porosity

Porosity is the fractional void volume of the ceramic. A simple and straightforward method was used to determine the porosity. Several samples were dried by a standard procedure in a temperature-controlled chamber at 105  $^{\circ}$ C until constant weights were achieved. The samples were then rinsed in distilled water until all the samples attained saturated weights. Since there was no change in dimensions of the dry and the wet samples (i.e, no water adsorption), the difference between the weights of the saturated and dry samples was used to determine the amount of water displaced. Then the volume fraction of the cell-wall substance was calculated. The value of the porosity tabulated in Table 2A is an average of four tests.

### A.3 Thermophysical Properties

The thermal conductivity, the product of density and specific heat were estimated by a technique used by Professor J.V. Beck. Two thin electrical circular heaters 3" in diameter were placed between two identical ceramic solid-cylinders. Both solid cylinders were also 3" in diameter. One of them was instrumented by the thermocouples. Two

aluminum solids were placed at both ends in order to obtain constant temperature boundary conditions.

The solids were allowed to heat up until a steady state condition was achieved and then an electrical step power input was applied to the heaters. Finally, the power was turned off. During the experiment temperature data was collected. The temperature history of the thermocouples in a typical test is shown in Figure 2A. The time for the step power input and the time when power was turned off are also shown. Voltage and current inputs were also measured.

Program PROP, provided by Professor J.V. Beck, was used to estimate the thermal conductivity and the density-specific heat product. This finite difference program solves a transient one-dimensional homogenous partial differential equation with temperature-dependent properties. The two boundary conditions are the heat flux at the upper surface and the constant temperature at the lower surface of the solid. The heat flux was calculated from the voltage and current measurements and from the area of the heaters.

Results of the thermophysical properties at four different temperatures are tabulated in Table 2A. From these results it may be concluded that the thermophysical properties of the ceramic are not strongly temperature-dependent. Table 2A also contains the constituents of the refractory ceramic.

# TABLE 1A

## In-depth Thermocouples Locations

| prior to casting (mm) |   | Least Square Fit (mm |  |  |
|-----------------------|---|----------------------|--|--|
| 0.0                   | I | 0.0                  |  |  |
| 1.3                   | I | 1.69                 |  |  |
| 2.9                   | 1 | 3.06                 |  |  |
| 5.5                   | 1 | 6.63                 |  |  |
| 6.3                   | I | 7.33                 |  |  |
| 10.5                  | 1 | 12.27                |  |  |
| 26.0                  | 1 | 26.56                |  |  |

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### TABLE 2A

## Magnesium Oxide Ceramic-Refractory Grade

# (i) Constituents or chemical analysis by weight

| Compound                       |   | Composition |  |  |
|--------------------------------|---|-------------|--|--|
| Mg O                           | 1 | 96.11%      |  |  |
| si o <sub>2</sub>              |   | 2.2%        |  |  |
| Fe <sub>2</sub> 0 <sub>3</sub> | 1 | 0.36%       |  |  |
| Al <sub>2</sub> O <sub>3</sub> |   | 0.19%       |  |  |
| Cr <sub>2</sub> 0 <sub>3</sub> |   | 0.06%       |  |  |
| Ca O                           | 1 | 1.08%       |  |  |

# (ii) Porosity 33.28%

# (iii) Thermophysical Properties

| Density               | Specific   | Thermal   | Thermal  |
|-----------------------|--|---|--|
|                       | Heat   | Conductivity  | Difussivity  |
| (gm/cm <sup>3</sup> ) | (J/gm.K)   | (W/cm.k)  | (cm <sup>2</sup> /sec.)  |
|                       |  |   |  |
| 2.053                 | 1.00   | 0.02283   | 0.0111   |
| 2.053                 | 1.076  | 0.02261   | 0.0102   |
| 2.053                 | 1.307  | 0.02225   | 0.0083   |
| 2.053                 | 1.307  | 0.02225   | 0.0083   |
|                       | Density<br>(gm/cm <sup>3</sup> )<br>2.053<br>2.053<br>2.053<br>2.053 | Density     Specific       Heat     Heat       (gm/cm <sup>3</sup> )     (J/gm .K)       2.053     1.00       2.053     1.076       2.053     1.307       2.053     1.307 | Density     Specific     Thermal       Heat     Conductivity       (gm/cm <sup>3</sup> )     (J/gm .K)     (W/cm.k)       2.053     1.00     0.02283       2.053     1.076     0.02261       2.053     1.307     0.02225       2.053     1.307     0.02225 |







