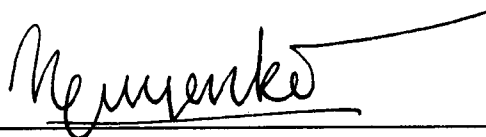


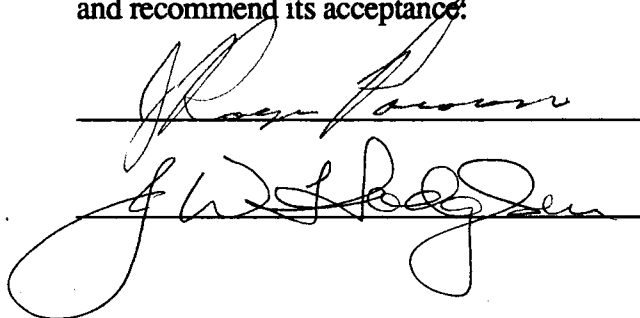
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**IGNITION AND COMBUSTION PHENOMENA
OF LASER-IGNITED BORON AGGLOMERATES AT
ELEVATED PRESSURES**

**A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville**

**Syed Wajahat H. Zaidi
December 1991.**

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ABSTRACT

Boron agglomerates having nominal initial diameter of 534 μm suspended at the tip of SiC fibers were ignited by a focused CO_2 laser in a quiescent O_2/N_2 (20/80) environment. A near-infrared two-color pyrometer and a broadband radiometer were used to measure the surface temperature and the radiation emitted by the burning agglomerates. From the temporal outputs of the pyrometer and radiometer, the ignition delay time, the burning time and the surface temperature of the boron agglomerates were obtained. High-speed cinematography (1000 f.p.s) was also used to observe the phenomenological events; i.e., the flame structure, and to correlate with the output obtained from the pyrometer and the radiometer.

In the range of pressures (1.7 to 20.4 atm) investigated in the present study, three distinct combustion regimes were observed. In regime I (1.7 to 5.1 atm) partial or incomplete burning of the boron agglomerates occurred. In regime II (6.8 to 13.6 atm) steady-state, full-fledged burning was observed, in which the burning time decreases with increasing pressure. In regime III (above 13.6 atm) violent combustion with frequent particle ejections was observed. The surface temperature of the burning boron agglomerates in regime I is consistently below the boiling temperature of boron oxide (2316 K). In regime II, the agglomerate surface temperature is in the vicinity of 2400 K, whereas, the surface temperature is above 2400 K in regime III. Due to very high surface temperatures attained, and corresponding fragmentation and shattering of boron agglomerates in regime III, it appears almost certain that the agglomerate melting does occur at higher pressures. Limited investigation of the effect of oxygen mole fraction on burning time showed that increasing the oxygen mole fraction has the same effect as that of increasing pressure; i.e., decrease burning time.

Qualitative studies of flame structure from high-speed cinematography showed that the flame surrounding the boron agglomerate at all pressures consists of two distinct zones: a luminous inner zone surrounded at its edge by a green envelope (BO_2). The size and luminosity of the inner zone increases with increasing pressure. The presence of boric oxide (BO_2) at all pressures investigated, imparts significance to its inclusion in the modeling of boron agglomerate combustion.

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CHAPTER 1

INTRODUCTION

Recently there has been a revival of interest in the utilization of slurry fuels for air-breathing propulsion systems. Slurry fuels consist of a mixture of finely milled micron size solid particles suspended in a conventional liquid fuel carrier. Slurry fuels offer advantages of liquid fuels (pumpability and injectability) and solid fuels (high volumetric energy density). Several metals such as aluminum, boron, magnesium etc., are possible candidates for the solid ingredient of slurry fuels. A useful figure of merit for use in weight or volume-limited air-breathing propulsion systems is the heat of combustion. Values of this parameter for several possible fuels are presented in Table 1.1.

As may be seen, the gravimetric heating value (for weight-limited applications), and volumetric heating value (for volume-limited applications) of boron are quite impressive. Thus the use of boron potentially offers many advantages, such as increased range and higher velocities .

Works on carbon-black slurries [1-3] have demonstrated that slurry droplets burn in a two stage process: In the first stage the liquid carrier of the slurry evaporates and burns, leaving behind an agglomerate formed from the solid particles in the slurry. In the second stage, this agglomerate then heats up, ignites and burns.

Since the liquid component of the slurry fuels burns rapidly in the vapor phase, it is the ignition and combustion of the agglomerates that determines the efficiency of the slurry fuels. In order to be a viable candidate for slurry fuel blend the agglomerates must ignite and burn to completion within the limited residence times (typically between 20 to 100 ms) in the combustor; otherwise, the presence of the partially burned agglomerates in the

TABLE 1.1 Heating Values Of Various Fuels [25].

Fuel	Gravimetric Heating value cal/g	Volumetric Heating value cal/cm ³
JP5	10,150	8,230
Shelldyne-H	9,860	10,000
Carbon	7,830	17,700
Aluminum	7,420	20,000
(CH ₂) _n	10,400	9,600
Boron	13,800	32,200
Magnesium	5,910	10,300

exhaust gas would have a detrimental effect on the performance of the air-breathing propulsion systems.

Difficulties encountered in burning highly-boron-loaded solid rocket propellants resulted in numerous studies aimed at ascertaining the ignition and combustion mechanisms of single boron particles. These include shock tube ignition studies [4-7], flat flame burner studies [8,9] plasma generator product stream studies [10] and laser ignition studies [11-13].

These studies have shed considerable light on the ignition and combustion phenomena of single boron particles, and a reasonably accurate picture of the overall processes appears to have evolved. Flat-flame burner studies of Macek [8,9] have shown that burning of the single boron particles takes place in two stages: The first stage being an ignition stage, during which the boron oxide coating is removed from the particle surface. The ignition stage is then followed by a second stage, in which the cleansed boron particle burns vigorously. It seems likely, on the basis of experimental observation and thermodynamic considerations that this second stage (referred to subsequently as the full-fledged combustion stage) is a multistep process with the major heat release occurring in a vapor phase reaction zone some distance removed from the particle surface, where a suboxide produced at the surface is oxidized to final product. This full-fledged combustion stage appears to be diffusion-controlled for large particles, but kinetics controlled for small particles, with transition in the general region of 15-30 μm diameter, the exact transition point depending upon pressure.

Although considerable attention has been devoted to study the burning of single boron particles, the ignition and combustion phenomenon of boron agglomerates have been addressed only recently. Shevchuk et al. [14] and Holl et al. [15] have conducted both

experimental and theoretical investigations. A detailed review of these studies is presented in chapter 2.

A subject of major importance, which to date has received practically little attention, is that of the ignition and combustion of boron agglomerates at elevated pressures. Very high operating pressures of air-breathing combustors using slurry fuels (1-20 atm), impart a practical importance to this subject. Consequently the objectives of the present investigation are :

1. To study the effects of pressure on the ignition delay time, the burning time and the surface temperature of boron agglomerates.
2. To study the flame structure during ignition and combustion via high speed cinematography.

In the present study, probe-supported boron agglomerates were mounted in a high-pressure combustion chamber and ignited by a CO₂ laser beam. The use of laser heating and high-pressure combustion chamber in the present study offers a number of advantages that were not present in the past investigations. For example, placing of the boron agglomerate in the combustion chamber would allow variation of gas composition and pressure. In addition laser heating technique would suppress the long ignition delay time. Since the air-breathing propulsion systems use atmospheric air, a O₂/N₂ (20/80) mixture was used to study the effects of pressure. Pressure was varied from 1.7 to 20.4 atm. Although beyond the scope of this study, a number of experiments were conducted at higher oxygen mole fraction to determine the effect of oxygen concentration on the burning times.

In contrast to previous studies in which the ignition temperature of boron agglomerate was deduced from the gas temperature, a fast-response, near-infrared two-

color pyrometer was used in this study to measure the surface temperature of the agglomerate throughout the ignition and combustion events. In conjunction with the two-color pyrometer, a broadband radiometer was also used to record the radiation emitted by the laser-ignited agglomerate for determining the burning time. Flame structure of the agglomerate during ignition and combustion was studied by employing a high speed motion picture camera. In this study ignition is defined as the instance at which the flame first appears, as evidenced from the film records. Accordingly, the time elapsed between the beginning of laser irradiation and the first appearance of the flame is the ignition delay time. Burning time is the time elapsed between the ignition point and the end of combustion.

A literature survey of previous studies in this area is given in chapter 2. Detailed discussion of the experimental apparatus and procedure is presented in chapter 3. Chapter 4 comprises of the results of this investigation. Finally conclusion and suggestions for future studies are outlined in chapter 5.

CHAPTER 2

LITERATURE SEARCH

The following chapter presents a review of past investigations of the ignition and combustion of single boron particles, and boron slurry agglomerates. In section 2.1 studies on the ignition and combustion of single boron particles are reviewed, whereas studies on boron agglomerates are reviewed in section 2.2.

2.1 IGNITION AND COMBUSTION OF SINGLE BORON PARTICLES .

Macek and Semple [8], conducted the first investigation to study the ignition and combustion phenomena of single boron particles. Particles ranging in diameter from 35-45 μm , were injected into a hot oxidizing gas stream. The gas stream was generated by the combustion of carbon-monoxide (CO) or propane (C_3H_8) in oxygen. Nitrogen was used as a diluent. They determined the ignition times and burning times of the particles by using time-elapsed photographs. They also determined the ignition limit of the boron particles, by lowering the hot gases temperature until the particles ceased to ignite.

The ignition temperature (minimum ambient temperature required for particle ignition) was found to be in the range of 1850-2000 K. Photographs showed that the flame structure of the burning particles consisted of a luminous front near the surface, surrounded by a 300-400 μm wide symmetric, but less luminous zone. They also observed the presence of up to 1 cm broad green envelope surrounding the less luminous zone. This green envelope was found to become brighter at higher gas temperatures. However, Macek and Semple [8] did not attempt to determine the nature of this green envelope.

The combustion of boron particles took place in two distinct stages, with a dark period in between. Second stage was the longer and the brighter of the two. The dark period was found to be a function of oxidant mole fraction. At higher oxidant mole fractions this dark period almost vanished, whereas at lower oxidant mole fractions it became prominent. They explained the two stages in the following manner: as the particles are first exposed to the gases, the boron oxide (B_2O_3) layer, which hinders ignition, melts and allows oxidant diffusion through the layer, increasing the reaction rate. Simultaneously, the oxide layer grows and there is a decayed reaction rate, causing the dark period. The temperature of the boron particle continues to increase and at 2316 K (boiling temperature of B_2O_3), the oxide layer vaporizes, allowing a rapid diffusion of oxidants to the particle surface and thus an increased reaction rate. The second stage of combustion was observed to be controlled by the gas-phase oxidant diffusion. Water vapor was found to promote ignition (i.e. lower ignition temperature) and enhance burning rate. An increase in the oxygen mole fraction was also found to increase the burning rate.

In second paper Macek and Semple [11], investigated ignition and combustion of single boron particles at pressures between 1 and 35 atm. Crystalline boron particles with a nominal diameter of 75 μm were ignited by dropping the particles through a focused CO_2 laser beam, in various oxidizing atmospheres; i.e., O_2 , O_2/Ar , air and CO_2 . Ignition and burning times were determined by using time-elapsd photography.

Their results show that in air and in O_2/Ar mixture, the burning time decreases with increasing pressure. The burning time obtained in pure oxygen at atmospheric pressure is much less than that in other oxidizing atmospheres. In pure CO_2 and in $O_2/N_2(7/93)$ mixture there was no induction period, no self ignition, and no steady-state combustion; particles must be brought to a burning regime by an external energy flux, and they were

able to maintain that regime for only a limited time before extinguishment. These results are listed in Table 2.1.

They also performed a theoretical study and showed that the classical theory of ignition and combustion accounts for all three burning modes observed in their study: metastable surface reaction during the pre-ignition period, rapid self-sustained diffusion-controlled combustion, and decaying combustion.

Mohan and Williams [12] experimentally and theoretically investigated the ignition and combustion of laser-ignited boron particles. Experimental studies involved granular and amorphous boron particles ranging from 50 -150 μm in diameter. The boron particles were held at the tip of 10 μm glass fibres in a chamber of various O_2/Ar compositions and ignited by a neodymium-doped glass laser. A high-speed motion picture camera (5000 fps) was employed to record the event.

Ignition of "granular" particles was found to be difficult at atmospheric pressure. During ignition, separation of fiber and particle spin after detachment was observed. They attributed this spin to one-sided burning. They suggested that boron melting occurs but did not provide any conclusive evidence. On the other hand, "amorphous" boron particles ignited and burned easily. A two-stage burning was observed even though the burning time was reduced considerably.

They also formulated two separate models: one for the ignition stage and one for the combustion stage. From the models they obtained ignition delay time and burning time as functions of oxide layer thickness, oxygen concentration and ambient temperature, etc. The agreement between the theoretically predicted values and experimental results was good.

In another paper Macek [13] presented a theoretical and experimental study of single boron particle ignition and combustion. Particles ranging in diameters from 37 to

TABLE 2.1 Burning Times of Boron Particles [11].

Pressure (atm)	No. of Particles	t_i (msec)	t_b (msec)
(a) Combustion in O ₂ /N ₂ (20/80)			
1.0	19	7.3±0.9	41.3± 2.7
7.8	23	8.4±0.9	34.3±2.6
21.4	28	24	28.5±2.5
35.0	22	70	20.5±1.7
(b) Combustion in Air			
1.0	33	5.0±1.7	50.0±5.6
7.8	29	5.3±1.0	40.8±4.1
21.4	16	26	39.6±5.8
35.0	24	71	21.6±2.2
(c) Combustion in Oxygen.			
1.0	40	1.8±0.2	0.8±0.5

124 μm were ignited by a focused CO_2 laser beam. Ignition delay and burning times were determined as functions of particle diameter, pressure and oxygen mole fractions.

He observed that at high oxygen mole fraction and pressure, the pre-ignition stage was relatively dim, whereas combustion stage was relatively bright. The two stages became indistinguishable, as the pressure and O_2 mole fraction decreased. Table 2.2 shows the effect of particle diameter and pressure on burning times of single boron particles. At sufficiently large values of oxygen mole fraction and pressure, the burning time was found to be proportional to the square of the particle diameter ; i.e., d^2 law.

Experimental results were also compared to the theoretically predicted values of Mohan and Williams [12] model at 1 atm. For large values of initial particle diameter, a close agreement was obtained, however, results deviated for smaller diameter. Furthermore, theory predicted a much greater decrease in burning time with increased oxygen mole fraction than observed from the experiment.

King [16] formulated a mathematical model for the ignition of single boron particles in hot oxidizing gas streams. This model was used to predict ignition times observed by Macek and Semple [8] in flat-flame burner single boron particle ignition studies, and to predict conditions required for ignition of boron particles in air-augmented rocket afterburners.

In this model, he assumed that oxide production is controlled by diffusion of oxygen through the liquid oxide film and derived a two-constant expression for the oxygen delivery rate to the boron core. He also assumed that the removal of oxide from the surface was evaporation-rate-controlled, with an activation energy equal to the oxide heat of evaporation. Three non-linear unsteady-state differential equations in boron mass, oxide mass, and temperature resulting from application of mass and enthalpy balances were integrated numerically for given boundary conditions and initial conditions to determine

TABLE 2.2 Effect of Particle Diameter and Pressure on Burning Times [13].

Pressure, atm	Burning Times, ms			
	Diameter, μm			
	37	54	98	124
0.50	-	-	-	139 $\pm$ 5
1.0	17.9 $\pm$ 1.5	27 $\pm$ 2	75 $\pm$ 3	121 $\pm$ 5
4.4	-	23 $\pm$ 2	-	-
7.8	11.1 $\pm$ 0.7	21 $\pm$ 1.5	64 $\pm$ 3	106 $\pm$ 4
14.6	-	-	56 $\pm$ 3	*
21.4	8.7 $\pm$ 0.9	-	-	*
28.3	6.1 $\pm$ 0.7	-	43 $\pm$ 3	*
35.0	7.7 $\pm$ 1.0	-	40 $\pm$ 10	*

* No Ignition

- No data

whether or not a given particle would ignite and, if so, how long the ignition delay time would be.

Ignition was found to be directly dependent upon the ambient temperature. For example at an ambient temperature of 2250 K and an effective radiation temperature of 3250 K, the particles ignited successfully as shown in Figure 2.1. Figure 2.2 shows the effect of lowering the ambient temperature to 2125 K. At this ambient temperature there was no ignition since oxide production rate exceeded oxide evaporation rate. A comparison between the predicted values of this model and experimental results of Macek and Semple [8] is shown in Figure 2.3. The degree of agreement is encouraging, although Macek's data indicates less dependence on ambient temperature and slightly more dependence on particle size than that predicted.

In another model King [17] revised the original model [16] and made the following three changes: First, the heat of fusion of boron, which must be supplied when the particle reaches the melting point of 2450 K was included in the modified treatment of the enthalpy balance. Second, a dimensionless group, $k/\alpha n$, was used to determine whether the liquid boric oxide removal rate was kinetics-controlled or diffusion-controlled. Finally, the effect of water vapor on the removal of the oxide layer was considered.

A major assumption in the development of these two models was that of an isothermal particle. This assumption is valid for flat-flame burner studies only because the rate of change of surface temperature in that case is small, but this assumption is not valid for laser-ignition studies, like the one described herein, because in the later case very high rates of change of surface temperature are encountered and particle is essentially non-isothermal.

Results from the second model indicated that for ambient surrounding temperatures of about 2100 K, ignition and self-sustained combustion occur readily. For temperatures

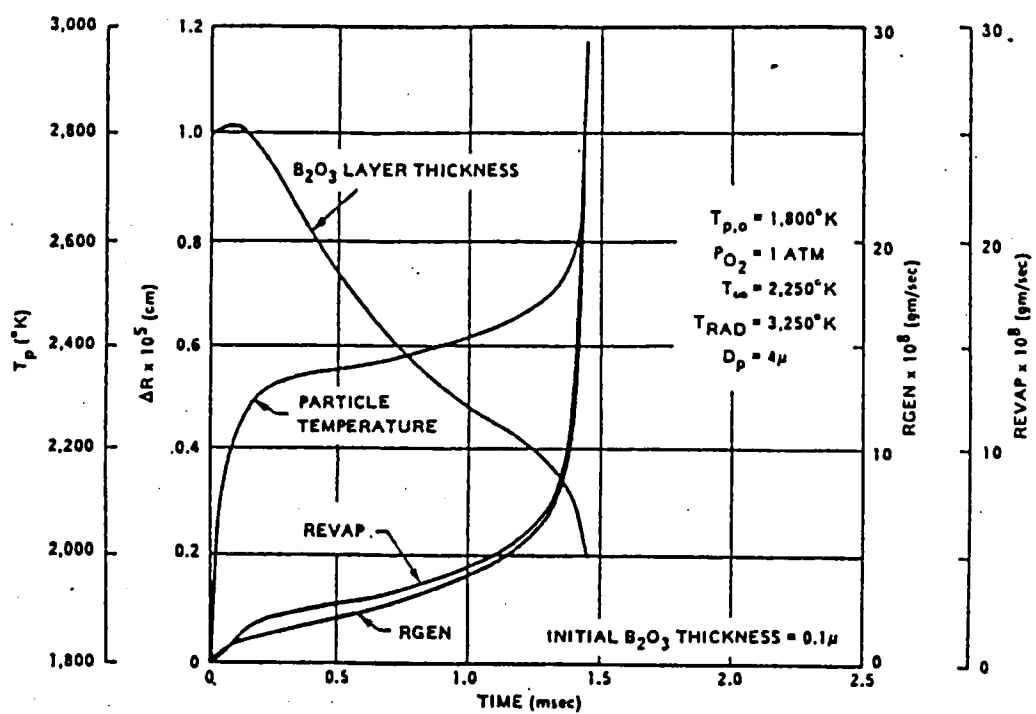


FIGURE 2.1 Predicted Time Dependence of Important Variables for Particle Which Ignites [16].