APPENDIX B

### APPENDIX B

### CRITICAL NOZZLES

This Appendix presents a description and the calibration procedure of the critical nozzles. These nozzles were used because they are a very accurate way of controlling and measuring mass flow rates of gases over a wide range of flow rates. Critical nozzles offer many advantages over other measuring devices; they do not have any moving parts which produce friction or wear [Aschenbrenner (1983)]; no pressure differential measurement is needed [Arnberg (1962)); simple construction, and simple flow equation that depends on pressure and temperature of upstream.

The configuration of the nozzles was adapted from [Arnberg [1962]]. The general design is shown in Figure 1B. A set of nozzles with different diameters were manufactured to deliver a flowrate of air anywhere in the range (175-32,000 ft<sup>3</sup>/hr). An identical set was also manufactured to be used for either  $O_2$  or  $N_2$ . A straight section of pipe approximately 120" was placed in front of the nozzle in order to achieve fully developed conditions at that location.

### **B.1 Mass Flowrate**

The theoretical mass flowrate of a given nozzle is directly proportional to upstream pressure and temperature. The static state is

given in ASME (1959)]. [Holman (1984)] presented the equation in the following form:

$$\dot{m} = A_{\star} P_{1} \sqrt{\frac{2 g_{c}}{R T_{1}}} \left[ \frac{\gamma}{\gamma + 1} \left( \frac{2}{(\gamma + 1)} \right)^{\frac{2}{\gamma - 1}} \right]^{\frac{1}{2}}, \quad (B.1)$$

where  $\dot{m}$  - mass flowrate (lbm/sec),

 $A_{\star} = \text{area of the nozzle (in.}^{2}),$   $P_{1} = \text{inlet static pressure (Psia)},$   $g_{c} = \text{gravitational conversion factor (32.2 lbm ft/lb sec}^{2}),$  R = gas constant (ft lbf/lbm R),  $T_{1} = \text{inlet temperature (°R)},$   $\gamma = \text{ratio of specific heats of the gas (C_{p}/C_{v}).}$ 

### **B.2 Calibration**

A trace gas technique was used for calibration of the nozzles. A known flowrate of methane (measured by a flowmeter) was introduced downstream of the critical nozzle. A sample of this mixture passed through the gas analyzer, where the percentage of methane was recorded by the gas chromatograph. The experiment was recorded by the gas chromatograph. The experiment was repeated several times for different upstream pressures at the known fixed amount of methane. The experimental gas flowrate was calculated by writing a mass balance of methane.

mole fraction of methane - 
$$\frac{\text{flowrate of methane}}{\text{total flowrate of gases}}$$
 (B.2)

The mole fraction of methane was determined from the chromatograph reading (A), the flowrate of methane is the measured value of the flowmeter  $(Q_1)$ , the total flowrate is the flowrate of air and methane  $(Q_a + Q_1)$ . Thus the flowrate of air  $(Q_a)$  is given by

$$Q_a = \frac{Q_1 - Q_1 A}{A}$$
 (B.3)

The experimental and the theoretical flowrates are compared in Figure 2B. A discharge coefficient of 0.964 is listed in the literature for a similar critical nozzle design. The discharge coefficient is the ratio of the experimental to the theoretical value. Figure 2B shows an error of ~ 4%. The discharge coefficient used here is 0.97. The error is probably due to the combined error in the measurements of the nozzle diameter, flowrate of methane, and the reading of the chromatograph.