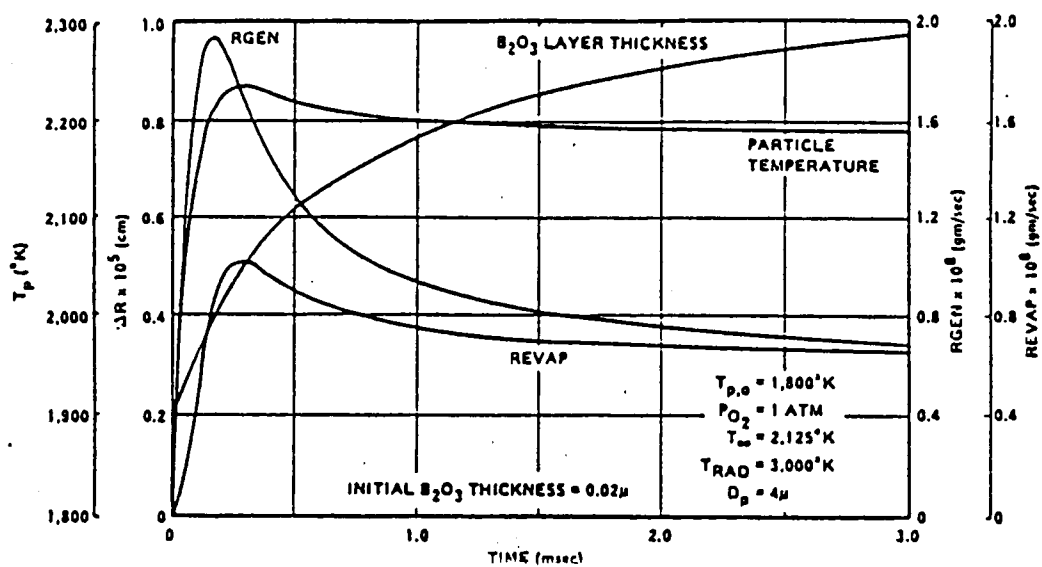


FIGURE 2.2 Predicted Time Dependence of Important Variables for Particle With No Ignition [16].

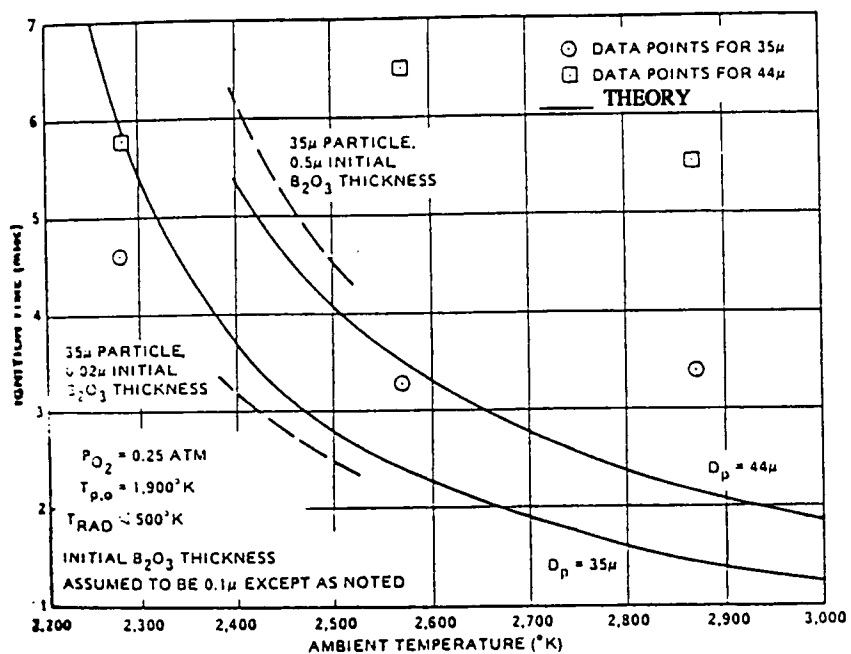


FIGURE 2.3 Comparison Between Predicted and Experimental Ignition Times As a Function of Particle Diameter [16].

of 2000 K, there is a "degenerate" ignition whereby quasi-steady-state is obtained, but there is no actual combustion. For temperatures of 1900 K, there was no ignition. A comparison between the predicted ignition times and experimental data of Macek and Semple [8] is presented in Figure 2.4.

Glassman et al [18] presented a physical and chemical interpretation of boron particle combustion. They re-investigated the ignition and burning processes of boron particles by dividing the overall combustion process into ignition phase and a combustion phase and then studying both of these phases separately.

A fundamental assumption made by nearly all previous investigators was that the rate of oxide production is controlled by the diffusion of oxygen from the gas, across the oxide coat to the boron-boron oxide interface where oxide producing reaction occurs [12]. Glassman et al suggested that boron diffusion across the oxide coat to the boron oxide-gas interface, with subsequent reaction, is more probable. They supported this claim by calculating the solubility of oxygen versus that of boron in boron oxide, rates of solution into and diffusion through the oxide by boron and oxygen, and a comparison of the characteristic boron diffusion time to the characteristic oxide vaporization time.

Although actual solubilities were unknown, their results, based on relative solubilities, indicated that boron is 10 times more soluble than oxygen in boron oxide. It was also shown that the rate of solution of both species were not rate limiting and the use of the solubilities as the initial concentrations for diffusion was justified. Lastly the comparison of characteristic diffusion times showed that the diffusion of oxygen through the gas to the particle is fast and is not likely to control oxide production.

In combustion phase, they studied both the chemical kinetics related factors and the mass diffusion (of oxygen) factors. Following the flammability limits definition of Mohan and Williams [12], they proposed that as this limit is approached, the combustion begins to

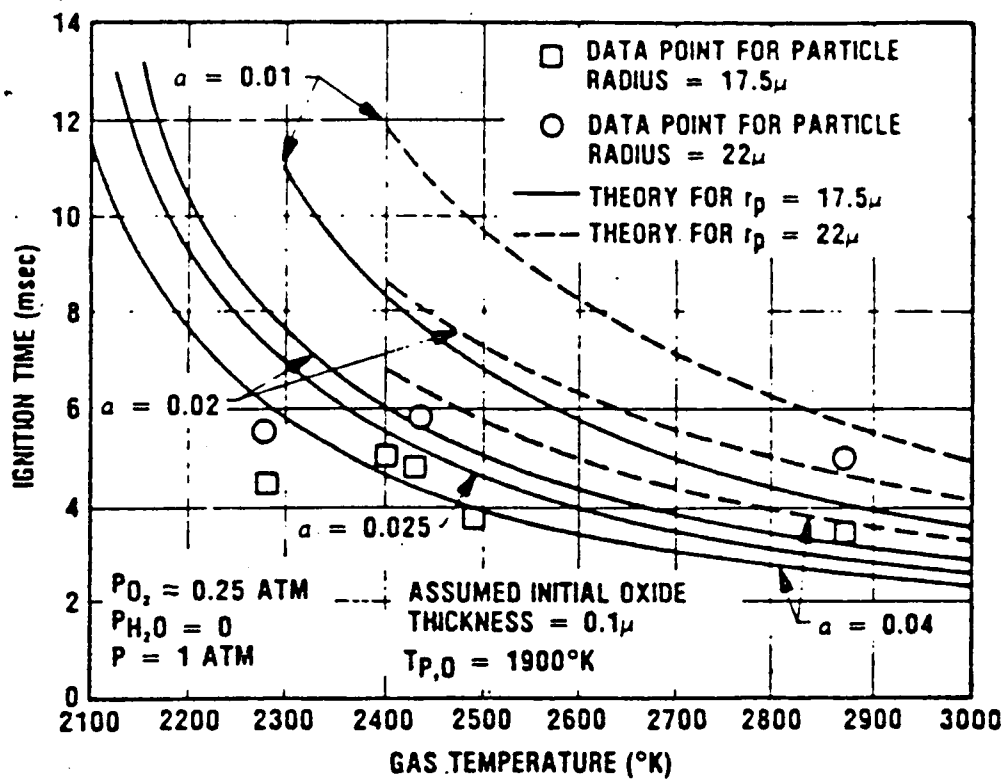


FIGURE 2.4 Comparison Between Predicted and Experimental Ignition Times As a Function of Particle Diameter [17].

occur in a regime of chemical kinetics control. In the chemical kinetics control region they proposed a mechanism for the consumption of boron into its products. The proposed mechanism was substantiated by reviewing the structure and heats of formations of the various gas phase molecules that could possibly be formed in a B,O,H system.

Makino and Law [19] performed a theoretical study of the combustion of an uncoated boron particle in an oxidizing environment. In their study they considered finite rate kinetics of both surface and gas-phase reactions. The study was conducted via a model they had previously formulated for carbon combustion [20], by adapting to the stoichiometry and thermo-kinetic parameters of the boron oxidation system, and by recognizing the restriction imposed by the possible surface condensation of boron oxide.

Chemical reactions considered included the surface $B-O_2$ and $B-B_2O_3$ reactions and the gas-phase $BO-O_2$ reaction. Generalized coupling functions were identified and combustion limits based on a feasibility regime for combustion were derived. The three limiting solutions respectively describe frozen, detached flame-sheet, and attached flame-sheet combustion. Frozen combustion occurs when the effect of the surface reaction is practically negligible. When the gas-phase reaction is infinitely fast, a "flame-sheet" limit is achieved, and a "flame-attachment" limit is reached when particle combustion rate decreases below a certain level. If two of the three gas-phase reactions occur infinitely fast, the classical diffusion-limited behavior is attained.

For various limiting solutions they also presented the feasibility regimes and burning rate as a function of particle temperature (see Figure 2.5), and the feasibility regime and particle temperature as a function of the ambient temperature (shown in Figure 2.6). The model predicts that as particle radius and pressure increase, the feasibility regime shifts to a lower ambient temperature, because of the increase in surface reactivity. Burning time was found to be linearly proportional to the square of the particle diameter, when particle

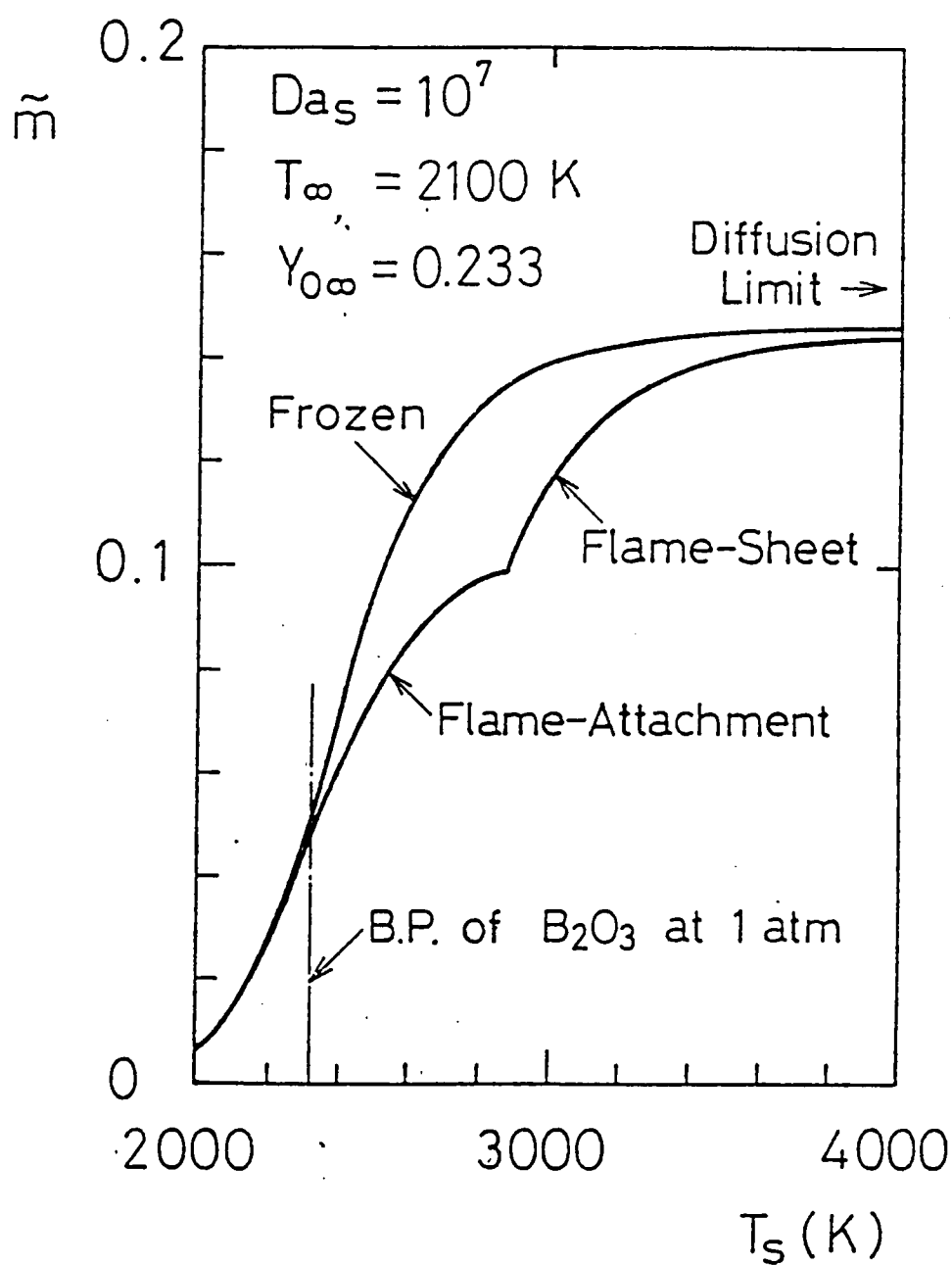


FIGURE 2.5 Feasibility Regime and The Burning Rate As a Function of The Particle Temperature For The Various Limiting Behaviors[19].

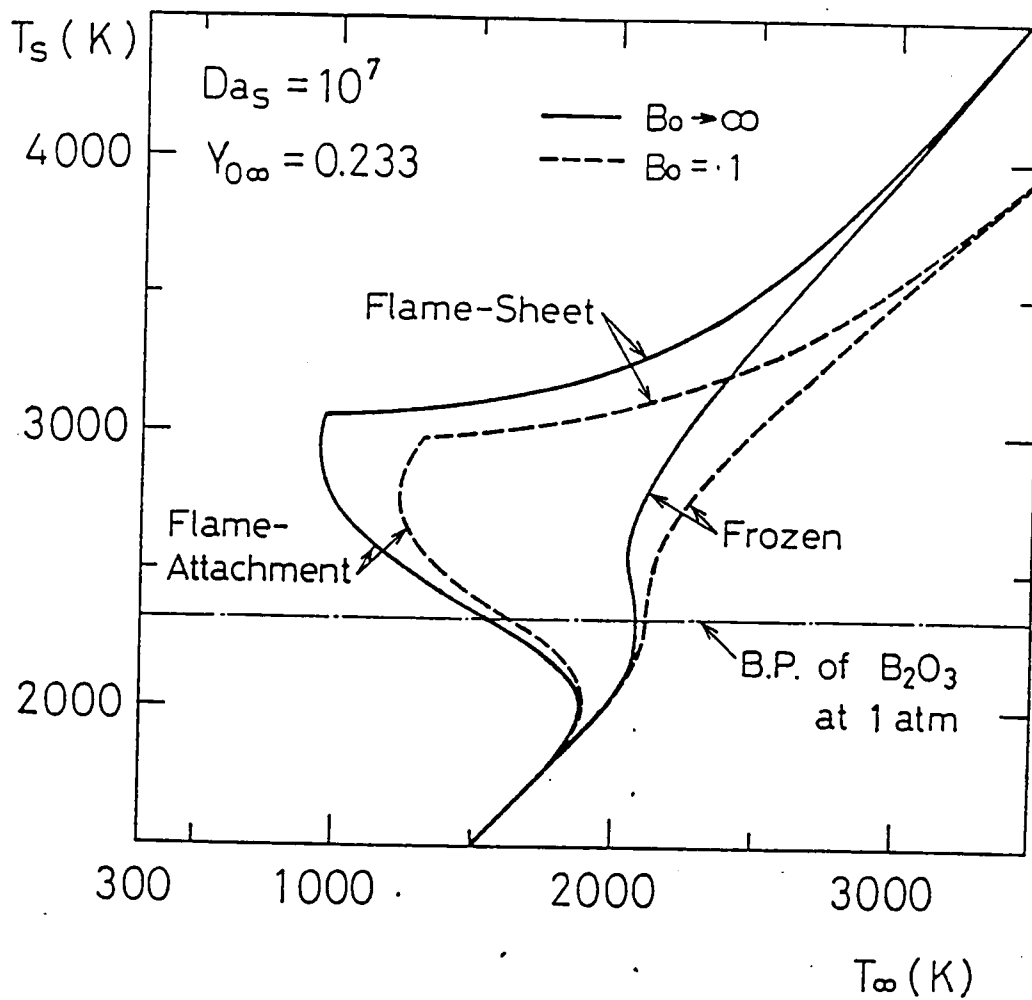


FIGURE 2.6 Feasibility Regime and The Particle Temperature As a Function of The Ambient Temperature for The Various Limiting Behaviors [19].

diameter was large. In other words d^2 law holds for larger size particles. The results of the theoretical study were found to be in good agreement with the experimental results of Macek [8].

Li et al.,[21] studied the kinetics of boron ignition and combustion by injecting fine (0.04-0.15 μm diameter) boron particles into the product gases of a flat-flame burner. The oxygen mole fraction in the product gases was varied from 0.08 to 0.80. Depending upon the flat-flame temperature three types of boron flame-plumes were observed. At flat-flame temperatures below 1800 K the plume was dark-yellow with dark-brown smoke emanating from its tip. At flat-flame temperatures between 1800 K and 1900 K, a yellow plume surrounded in its lower part by green emission was observed. Above 1900 K, however, the plume was bright-yellow surrounded entirely by bright-green emission. These three boron flame-plumes are shown in Figure 2.7. It is interesting to note that the flame structure at 1900 K was also observed by Macek and Semple [8]. Li et al., analyzed the spectra of three observed boron flame-plumes, and attributed the bright-yellow plume to boron ignition and the bright green to BO_2 emissions from boron combustion products. From height measurements of the yellow plume as a function of the flat-flame temperature, they extracted the overall rate parameters and interpreted these rate parameters in terms of existing and new potential models for the ignition and combustion of boron particles.

Takahashi et al.,[22] investigated the combustion behavior of free boron/JP-10 slurry droplets. Droplets of initial diameter of 0.3-0.5 mm and solid mass fraction of 0.05-0.3 were projected downward into high temperature post-combustion gases of ca.1900K and oxygen mole fractions of 0.07-0.39 under conditions of low Reynolds number (< 2).

Boron slurry droplets burned for short periods of time quiescently with an envelope diffusion flame of vaporized JP-10, but then experienced disruption. This disruption occurred in all cases, except for the droplets of low solid loading in the lowest oxygen

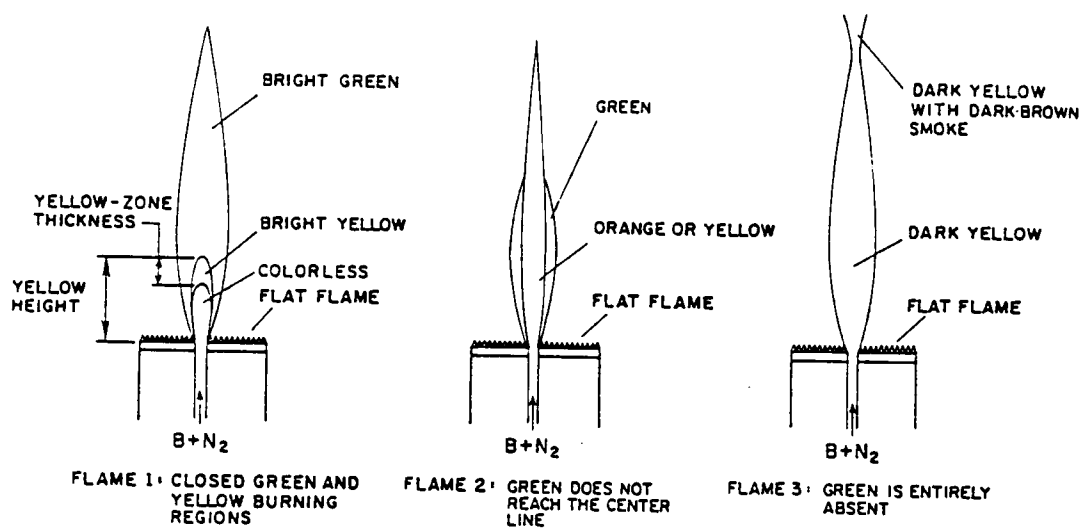


FIGURE 2.7 Schematic Illustration of The Appearance of The Three Different Types of Boron Flame Plumes [21].

environment. The disruption process was accompanied by a " popping " sound and a bright greenish flash of luminosity. They attributed the green color of the emission to the excited boric oxide (BO_2), and the audible noise to the rapid heat release associated with the ignition of the boron particles emitted from the primary slurry droplet. It was found that, as the oxygen content was increased for a fixed boron mass fraction, the disruption occurred at earlier times. On the other hand, as the boron loading was decreased, the time to disruption was found to increase significantly.

2.2 IGNITION AND COMBUSTION OF BORON AGGLOMERATES .

A literature search has produced only two papers dealing with the ignition and combustion phenomena of boron agglomerates. In the first paper, Shevchuk et al.[14] developed a theoretical model for predicting the critical ambient temperature required for ignition of boron agglomerates in a hot flowing oxidizing environment. Unfortunately, their model cannot be used to predict the ignition delay and the total combustion times. In their analysis they assumed that the evaporation of the boron oxide and the heat loss by radiation are negligible during the preignition period. In addition, they assumed that the reaction surface is equally accessible to the oxidant. This assumption implies that the concentration of the oxidant in the pores is the same as at the surface of the agglomerate. With these assumptions they obtained two governing equations for the time rates of change of oxide thickness and agglomerate temperature. From these two equations they determined the critical ambient temperatures or ignition temperatures as functions of agglomerate sizes, individual boron particle diameters, agglomerate densities, and oxygen partial pressures.

Results from their analysis indicated that the critical ambient temperatures which they referred to as the ignition temperatures decrease as the ratio of the reactive surface area within the agglomerate to the apparent agglomerate surface area increases. This surface area ratio can be increased by either using a larger agglomerate size or smaller boron particles within the agglomerate. The results predicted by their theoretical model are hardly surprising, since the larger the surface area ratio the larger the energy release due to chemical reactions and the smaller the heat loss due to convection, resulting in a lower ignition temperature. The ignition temperatures of agglomerates were also predicted to decrease continuously with increasing agglomerate density and oxygen partial pressure.

In conjunction with the theoretical model, Schevchuk et al. conducted experiments to investigate the effect of the aforementioned parameters on the ignition temperatures of the boron agglomerate. In their experiments, agglomerates of diameters between 250 and 2500 μm were formed by depositing boron particles onto tungsten wire supporting probe of 50 μm diameter. The probe-supported agglomerates were exposed to an electrically heated flowing oxidizing environment, with total pressure ranging from 0.2 to 1.0 atm. The temperature of the gas at the instance of ignition was referred to as the critical ambient temperature or also as the ignition temperature of the boron agglomerate. They found that the results obtained from the experiments agree quite well with those predicted from the theory. Ignition temperatures as low as 750 K were obtained, which are substantially below the ignition temperatures of single boron particles, ca. 2000 K.

In a very recent paper, Holl et al. [15] presented a more detailed investigation of the ignition and combustion phenomena of loosely bound, high porosity boron agglomerates. In their study, agglomerates having initial diameters of 175 to 800 μm diameter were formed by depositing boron particles on a quartz probe. Agglomerates of 175 μm diameter were used in the ignition and burning rate studies, whereas, larger agglomerates (300-800

μm) were used for surface morphology studies. The probe-supported agglomerates were exposed to the product gases of a flat-flame burner. Ignition times and burning rates of the agglomerates were determined as function of oxygen concentration and water vapor content by analyzing high-speed cinematography. All experiments were conducted at gas temperatures between 1690 and 1975 K and a total pressure of 1 atm. In their study they distinguished between two different ignition times. The first is called the first-stage ignition time which is the time elapsed between the first introduction of the agglomerate into the hot gas stream and the first appearance of agglomerate glowing. On the other hand, the second-stage ignition time is the total time elapsed between the first introduction of the agglomerate into the hot gas stream and the point at which rapid diameter decay begins to occur. Results from their studies showed that the first and second-stage ignition times decrease with increasing oxygen concentration. In addition the first-stage ignition times are slightly lower in dry-flame conditions than in wet flames, whereas the second-stage ignition times are not significantly affected by the ambient water vapor concentration. They also found that the burning rates of the boron agglomerates increase with increasing oxygen concentration, and the effect of the ambient water vapor is to increase the burning rate.

A d^2 -law was observed when the second-stage ignition times of the boron agglomerates obtained in their studies were compared to the ignition times of single particle obtained by Macek and Semple [8] under the same experimental conditions. They suggested that a relationship of this type indicates that the ignition of boron agglomerate is largely associated with agglomerate heating to a fixed ignition temperature. In other words, the heating process is the dominant phenomenon controlling ignition of boron agglomerates.

From the surface morphology studies of partially-reacted boron agglomerates and high-speed cinematography analysis they suggested that the ignition and combustion process of boron agglomerates consists of two distinct stages. The first stage is characterized by agglomerate glowing with negligible diameter decay. The time elapsed of this stage corresponds to the second-stage ignition delay time. The second stage or the full-fledge combustion stage is characterized by a brighter agglomerate glowing and rapid diameter decay. They suggested that during this stage the boron agglomerate is a molten liquid droplet since the melting temperature of boron has been reached, ca. 2450 K.

CHAPTER 3

EXPERIMENTAL APPARATUS AND PROCEDURE

A brief description of the experimental apparatus and procedure used in this study is outlined herein. An overview of the experimental method is presented in section 3.1. Test condition are presented in section 3.2. Section 3.3 addresses the production technique of boron agglomerates. Brief description of experimental apparatus is given in section 3.4, while sections 3.5 and 3.6 refer to the experimental procedure and methods of data analysis, respectively.

3.1 OVERVIEW OF EXPERIMENTAL METHOD

The ignition and combustion of boron agglomerates in quiescent oxygen and nitrogen environment was studied as a function of ambient pressure using the apparatus schematically shown in Figure 3.1. The boron agglomerate was mounted vertically in a combustion chamber, and ignited by a cw CO₂ laser beam.

The CO₂ laser beam is focused onto the front and back surfaces of the agglomerate by a focusing system consisting of mirrors (M), ZnSe beam splitter (F1), Ge beam splitter (F2) and plano-convex lenses (L1 and L2) of 250 mm effective focal length. The Ge beam splitter (F2) splits the CO₂ laser beam into two separate beams. The reflected beam (55%) travelling path A irradiates the front surface of the agglomerate, whereas transmitted beam (45%) irradiates the back surface, following path B. In such a manner the front and back surfaces of agglomerate are irradiated simultaneously, and thereby preventing the development of a preferentially localized ignition spot.

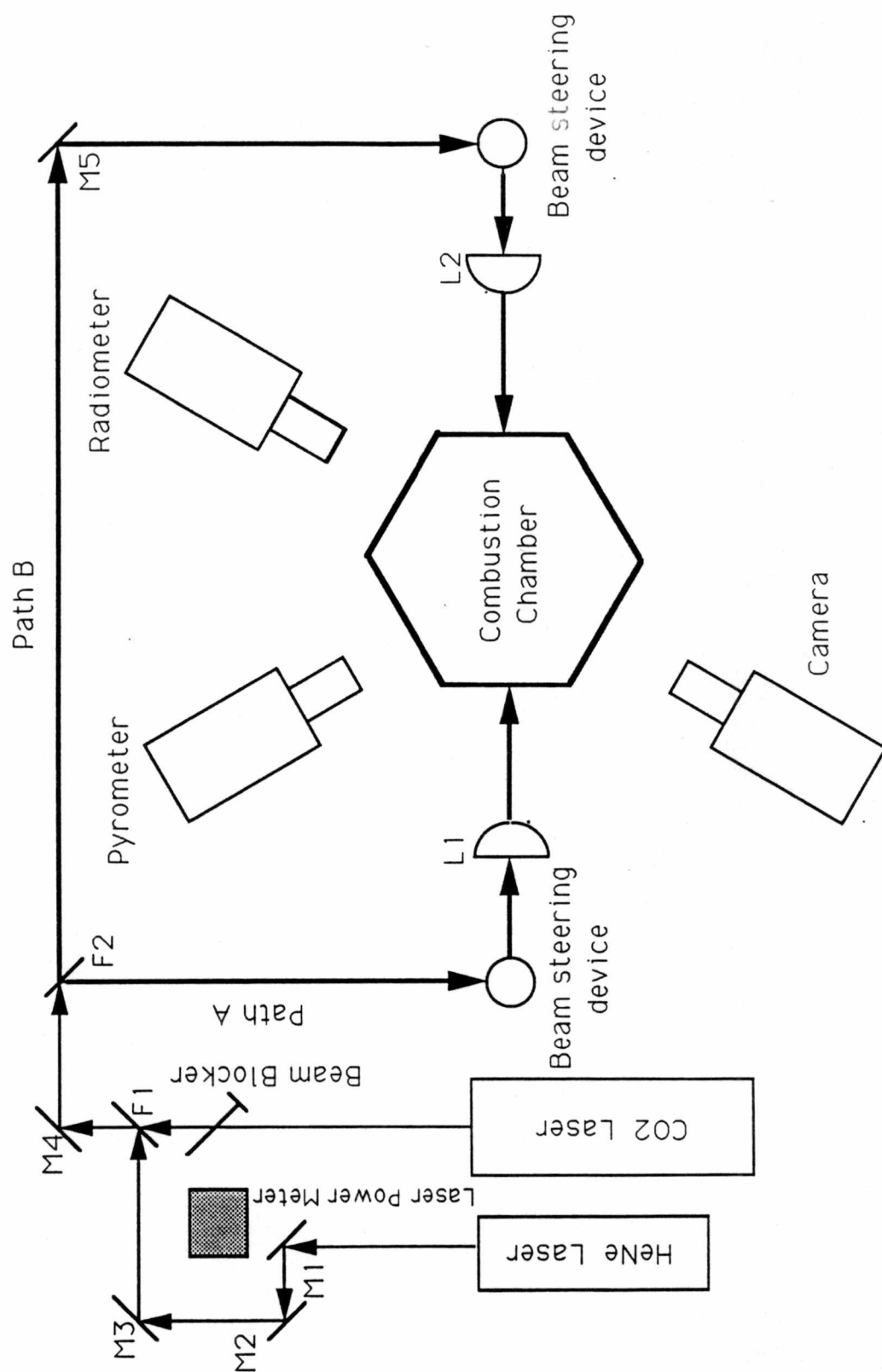


FIGURE 3.1 Schematic Layout of The Experimental Setup.

Since the output of the CO₂ laser beam is in the far infrared (10.6 μm), the suspended boron agglomerate is aligned for the focused CO₂ laser beam using a He-Ne laser. The visible He-Ne laser beam is introduced into the focusing system by mirrors M1,M2, M3 and ZnSe optical flat F1. As the He-Ne laser beam emerges from the optical flat, it coincides with the CO₂ laser beam. Alignment is achieved when He-Ne laser beam illuminates the agglomerate. Once alignment is perfected, the He-Ne laser beam is blocked off and the CO₂ laser beam is introduced into the focusing system by means of a solenoid-activated beam blocker. By energizing the solenoid, the spring-loaded arm of the beam blocker swings out of the way of the CO₂ laser beam, creating a shutter. In this manner the duration of the "laser pulse" can be varied from 40 ms to 1.0 s.

The surface temperature of the laser-ignited boron agglomerate was measured by a fast-response, two-color pyrometer that operates in the near-infrared at two narrow spectral regions centered at 0.8 and 1.0 μm. A broadband radiometer was used to record the radiation emitted by the agglomerate in the spectral region from 0.3 to 1.1 μm.

In conjunction with the two-color pyrometer and radiometer, a Hycam 16 mm high-speed motion picture camera was used to observe the phenomenological events occurred during the burning of the agglomerate: i.e., the flame structure and to correlate between the filmed events and the outputs obtained from the pyrometer and radiometer.

3.2 TEST CONDITIONS

In the present study, all experiments were conducted in a quiescent O₂/N₂(20/80) environment, except when effect of oxygen mole fraction on burning times was studied. The gas mixture was desiccated prior to induction into the combustion chamber to avoid any effects of water vapor on the burning times.

A laser power of approximately 30 W was used at all ambient pressures investigated in this study. The duration of the laser pulse required for ignition were found to be between 100 and 130 ms, with longer pulse duration at lower pressures.

3.3 BORON AGGLOMERATES PRODUCTION TECHNIQUE

Boron agglomerates used in this study are produced at the tip of SiC fibers by a technique developed in this laboratory. Powdered (1-2 μm) amorphous boron is mixed with alcohol and deposited at the tip of the fiber using hypodermic syringe. The alcohol evaporates rapidly, leaving behind a thin coating of solid boron powder. Additional applications of the droplet result in sufficient boron to form a nearly-spherical agglomerate. Measurement and assurance of uniformity of the produced agglomerates are obtained by using a comparator microscope. Figure 3.2 shows a typical agglomerate used in the present study.

3.4 BRIEF DESCRIPTION OF EXPERIMENTAL APPARATUS

This section briefly outlines the experimental apparatus used in this study. For a detailed description readers are referred to reference [23].

3.4.1 HIGH-PRESSURE COMBUSTION CHAMBER

The combustion chamber consists of the main body and the enclosure flange. The enclosure flange with a diameter of 12 cm and thickness of 100 mm is machined from 316

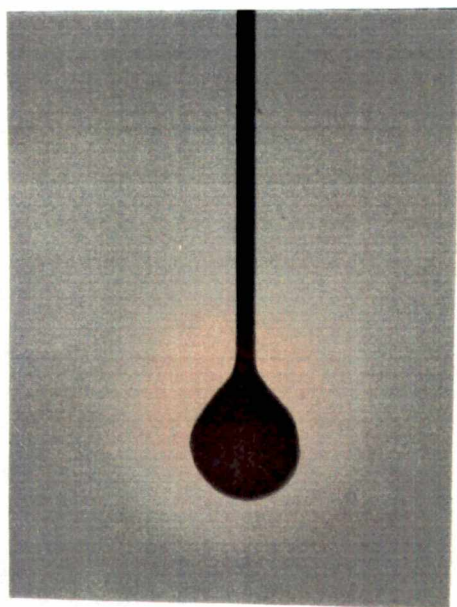


FIGURE 3.2 Photomicrograph of a Typical Boron Agglomerate.

stainless steel and secured to the main body by eight machined screws. Sealing between the flange and the main body is provided by O-ring seal.

The main body of the combustion chamber is also fabricated from 316 stainless steel. It has an internal diameter of 6.5 cm, and a height of 12 cm and a wall thickness of 3 cm. The 0.8 l internal volume of the combustion chamber is large enough to facilitate the manual placement of the boron agglomerate as well as to prevent pressurization caused by the heated ambient from being excessive.

There are six optical windows on the periphery of the main body of the combustion chamber. The six equally-spaced windows are placed at an angle of 90° with respect to the combustion chamber axis. Two of these optical windows are of ZnSe and are used to transmit the CO₂ laser beam. Of the remaining four, two are used to provide optical access for the two-color pyrometer and broadband radiometer. The third window is reserved for the high-speed camera, whereas the fourth is used for observing the event during the course of the experiment. Figure 3.3 shows the cross-sectional view of the high-pressure combustion chamber.

3.4.2 CO₂ LASER SYSTEM

The laser optical resonator, shown in Figure 3.4 is formed by a gold coated spherical concave copper mirror (100 % reflection) of 10.0 m radius of curvature and ZnSe flat partial reflector (35% reflection). The two mirrors are separated by approximately 2.5 m. Since the separation distance between the two mirrors is less than the radius of curvature of the concave spherical mirror, the resonator is referred to as a quasi-hemispherical resonator. Power is coupled out of the laser through the ZnSe partially-reflective flat mirror via the zero-order reflection.