Figure 1B Configuration of the Critical Nozzles.

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APPENDIX C

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### APPENDIX C

#### THE GAS ANALYSIS SYSTEM

This Appendix describes the gas analysis system. This system is mainly composed of the individual instruments used to make continuous measurements of  $H_2O$ ,  $CO_2$ , CO,  $O_2$ , and total hydrocarbons (THC) in a sample stream. A schematic of the instruments along with the accessories needed to feed the desired sample stream into each analyzer is shown in Figure 2.4.

The gas sample was pumped by a metal bellows pump model MB 601 HT driven by 0.75 HP motor. This pump was originally designed as a two stage pump. The two stages were used as separate pumps; one side was used as a suction pump for  $H_2O$  and  $CO_2$  analyzers, the other side was used to pump through the rest of the analyzers. A pressure regulator originally built in the THC analyzer was used to regulate the pressure before the CO and the  $O_2$  analyzers. A heating tape was used to heat the sample line in order to prevent condensation of HC and  $H_2O$ . Needle valves were used to control the flow through each analyzer which was monitored by rotameters. The sample gas supplied to the CO analyzer was dried by cooling it to -5°C, using a Neslab U-cool immersion cooler as required by the instrument. The sample lines were constructed from teflon tubing and stainless steel swagelock fittings.

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C.1 H<sub>2</sub>O - CO<sub>2</sub> Analyzer

An infrared AR-600 nondispersive infrared dual gas, dual range analyzer was used for  $H_20$  (Full scale 0 - 5.0%) and  $CO_2$  (full scale 0 -1.0%) analysis. The analyzer has a 100 ms response time and an accuracy of ± 1% of full scale (F.S.). The sample gas is drawn through a 0.5 micron inline filter, and into the analyzer by pump suction. This arrangement yields the fastest response time.

This analyzer has certain unique features: (1) the measurements are not affected by the presence of HC in the sample gas, (2) it is equipped with a sophisticated differential signal processing techniques which eliminate zero drifts, (3) it is instrumented with an interconnection network between the  $CO_2$  and  $H_2O$  measurements which provides a spectral balance adjustment for the spectral interference range of the two gases, and finally (4) the analyzer is equipped with an internal calibration reference for setting the span control whenever a calibration gas is not available.

# C.2 CO Analyzer

A Beckman 315B infrared analyzer was used to continuously analyze the concentration of CO in the sample gas. The analysis is based on a differential measurement of the absorption of infrared energy. The analyzer has an adjustable full scale sensitivity, 90% response time in 0.5 second at 400 cc/min, and an accuracy of  $\pm$  1% of full scale.

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C.3 0, Analyzer

A Beckman 778, a polaragraphic analyzer was used for  $0_2$  analysis. The concentration of  $0_2$  is determined by measuring the partial pressure of oxygen. Because of this characteristic, it is important that the sample gas mixture be kept under the same total pressure as when the instrument was calibrated. The analyzer has three measuring ranges (0-5, 0-25, 0-100%), 90% response in 10 seconds, and an accuracy of  $\pm$  1% F.S.

# C.4 Total Hydrocarbon (THC) Analyzer

A Beckman 400 was used for analysis of non-condensible hydrocarbons. The analyzer utilizes the flame ionization method. The sensor is a burner; a regulated flow of sample gas passes through a flame sustained by regulated flows of a fuel gas (40%  $H_2 - 60 H_e$ ) and air. The concentration of total hydrocarbons in the original sample is essentially measured by determining the rate at which carbon atoms enter the burner.

The analyzer has an adjustable range from 1 ppm  $CH_4$  to 2%  $CH_4$ , response time for 90%  $CH_4$  of 0.5 second with sample by-pass flow of 3 liters/minute, and an accuracy of  $\pm$  1% F.S.

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