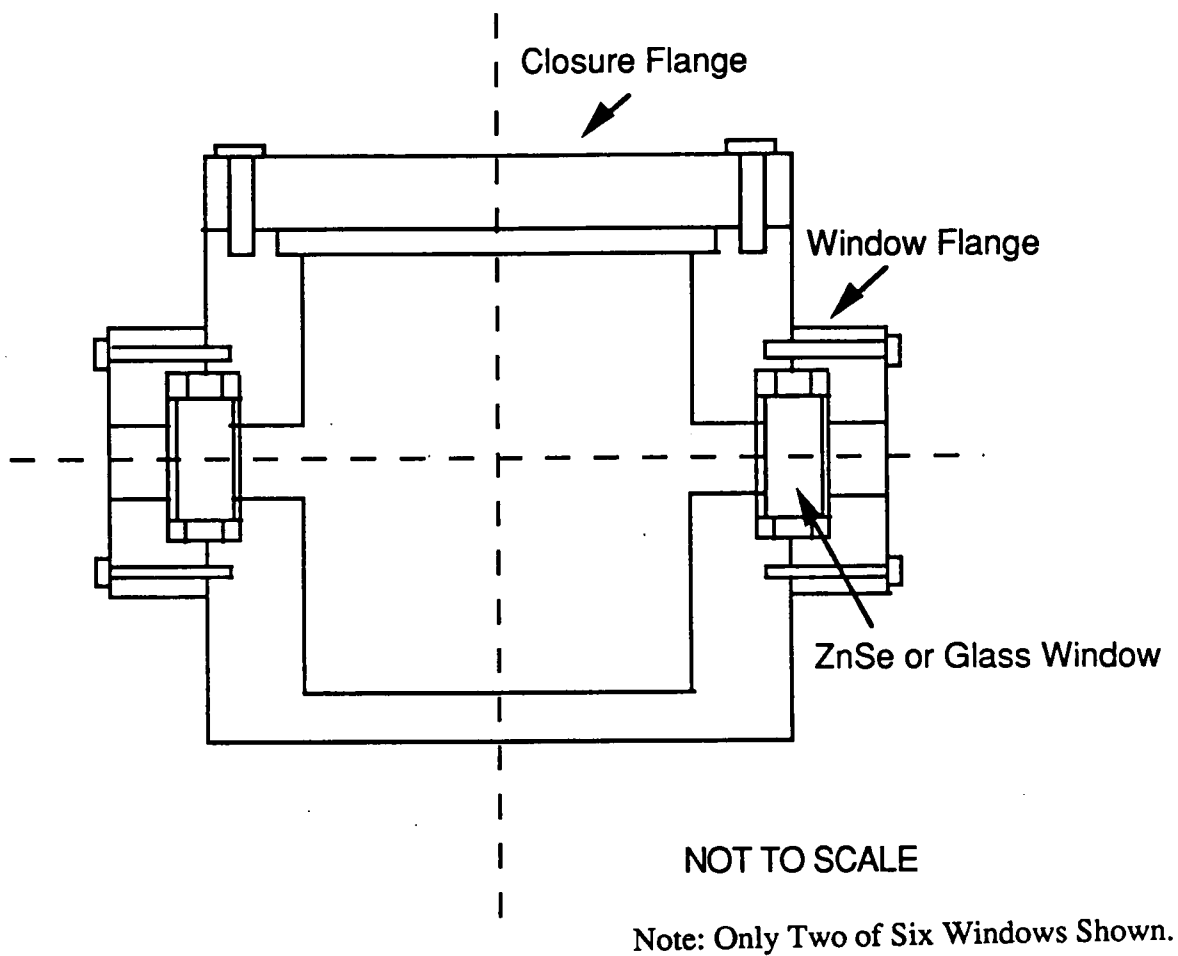


FIGURE 3.3 Cross-Sectional View of The Combustion Chamber.

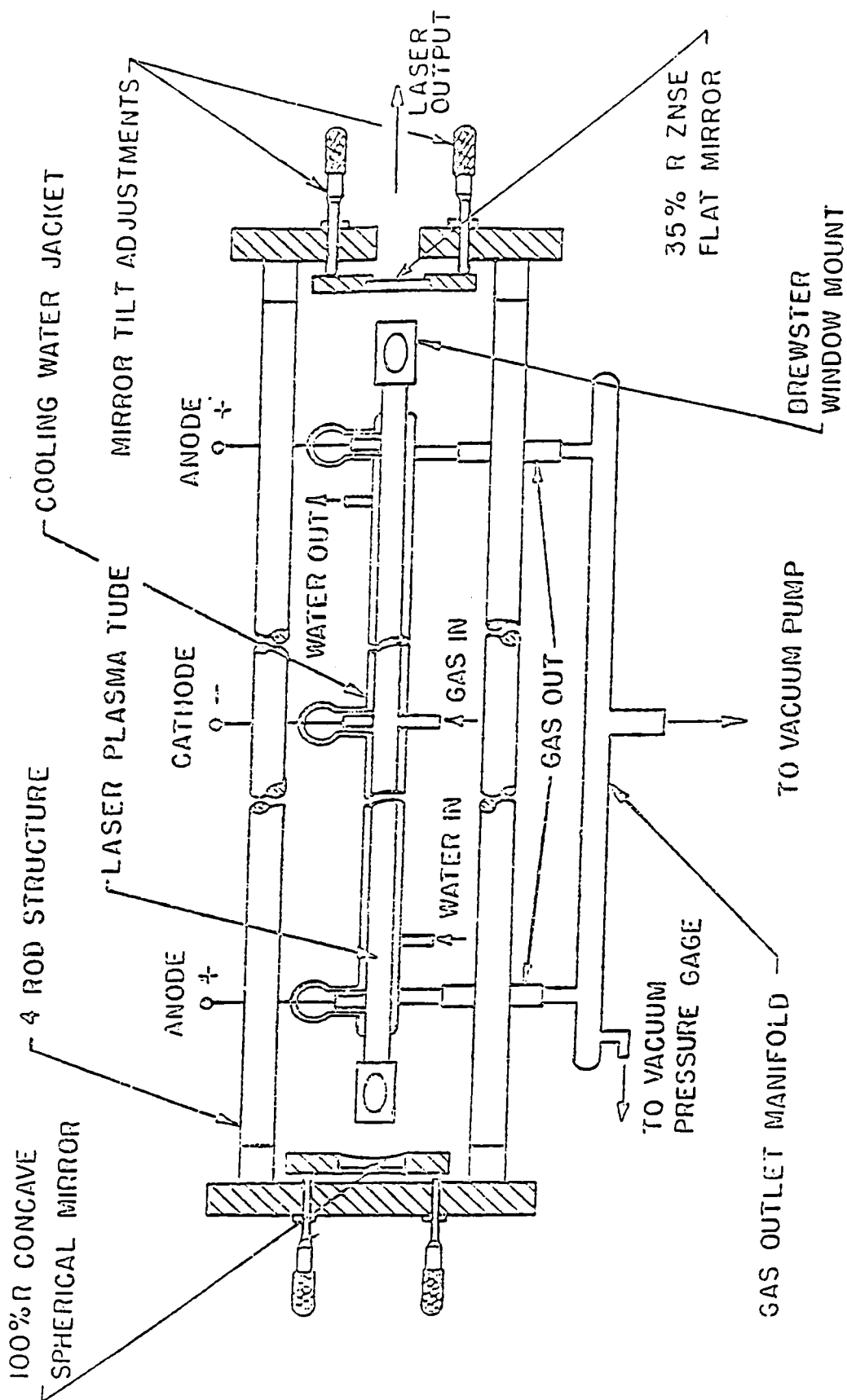


FIGURE 3.4 Top View of Laser Optical Resonator, Laser Plasma Tube and Mechanical Structure of CO₂ Laser.

A four rod structure is used to support the mirror assemblies and the laser plasma tube as shown in Figure 3.4. The laser plasma tube is constructed of Pyrex and consists of an 18 mm inner bore and a 38 mm diameter water cooling jacket. There are two anodes located towards the ends of the laser plasma tube and a cathode located at the midsection of the tube. One central gas inlet and two outlets are provided to ensure a uniform gas flow throughout the amplifying region. At the ends of the laser plasma tube, two rectangular ZnSe windows are mounted at a Brewster angle of 67.4° . At this Brewster's angle, the electric-field component in the plane of incident is transmitted with no reflection, thereby the output of CO₂ laser in the current design is linearly polarized in the vertical direction.

The gases are premixed in a mixing chamber and drawn through the laser plasma tube by a vacuum pump. For optimum operation a mixture of 80% helium, 15% nitrogen, and 5% CO₂ is used. The pressure of the laser plasma tube is monitored at the outlet of the tube by a vacuum gauge. The best performance of the laser is obtained at a gas flow rate of 1.5 lpm and a pressure of 10 torrs.

A 20KV, 100 mA universal Voltronics power supply is used to excite the gas mixture. The maximum power of CO₂ laser is obtained at a discharge current of 70 mA. For safety consideration a number of safety switches and relays are installed to ensure that the power supply will not operate when the laboratory door is accidentally opened.

3.4.3 NEAR INFRARED TWO-COLOR PYROMETER

A fast response, two-color pyrometer is used for temperature measurement. The pyrometer operates in the near-infrared at two spectral regions centered at 0.8 and 1.0 μm , and has a temperature range from 1500 to 4000 K with a maximum time resolution of 25 μs .

A schematic diagram of the optical arrangement of the two-color pyrometer is shown in Figure 3.5. Radiation emitted by the laser-ignited boron agglomerate is focused onto the common leg of a bifurcated fiber optic cable by a zoom lens and a swinging mirror M. The bifurcated fiber optic cable splits the incident beam into two separate beams. Both beams transmitted by the fiber optic cable are focused on to detector housings after passing through interference filters. One interference filter (IF_1) is centered at $0.8\ \mu\text{m}$, whereas the other (IF_2) is centered at $1.0\ \mu\text{m}$.

Precise alignment of two-color pyrometer to the agglomerate is obtained by centering the image of the agglomerate on the center of the crosshair graticule fitted to the wide field Ramsden-type eyepiece with a magnification of 10. Once the alignment has been obtained, the mirror is lowered and radiation emitted by the burning agglomerate is directed to the common leg of the fiber optic cable.

3.4.4 BROADBAND RADIOMETER

The broadband radiometer is similar in design and construction to the two-color pyrometer. However, only a single fiber optic cable was employed rather than a bifurcated cable. Since no interference filter was used, the radiometer is sensitive to a broad spectral region between 0.3 and $1.1\ \mu\text{m}$. The output of the radiometer is proportional to the radiation emitted by the burning boron agglomerate. The optics of the broadband radiometer is similar as that of the pyrometer, but the field of view at the target point is of the order of $2\text{-}3\ \text{mm}$.

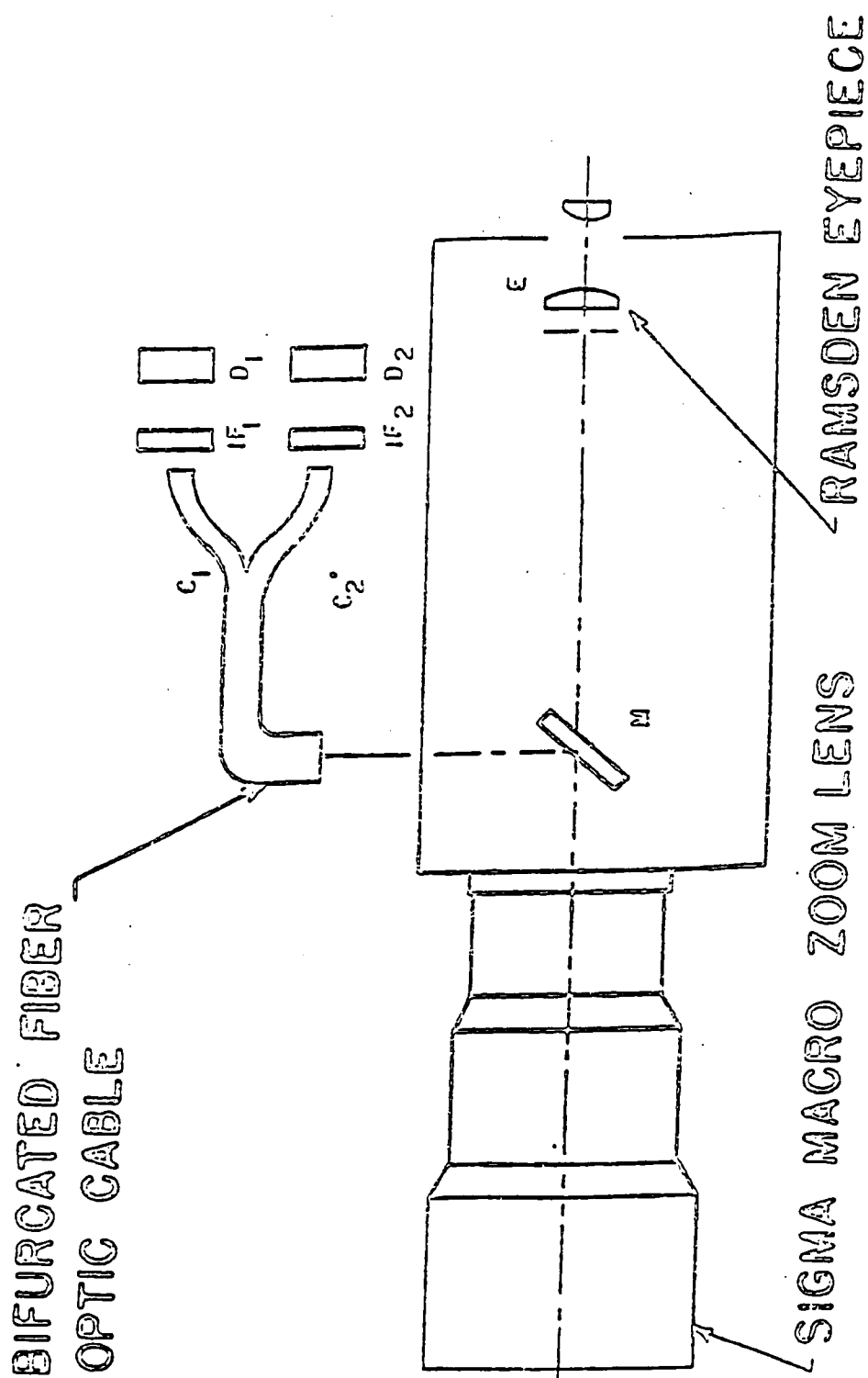


FIGURE 3.5 Schematic Diagram of The Optical Arrangement of Two-color Pyrometer.

3.4.5 HIGH-SPEED CAMERA

A Hycam model K2004E-115 16 mm high-speed motion picture camera is employed to make observations of the combustion phenomena during selected test runs. The camera is positioned such that its objective lens is approximately 5 cm from the boron agglomerate.

When the camera is employed on a test run, there is a provision whereby the shutter/beam blocker timer circuit and the data acquisition system are both triggered after approximately 25 feet of the 100 foot roll of film has been exposed. This allows the camera to reach a steady frame rate as well as allowing the first several layers of film which have been partially exposed during the loading process, to move past its shutter. A neon light in the camera body is connected to a regulated timer which sends out pulses at a 1kHz frequency. This produces a dot at the edge of the film which allows an accurate correlation between the events on the film and the data being logged from the pyrometer and radiometer.

3.5 EXPERIMENTAL PROCEDURE

The experimental procedure is described in this section. Prior to performing the experiments, several preliminary steps are taken. The two-color pyrometer is first calibrated, for any drift of the electronic circuitry caused by the fluctuating temperatures. The vacuum pump for the CO₂ laser is then used to evacuate the plasma tube, and simultaneously water circulation through the laser plasma tube cooling jacket is started. Ionizing gases (He, N₂, CO₂) are pumped into the plasma tube prior to activating the

power supply. The power supply is then activated, with the current set to its maximum value (100 mA). The current is reduced to about 50 mA immediately afterwards.

To produce the brightest, most uniform laser beam (i.e., a Gaussian distribution) the Zn-Se partial reflector is adjusted such that it is perfectly parallel to the gold coated copper mirror. The CO₂ laser power can be adjusted by either varying the power supply current or the plasma tube gas flow.

The alignment of CO₂ laser beam with the agglomerate is checked by using the He-Ne laser beam as the visual reference. In the first step, the plastic strip vertically mounted in the combustion chamber is simultaneously irradiated by the reflected CO₂ beam travelling path A (see Figure 3.1) and the He-Ne laser beam. By adjusting mirrors M1, M2 and M3 the He-Ne can be manipulated such that it is coincident with the CO₂ laser beam. In the second step, the transmitted portion of CO₂ laser beam travelling path B is aligned with the reflected portion of the CO₂ laser beam travelling path A. After laser alignment is achieved, the probe-supported boron agglomerate is mounted inside the combustion chamber with a thin plastic clamp.

The two-color pyrometer, the radiometer and the high-speed camera are then aimed at the agglomerate by sighting through the eyepieces. On subsequent experiments, alignment of the agglomerates is achieved by simply using the initial position settings of the pyrometer and radiometer. The combustion chamber is then evacuated prior to pressurization with oxygen and nitrogen.

Finally the data acquisition system is set and the test is initiated by triggering the beam blocker manually or by starting the high speed camera, if used in a particular test run. Immediately after experiment, a superimposed plot of surface temperature and relative intensity versus time appears on the monitor screen, and the data can be saved to the hard disk for future analysis.

3.6 DATA ACQUISITION AND ANALYSIS

The output voltages from the two-color pyrometer and the radiometer are logged into an IBM PC through a Metrabyte Dash 16 analog/digital converter system. The system has the capability to sample at a rate of 50,000 samples per second. The system is activated by a signal from the shutter timer. When the high-speed camera is used, it activates the timer, thus initiating data collection. As data is collected, a graph of the output voltages as a function of time from both pyrometer and radiometer appears on the monitor screen. The data is processed by a conversion program, before it is saved on the hard disk. The radiometer data is normalized so that the saved information is the relative intensity of the emitted radiation as a function of time. The pyrometer data is processed by the calibration equations so that the surface temperature of the boron agglomerate is saved as a function of time. From the temporal outputs of these two instruments, the three important parameters of this study; i.e., the ignition delay time, the burning time and the surface temperature of the boron agglomerates are determined as will be explained later.

In the present study, boron agglomerates of 534 μm nominal diameter were ignited at pressures between 1.7 and 20.4 atm. A minimum of ten experiments were performed at each pressure to minimize the inherent random error. The burning time of each agglomerate is considered a separate data point. Some experimental data points are eliminated (rejected) directly from inspection (e.g. consumption of fiber). Other experimental data points are eliminated by using Chauvenet's criterion for statistical analysis [24]. Chauvenet's criterion basically states that a data point can be eliminated if the probability of obtaining the particular deviation from the mean (average) is less than $1/2n$, where n is the number of data points in the set. In general, if the ratio of the deviation of a particular reading to the

standard deviation is "relatively" large (depending on the total number of readings), then that data point is eliminated.

CHAPTER 4

EXPERIMENTAL RESULTS AND DISCUSSION

The ignition and combustion of laser-ignited boron agglomerates were studied as a function of ambient pressure and experimental results are given in this chapter. Boron agglomerates of nominal diameter 534 μm were mounted inside a combustion chamber and ignited by a CO_2 laser pulse. A two-color pyrometer and a broadband radiometer were used to measure the surface temperature and the radiation emitted by the burning agglomerate, respectively. Phenomenological study was performed by using high speed cinematography on selected runs.

In the range of pressures (1.7 to 20.4 atm) investigated in the present study, three distinct combustion regimes were observed. In regime I (1.7 to 5.1 atm) partial or incomplete burning of the boron agglomerates occurred. In regime II (6.8 to 13.6 atm) steady-state, full-fledged burning was observed, in which the burning time decreases with increasing pressure. In regime III (above 13.6 atm) violent combustion with frequent particle ejections was observed. A detailed discussion of these regimes is presented in sections 4.1, 4.2, and 4.3 respectively, and the effect of oxygen mole fraction on the combustion of boron agglomerates is discussed in section 4.4.

4.1 REGIME I

In this regime experiments were conducted between the pressure of 1.7 and 5.1 atm. This regime can be classified as a regime of incomplete combustion. After normal ignition and subsequent combustion, agglomerates failed to burn to completion. Section

4.1.1 describes the general observations, as observed from film records and section 4.1.2 discusses the effects of pressure on incomplete burning times.

4.1.1 GENERAL OBSERVATIONS

Figures 4.1a and 4.1b show radiation intensity and surface temperature curves of a laser-ignited boron agglomerate at 5.1 atm, respectively. For this particular experiment, a laser duration of 120 ms was used. The photographs in Figure 4.1a depict the sequence of events in the four prominent stages (discussed below). The events described herein are similar for all pressures investigated in this regime.

In the present study, ignition is defined to occur at the instance at which the flame first appears as evidenced by the film records. Accordingly, the time elapsed between the beginning of laser irradiation and the first appearance of flame; i.e., stage I in Figure 4.1a is the ignition delay time. It should be noted that the small segment of radiation intensity curve preceding the beginning of stage I (~ 30 ms) is due to the mechanical lag of the beam blocker. The ignition delay time or stage I is generally very short for boron (approximately 30 ms) and is consistent at all pressures investigated in this study. Thus, the use of laser ignition technique has suppressed the long ignition delay time, typically obtained from flat flame burners (Macek and Semple [8]; Holl et al [15]). Photograph a shows the flame at the time of its first appearance, and marks the beginning of the burning. The flame envelopes the whole agglomerate and no localized ignition spots are visible. At this juncture the flame consists of a highly luminous inner white zone surrounded by a green envelope as shown in Figure 4.2. However, because of electronic reproduction of the color film, the green color of the BO_2 envelope appears as blue in the photographs. This flame structure has been observed throughout the four stages. The aforementioned

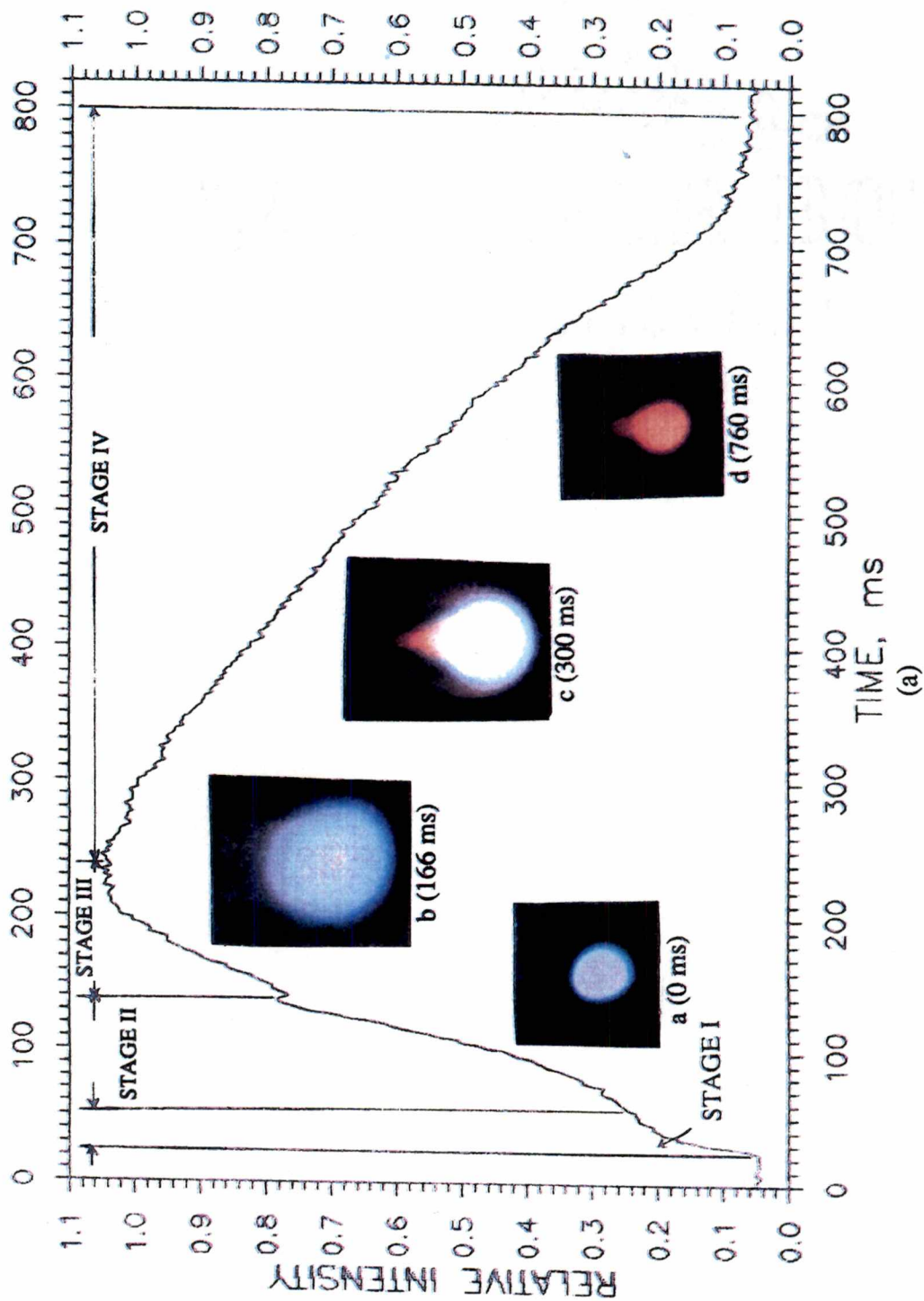


FIGURE 4.1 Typical Sequence of Events (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at an Ambient Pressure of 5.1 Atm (Regime I).

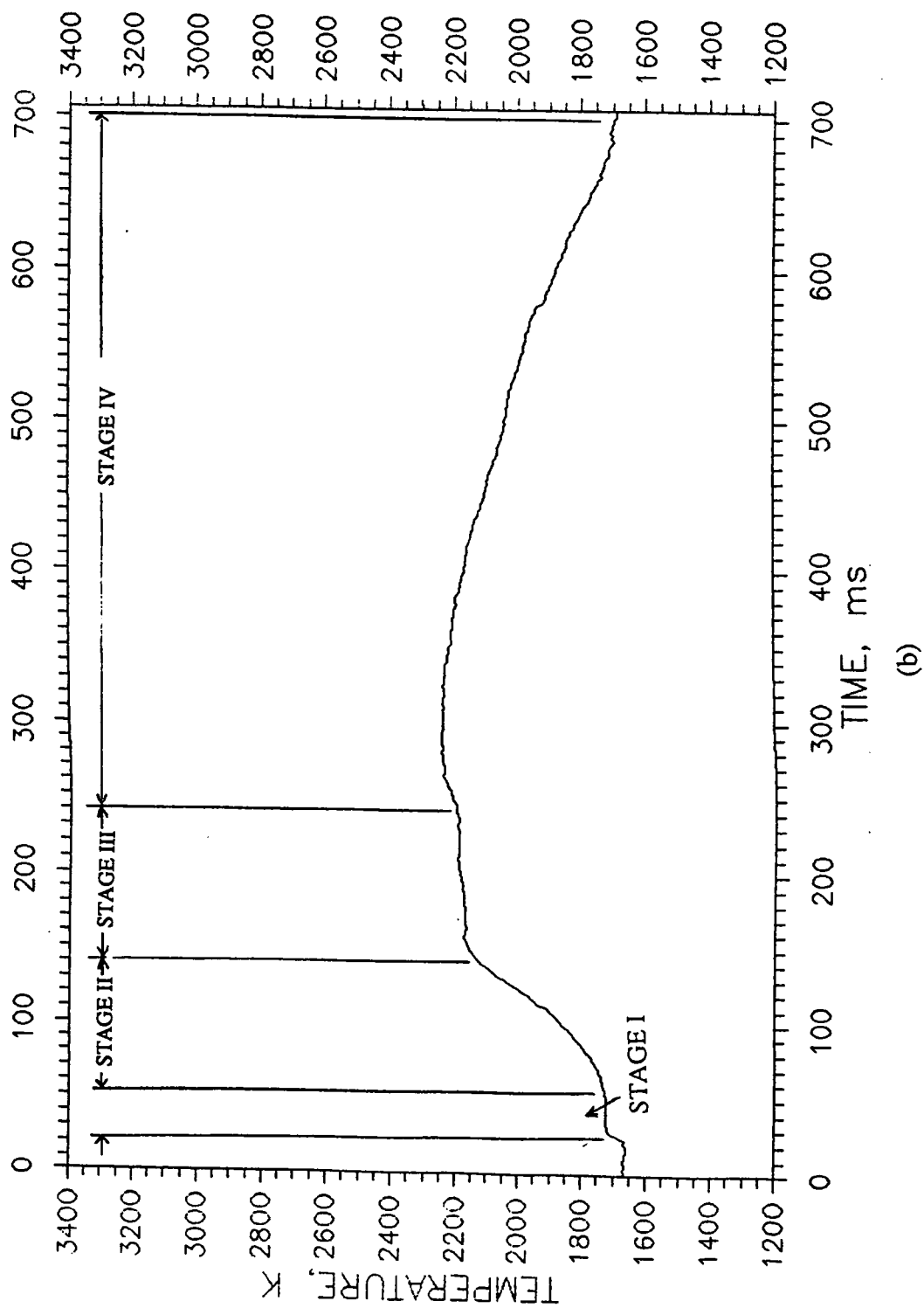


FIGURE 4.1 (continued)

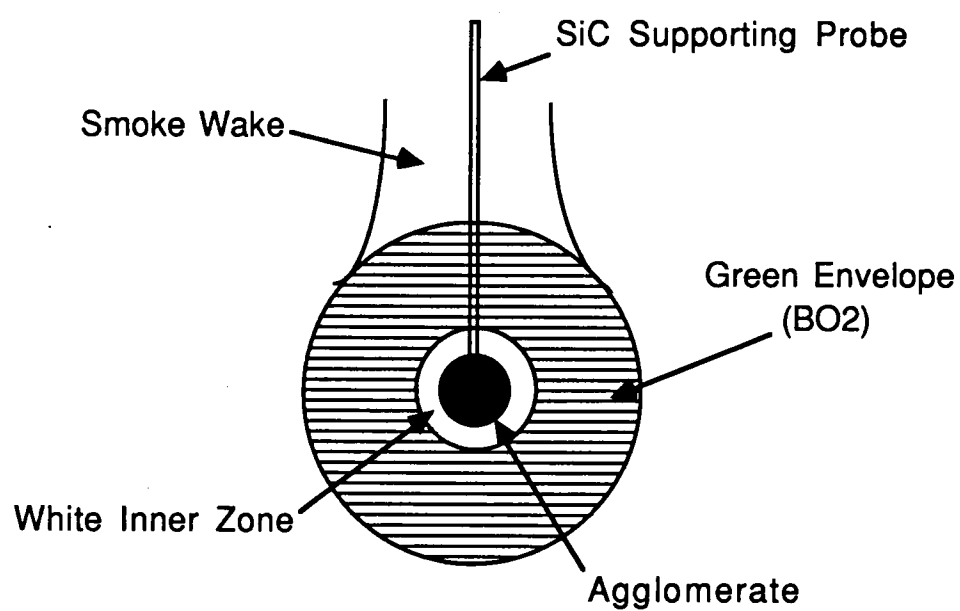


FIGURE 4.2 Typical Flame Structure in Regime I.

green envelope has been observed in previous studies of boron combustion [8,21,22]. Li et al., [21] and Takahashi et al.,[22] have attributed the green color to excited boric oxide (BO_2) emissions. Even in this stage the intensity of the inner zone is so bright such that it is impossible to discern the outline of the agglomerate.

Stage II follows ignition and during this stage the flame grows both in size and intensity. The brightness of the inner zone increases as shown in photograph b taken at 166 ms after ignition. Evidence of brownish smoke trail moving upwards in the wake of the flame is also seen for the first time in this stage. During the subsequent burning this smoke sometimes becomes very thick and surrounds the whole agglomerate.

At the beginning of stage III the flame size reduces and this reduction corresponds to the end of the laser pulse. But soon after laser withdrawal the flame resumes its growth and at approximately about 200 ms after ignition, the intensity curve reaches its peak. In the rest of the stage III, the flame steadily loses its intensity and size, although the flame structure maintains its integrity. Photograph c taken at 300 ms after ignition shows the decaying flame structure during this stage.

Stage IV is the continuation of stage III and the transition is very subtle. The thickness of the green envelope during this stage reduces, and the flame almost clings to the surface of the agglomerate. Stage IV lasts about 250 ms and culminates at the extinguishment of the flame. Radiation intensity curve depicts end of event by returning to the initial value. Photograph d taken at 760 ms after ignition shows the last glowing of agglomerate, before extinguishment. The burning time for this particular experiment was determined to be approximately 770 ms. Almost in all experiments in this regime, only half of the agglomerate was consumed and the remains were found covered by a white colored powder characteristic of boron oxide (B_2O_3).

4.1.2 EFFECTS OF PRESSURE

Figures 4.3 and 4.4 show the surface temperature and radiation intensity curves of typical boron agglomerates ignited at 1.7 and 3.4 atm, respectively. The effect of pressure on the burning times of boron agglomerates in this regime is shown in Table 4.1. Results show that the agglomerates extinguish earlier at lower pressures than at higher pressures in this regime.

Results seem to indicate that the reduction in oxygen quantity at low pressures is responsible for this observed trend. Furthermore in this regime the surface temperature of the agglomerates never exceeds the boiling temperature of boron oxide (2316 K) as seen in Figures 4.3b and 4.4b. In such a case the rate of production of boron oxide exceeds the rate of removal and eventually leads to the thickening of the boron oxide layer which causes the combustion process to cease. Radiation intensity curves, for all pressures investigated in this regime, exhibit a similar dome shaped curve signifying incomplete combustion as shown in Figures 4.3a and 4.4a.

In this regime the flame appears to be less intense throughout the burning of the agglomerate. The inner white zone appears to be contiguous to the agglomerate surface and its intensity is also less than that in other regimes. Due to these reasons the green envelope (BO_2) extends all the way out from the agglomerate surface. Brownish smoke is produced in great quantities in this regime, and at times the smoke appears to form a covering screen around the flame zones, thus hiding the flame partially. In this regime particle ejections are also completely absent, and the flame slowly diminishes in intensity and color resulting in partial combustion of agglomerates.

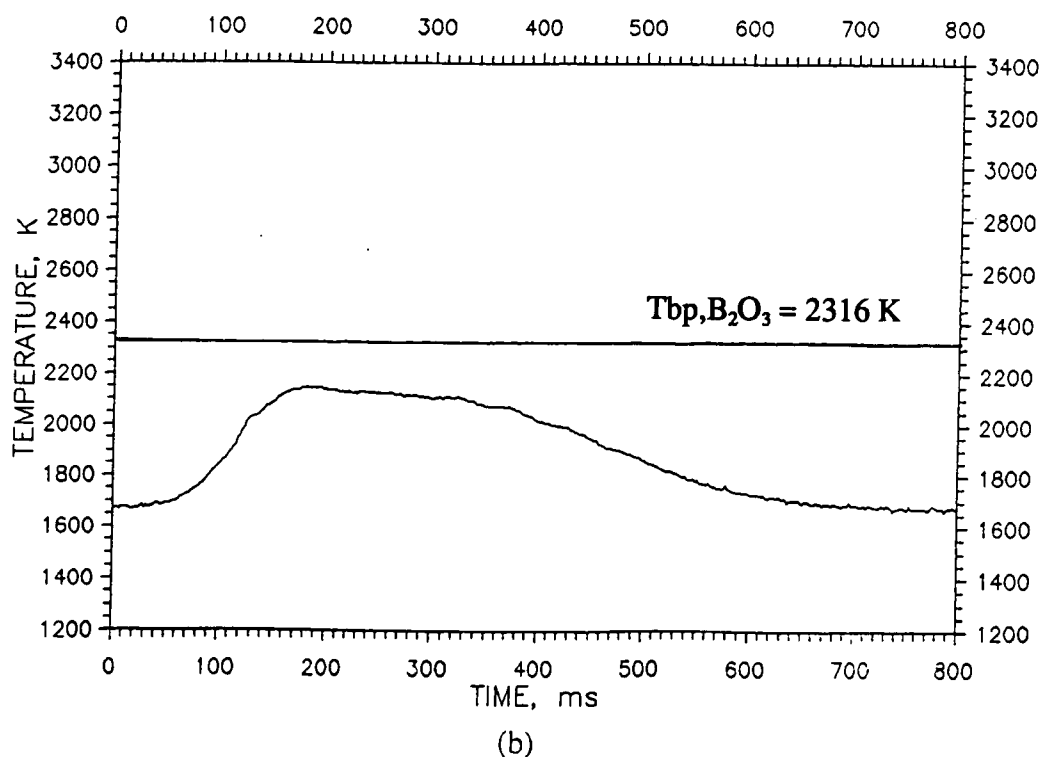
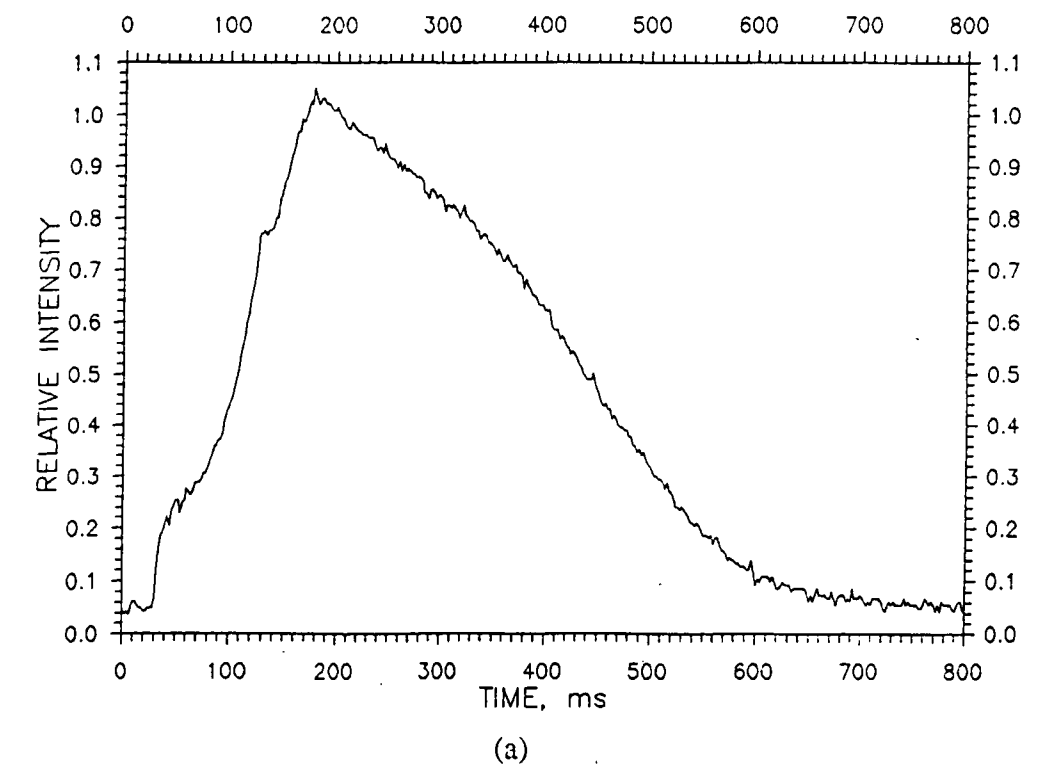
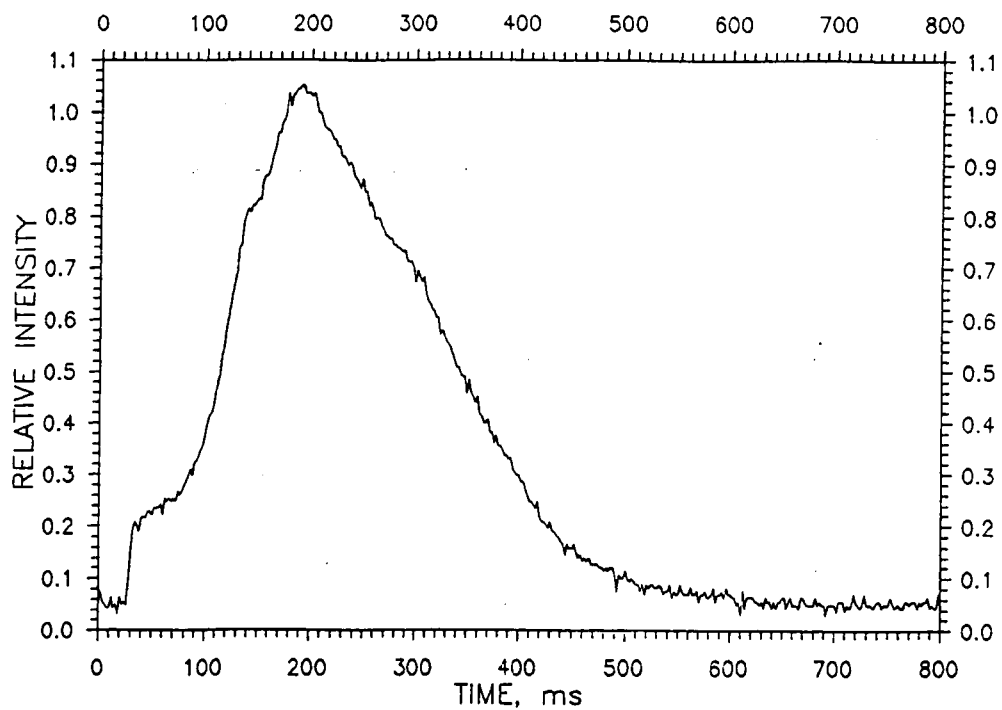
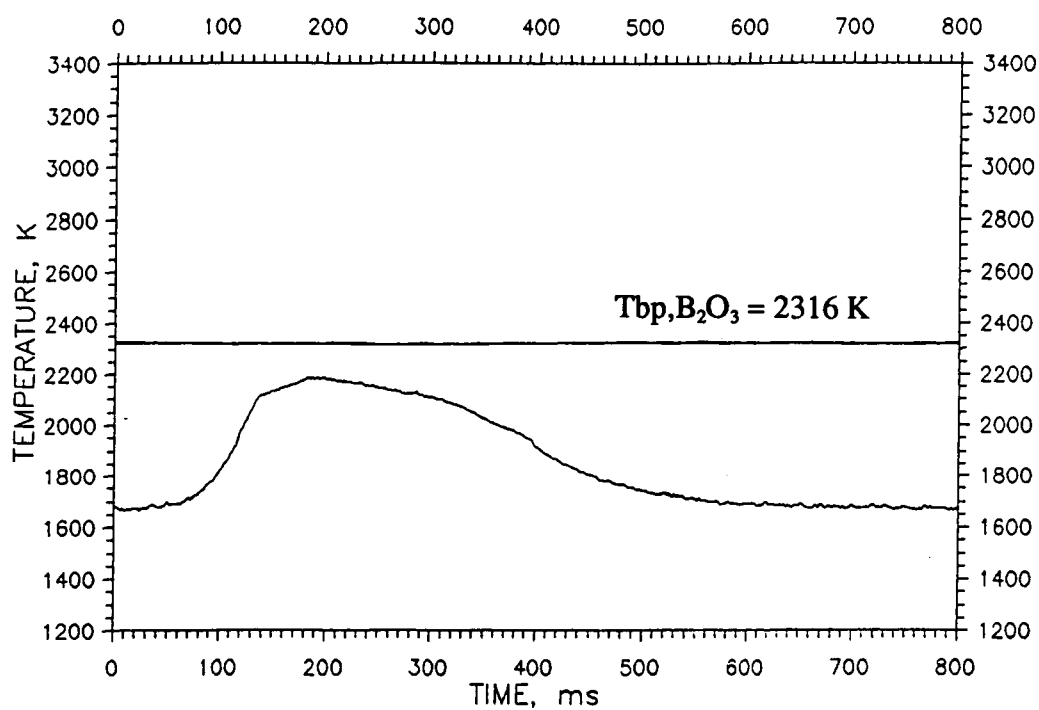


FIGURE 4.3 Typical Radiation Intensity Curve (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at Ambient Pressure of 1.7 Atm (Regime I).



(a)



(b)

FIGURE 4.4 Typical Radiation Intensity Curve (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at Ambient Pressure of 3.4 Atm (Regime I).

TABLE 4.1 Burning Times of Boron Agglomerates in Regime I.

Pressure (atm)	Burning Times (ms)*
1.7	600 ± 120
3.4	668 ± 182
5.1	853 ± 137

*Incomplete Combustion.

4.2 REGIME II.

This regime is characterized by steady-state full-fledged burning with sporadic particle ejections. In this regime experiments were performed between the pressures of 6.8 and 13.6 atm. Section 4.2.1 includes the general observations, whereas effects of pressure on burning time is addressed in section 4.2.2.

4.2.1 GENERAL OBSERVATIONS

Figures 4.5a and 4.5b show the radiation intensity and surface temperature curves, of a typical laser-ignited boron agglomerate at 6.8 atm, respectively. The photographs in Figure 4.5a represent the sequence of events in the four prominent stages (discussed below). Laser duration for this particular experiment was 100 ms. Phenomenological events described herein are general in nature for this regime and have been observed repeatedly.

According to the definition of ignition in this study, stage I is the ignition delay time. The surface temperature of the agglomerate at the time of ignition, as obtained from pyrometer, is approximately 2300 K, which is of the same order as the melting temperature of boron oxide. Photograph a shows the flame at the time of its first appearance; i.e., ignition. Again there is no localized ignition and the flame is composed of a inner white zone surrounded at its edge by a green boric oxide (BO_2) envelope, as shown in Figure 4.6.

Stage II follows the ignition and lasts until the end of the laser pulse duration (~ 70 msec). During this period the flame grows both in size and intensity. The brightness of the inner region increases many folds as depicted by photograph b taken at 70 ms after

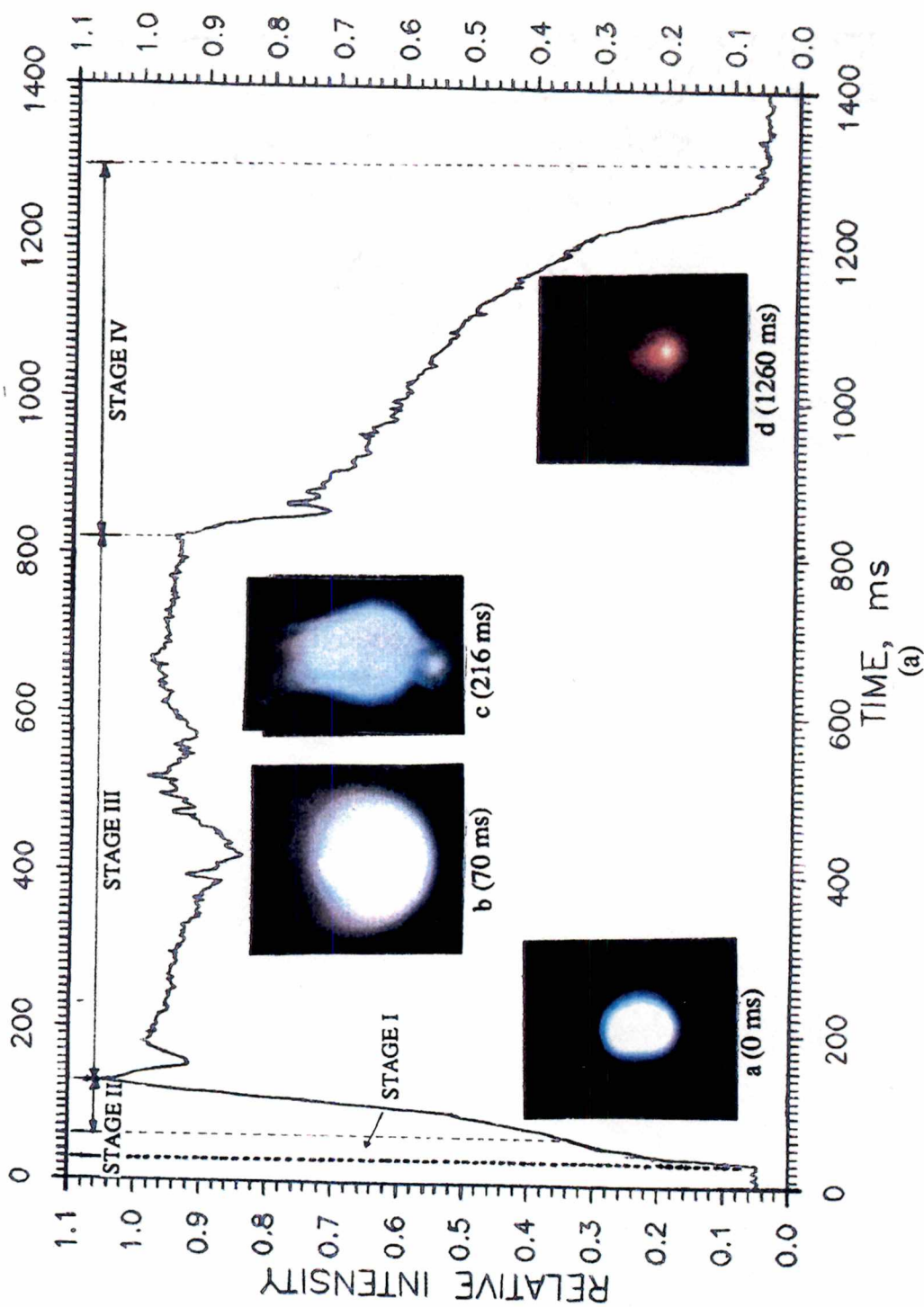


FIGURE 4.5 Typical Sequence of Events (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at an Ambient Pressure of 6.8 Atm (Regime II).

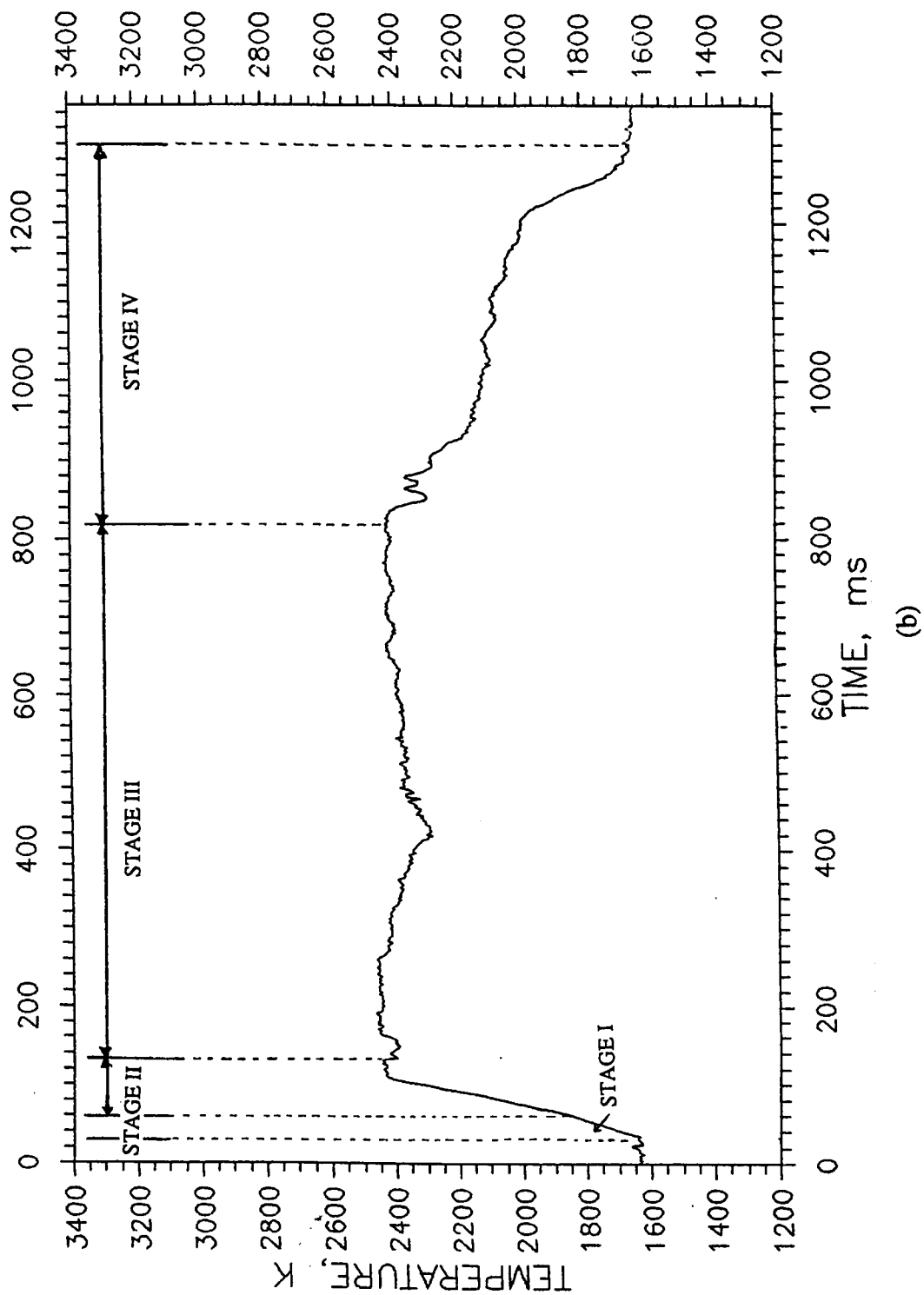


FIGURE 4.5 (continued)

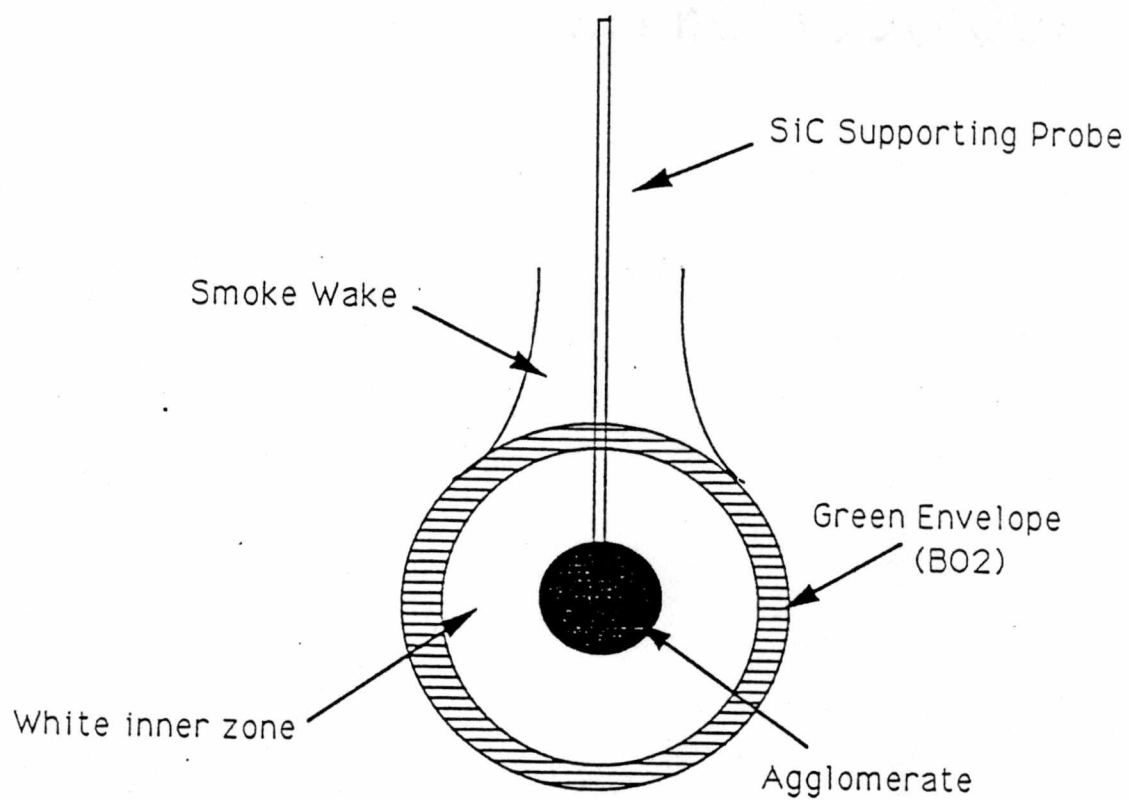


FIGURE 4.6 Typical Flame Structure in Regime II.

ignition. As in regime I, evidence of upward moving smoke trail in the wake of the flame was observed for the first time. However, the generated smoke is lesser in quantities in this regime and its color is also not as bright. The upward movement of the smoke could be attributed to the effects of thermal convection as the combustion is taking place in a stagnant environment.

Smooth transition from stage II to stage III follows with a reduction in the flame size. Laser shut-off time coincides with this reduction in flame size, delineating the withdrawal effects of a physical heating source. But after a minute initial reduction, the flame stabilizes and the agglomerate continues to burn in a self sustained combustion mode. The flame is detached and is much bigger than the agglomerate diameter. Nevertheless the agglomerate outline is still indiscernible. Although the combustion in this regime is very stable, sporadic instances of particle ejection do occur in stage III, as shown in photograph c taken at 216 ms after ignition. Higher activity and subsequent pressure build-up beneath the surface causes these particle ejections. These ejected particles burn in exactly the same manner as the parent agglomerate; i.e., the surrounding flame structure is similar.

Stage IV comprises the decaying combustion phase. During this stage the combustion becomes progressively less intense, with a rapid decrease in flame luminosity and agglomerate diameter. Green envelope surrounding the agglomerate becomes thinner and loses some of its intensity (see photograph d). The end of this stage is marked by the disappearance of flame on the film records and a corresponding flattening of intensity curve. In this particular experiment the burning time is determined to be 1340 ms.

4.2.2 EFFECTS OF PRESSURE

This regime is characterized by steady-state burning with sporadic particle ejections of boron agglomerates. In this regime experiments were performed between pressures of

6.8 and 13.6 atm. All experiments proceeded to complete combustion and no traces of boron was found on the SiC fiber. Also in this regime, no instances of fiber consumption were observed.

Figures 4.7 and 4.8 show the surface temperature and radiation intensity curves of boron agglomerates ignited at 10.2 and 13.6 atm, respectively. The effect of pressure on burning time in this regime is shown in Table 4.2 and plotted in Figure 4.9. Results show that the burning times decrease with increasing pressures. Macek and Semple[11], while studying combustion of boron particles at elevated pressures also observed a similar trend, yet the agglomerate burning times determined in the present study are not consistent with the burning times of boron particles, therefore it is not possible to identify any similarities or differences between the single particle combustion and the agglomerate combustion. The surface temperature histories of agglomerates in this regime (see Figures 4.8b and 4.9b) show that the temperatures in this regime are consistently in the vicinity of boiling temperature of boron oxide (2316 K). This feature is in contrast to regime I, in which the temperatures seldom exceed 2200 K. The radiation intensity curves are also different from the dome shaped curves of regime I.

The flame structure in this regime also consists of two zones: A bright inner white zone and a surrounding green (BO_2) envelope. The apparent size of the flame (inner zone and green envelope) is of the same order as in regime I, yet the relative sizes of two zones have changed. Since the size of the inner zone is thinnest in regime I, the green envelope can be seen to extend all the way out from the agglomerate surface. However in regime II, the green envelope is visible only at the edge of the inner white zone. This is not surprising since the white color of the inner zone is a combination of a number of different colors. In addition, the intensity of the inner zone also increases in regime II. The increase in the intensity of the inner zone could possibly be due to higher surface temperatures attained in

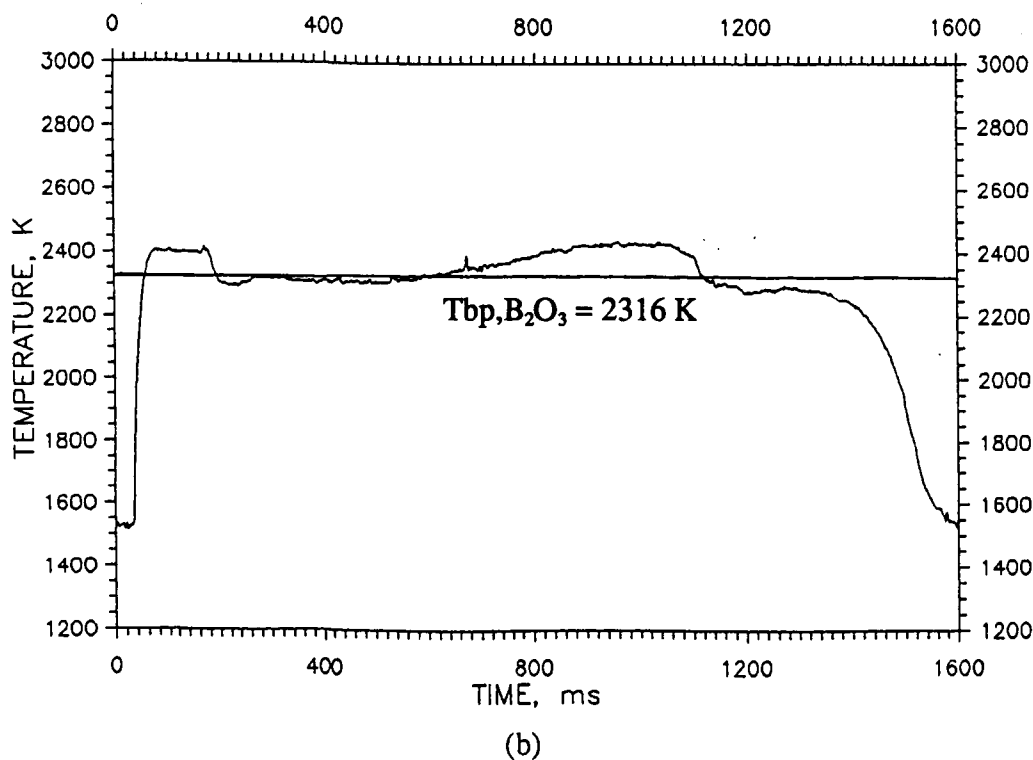
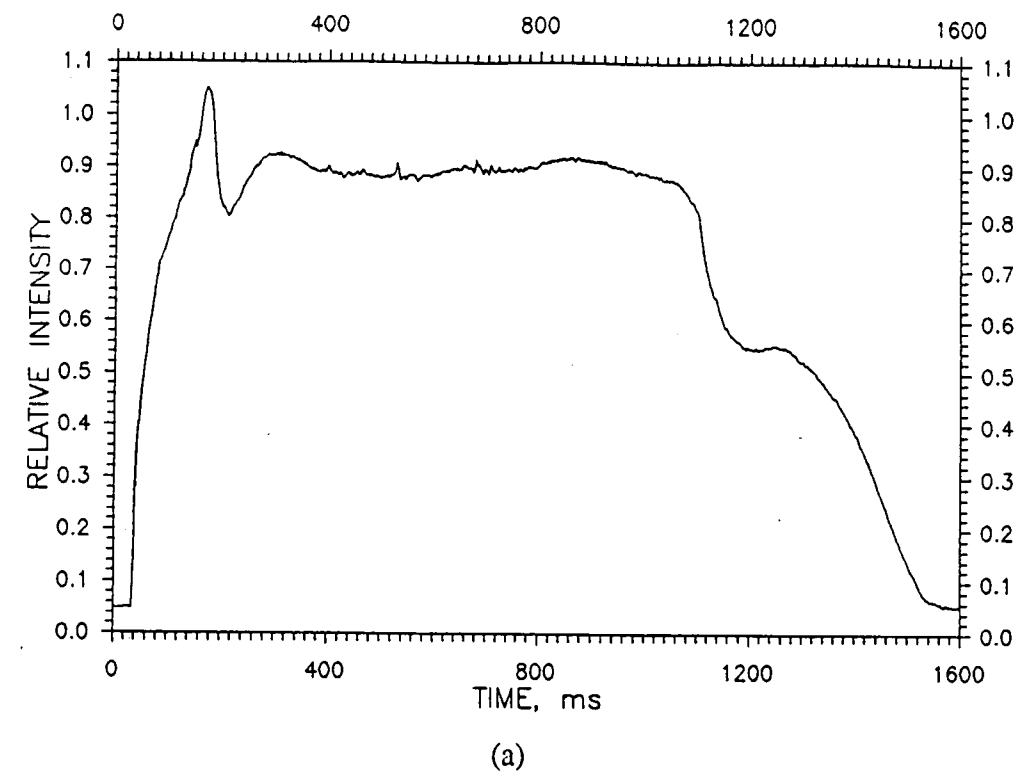
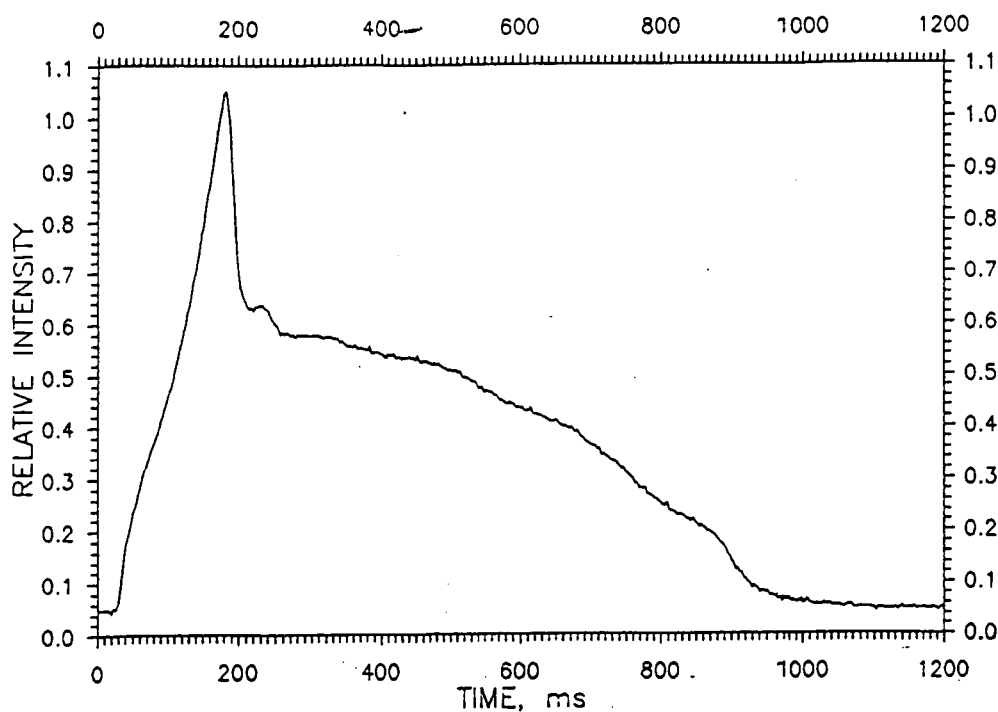
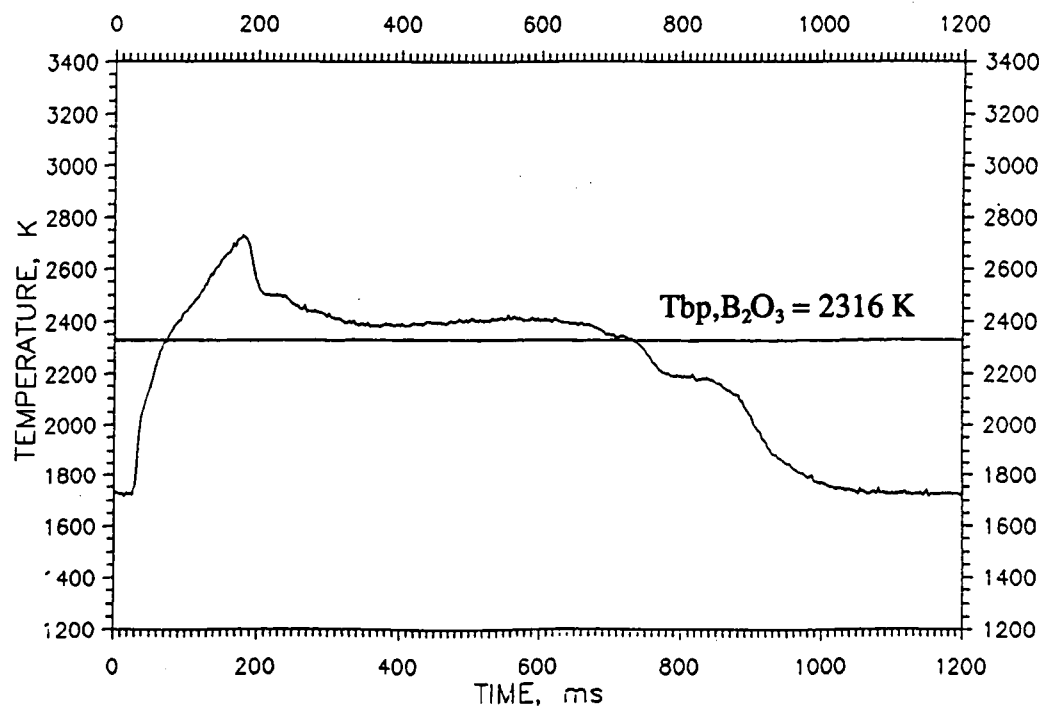


FIGURE 4.7 Typical Radiation Intensity Curve (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at Ambient Pressure of 10.2 Atm (Regime II).



(a)



(b)

FIGURE 4.8 Typical Radiation Intensity Curve (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at Ambient Pressure of 13.6 Atm (Regime II).

TABLE 4.2. Burning Times of Boron Agglomerates in Regime II.

Pressure (atm)	Burning Times (ms)
6.8	1666 ± 192
10.2	1038 ± 196
13.6	975 ± 117

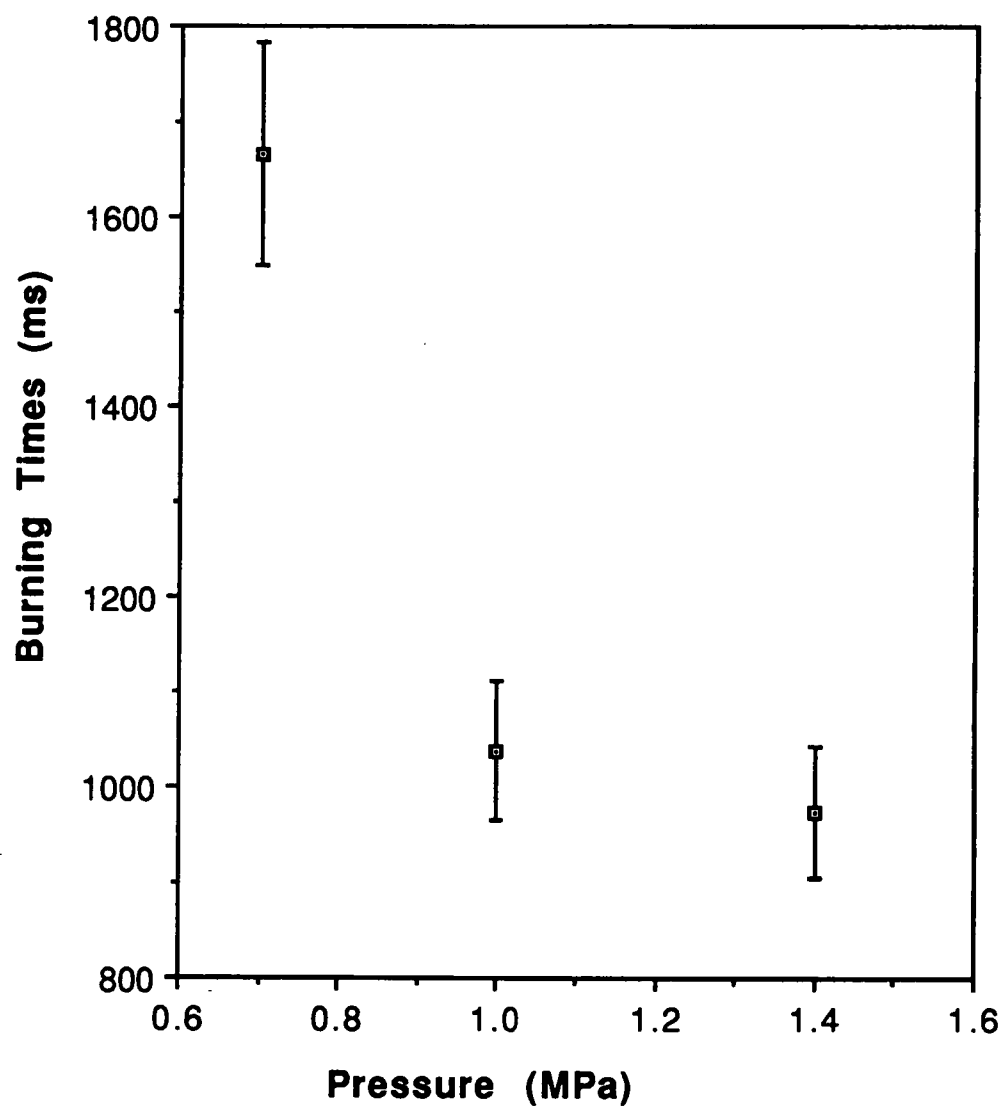


FIGURE 4.9 Effect of Pressure on Burning Times in Regime II.

this regime. The smoke generation in this regime was also much less than that in regime I, and the smoke appeared to be emanating only from the top of the flame.

4.3 REGIME III .

In this regime experiments were performed at pressures above 13.6 atm. This regime is characterized by violent burning with frequent particle ejections. Fiber consumption rendered the burning time data unreliable, therefore only phenomenological events, as observed from film records are presented for this regime.

Radiation intensity and surface temperature curves of a typical boron agglomerate, ignited at 20.4 atm are shown in Figures 4.10a and 4.10b, respectively. The photographs in Figure 4.10a depict the sequence of events during ignition and combustion.

Stage I in Figure 4.10a is the ignition delay time. Photograph a shows the flame at the time of its appearance. As in regimes I and II, the flame front consists of two distinct zones at ignition: an inner white zone surrounded at its edge by a green (BO_2) envelope.

Stage II begins with rapid growing of flame. Soon the flame movement about the fiber begins and apparent swelling of the agglomerate is observed as shown in photograph b taken at 120 ms after ignition. The flame size reaches its acme at about 170 ms after ignition. The diameter of the inner zone is largest in this regime and is brighter than that in the previous regimes. In this regime also the green envelope is only visible at the edge of the inner zone, as in regime II. However, the flame size (combined size of zones I and II) is still of the same order as in other regimes. Figure 4.11 shows a typical flame structure in regime III, just before the beginning of most violent activity during stage III.

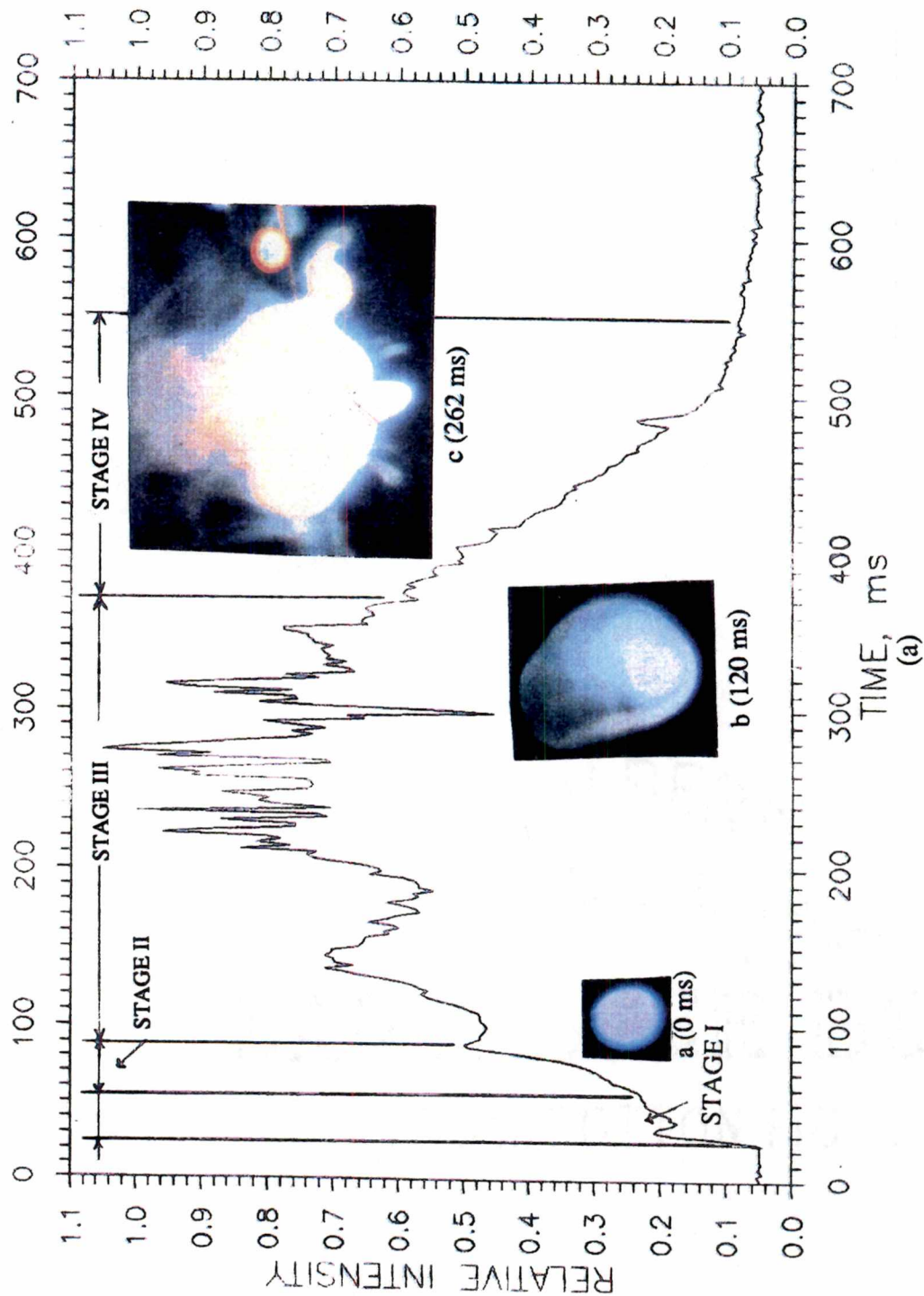


FIGURE 4.10 Typical Sequence of Events (a) and Surface Temperature Curve (b) of a Laser-Ignited Boron Agglomerate at an Ambient Pressure of 20.4 Atm (Regime III).

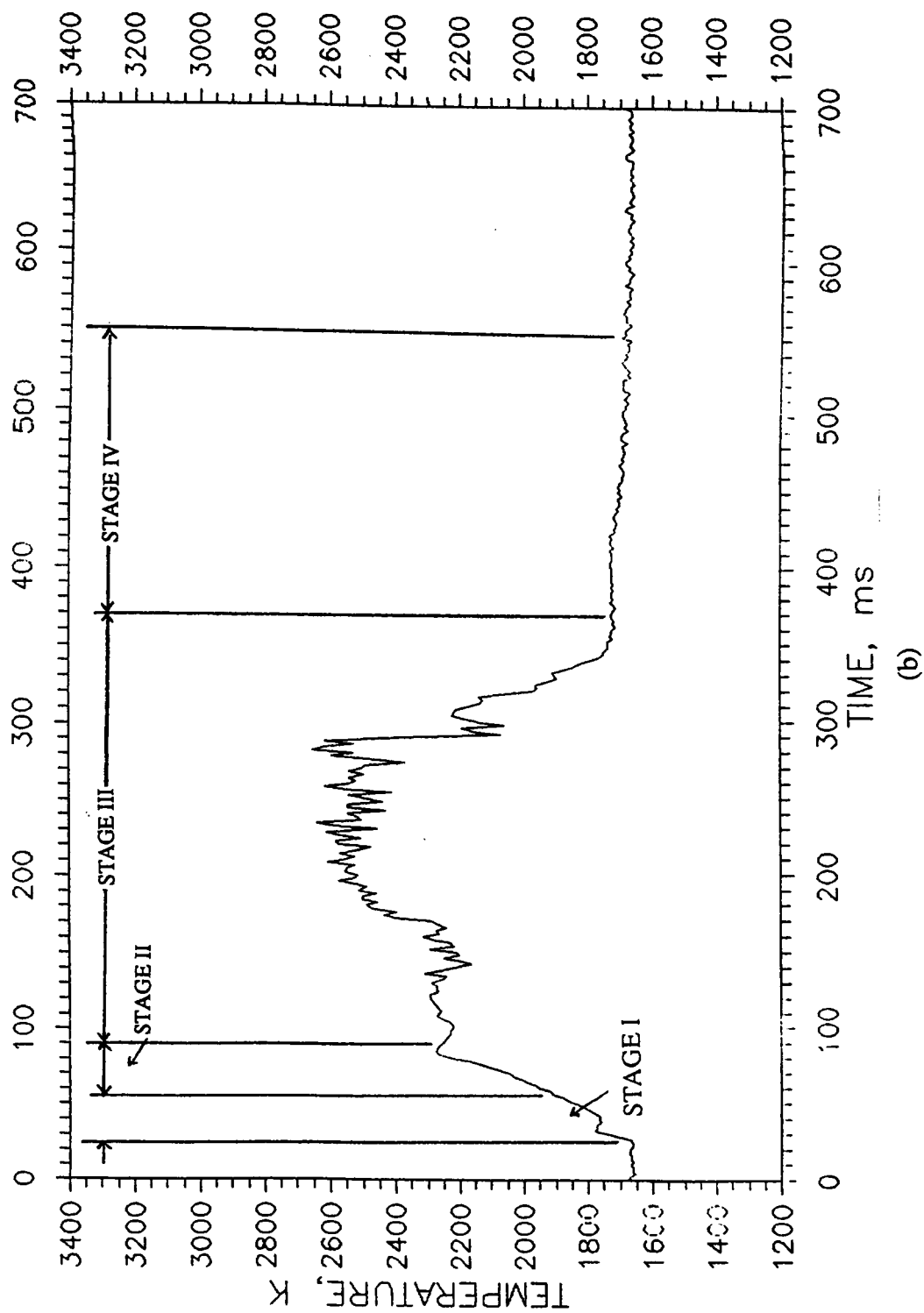


FIGURE 4.10 (continued)

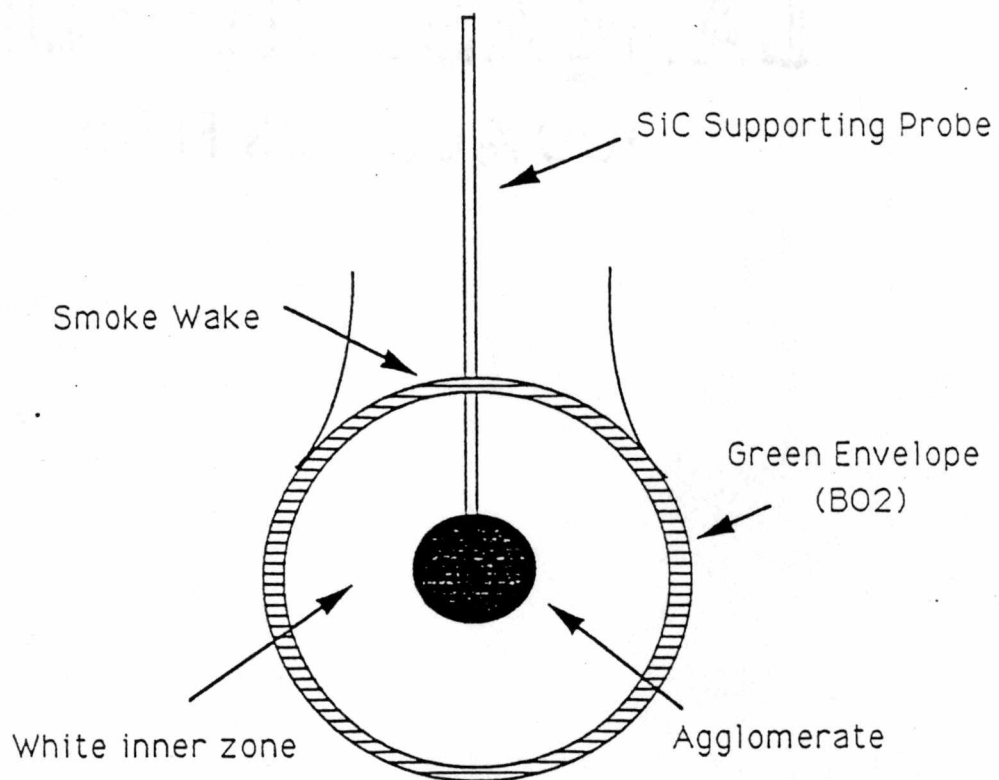


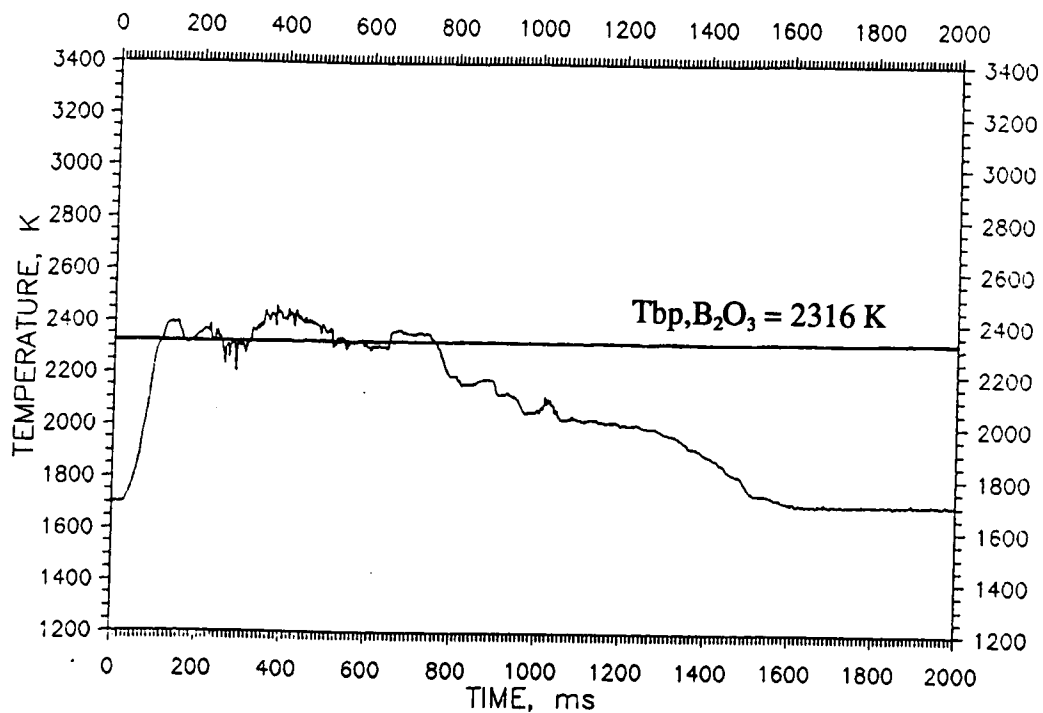
FIGURE 4.11 Typical Flame Structure in Regime III.

Stage III is the stage of most violent activity. During this stage very frequent particle ejections are observed, but the flame structure of these ejected particles is still the same as that observed in regime II (see photograph c taken at 262 ms after ignition). Radiation intensity curve marks these particle ejections as spikes of suddenly changing intensity as shown in Figure 4.10b. Surface temperature of boron agglomerate during this stage of violent activity, is in the vicinity of 2600 K (see Figure 4.10b). High surface temperature, supported by apparent swelling of the agglomerate during stage III of regime III, signifies that the melting of the boron agglomerate has occurred during this stage.

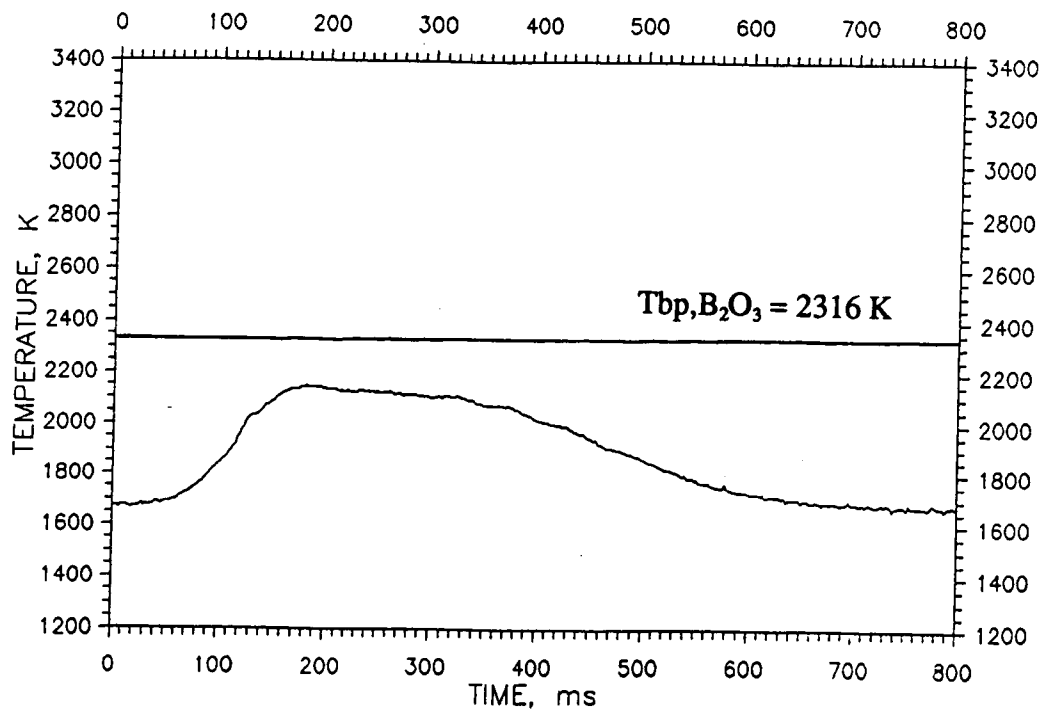
Stage IV follows stage III and during this stage the radiation intensity curve gradually decays to the initial value. Film records showed that during this stage, the agglomerate moves up the fiber and out of the view of the instruments. The upward movement of the agglomerate exposes the fiber resulting in fiber consumption. The difference in the total event time as recorded by the two-color pyrometer and the radiometer is due to the narrower field of view of pyrometer (see Figures 4.10a and 4.10b)

4.5 EFFECT OF OXYGEN MOLE FRACTION.

Although, majority of the experiments in this investigation were conducted in a quiescent $O_2/N_2(20/80)$ environment, a limited number of experiments were also performed at increased oxygen mole fractions to study the effect of oxygen mole fraction on burning time. Figure 4.12 shows a comparison between the surface temperature curves of boron agglomerates ignited in $O_2/N_2(30/70)$ and $O_2/N_2(20/80)$ environments respectively, while the pressure was kept constant at 5.1 atm. The effect of oxygen mole fraction on burning time is shown in Table 4.3.



(a)



(b)

FIGURE 4.12 Effect of Oxygen Mole Fraction On Surface Temperature:
(a) O_2/N_2 (30/70) (b) O_2/N_2 (20/80).

Results seem to indicate that at lower pressures increasing the oxygen mole fraction increases the surface temperature (see Figure 4.12a). It is also interesting to observe that by increasing the oxygen mole fraction, agglomerates which had previously burned partially, were consumed completely. The results also showed that the burning times obtained at an ambient pressure of 5.1 atm and oxygen mole fraction of 0.3 were approximately the same as those obtained at an ambient pressure of 6.8 atm and oxygen mole fraction of 0.2. These results suggest that there is a relationship between the oxygen mole fraction and the ambient pressure. Unfortunately due to fiber consumption at oxygen mole fractions greater than 0.4, this relationship could not be determined in this study.

It is possible that the explicit dependence of burning on pressure, observed in transition from regime I to II and then to regime III, is implicitly due to the increasing oxygen quantity with pressure. However, it should be realized that the results presented here are not enough to substantiate these propositions and more investigation will be required.

TABLE 4.3. Effect of Oxygen Mole Fraction on Burning Times.

Pressure (atm)	O ₂ /N ₂	Burning Times (ms)
3.4	40/60	1177 $\pm$ 125
5.1	30/60	1623 $\pm$ 165

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE STUDY

The objectives of this investigation were to study the effects of pressure on the ignition delay time, the burning time and the surface temperature of boron agglomerates, and to study the flame structure during ignition and combustion via high speed cinematography. Boron agglomerates having initial nominal diameters of 534 μm were ignited by a focused CO_2 laser pulse in a quiescent O_2/N_2 (20/80) environment. A two-color pyrometer and a broadband radiometer were used to record the surface temperature and the radiation emitted by the agglomerate during ignition and combustion respectively.

Although, ignition in the present investigation was defined to occur at the first appearance of flame as evidenced by film records, extremely consistent ignition delay times (the time elapsed between the first exposure of laser and the appearance of flame) at all pressures investigated were obtained. This eliminated a need for capturing every experiment on film and hence burning times in this investigation were determined solely from the temporal outputs of two-color pyrometer and broadband radiometer.

In the range of pressure investigated in this study, three distinct combustion regimes were observed. In regime I, partial or incomplete burning occurred. In regime II, steady-state full-fledged combustion was observed in which the burning time decreases with increasing ambient pressure. On the other hand, violent combustion accompanied by frequent particle ejections characterized regime III. The surface temperature of the burning boron agglomerates in regime I is consistently below the boiling temperature of boron oxide (2316 K), which causes the oxide production rate to exceed the oxide removal rate, resulting in incomplete combustion. In regime II, the agglomerate surface temperature is in the vicinity of 2400 K, whereas, the surface temperature is above 2400 K in regime III.

Due to very high surface temperatures achieved and corresponding fragmentation and shattering of boron agglomerates in regime III, it appears almost certain that the agglomerate melting does occur at higher ambient pressures.

Qualitative studies of flame structure from high-speed cinematography indicated that the flame surrounding the boron agglomerate at all pressures consists of two distinct zones: a luminous inner zone surrounded at its edge by a green envelope (BO_2). The size and luminosity of the inner zone increases with increasing pressure, most probably because of an increase in the generation of excited species (besides BO_2) at higher surface temperatures achieved at higher pressures. Pressure sensitivity of boric oxide (BO_2) imparts significance to its inclusion in the modeling of boron agglomerates combustion, as an intermediate product. In regime I the flame is contiguous to the agglomerate surface, whereas flame diameter is much larger in regimes II and III. The generation of brownish colored smoke also appeared to be pressure dependent. In regime I, smoke was produced in excessive quantities and on several occasions it surrounded the whole agglomerate, especially in the beginning and towards the end of the experiment, thus partially obscuring the view. Smoke production was greatly reduced in regime II, although its color still remained brown. In regime III, very little smoke was observed due to agglomerate fragmentation and particle ejections.

In this investigation it was observed that at high pressures (regime III), combustion becomes very violent and fragmentation or shattering of agglomerate occurs. This fragmentation of agglomerates is very desirable from practical point of view, because it would enhance the burning rates and thereby reduce the residence time of agglomerates in the combustor. It is therefore recommended that this line of investigation be further pursued to determine efficient and controllable ways of causing agglomerate fragmentation.

Limited investigation of the effect of oxygen mole fraction on burning time showed, that increasing the oxygen mole fraction has the same effect as that of increasing pressure; i.e., decrease burning time. However, exact relationship between the pressure and oxygen mole fraction was not determined. Due to fiber consumption, experiments could not be performed at oxygen mole fractions greater than 0.4. It is recommended that a more extensive investigation towards studying the effect of oxygen mole fraction on burning time be carried out.

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