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Abstract

The prospects for using Mg as fuel in a CO_2 breathing engine in Mars atmosphere without oxygen lead to this experimental study on the ignition and combustion of Mg performed in pure CO_2 and CO_2/CO streams over a wide range of pressure, velocity and CO concentration of the streams. The critical ignition temperature of Mg in CO_2 streams decreased with decreasing the ambient pressure, and was insensitive to the stream velocity. The ignition temperature in CO_2/CO streams varied little at first, but increased with decreasing CO_2 concentration. Ignition occurred in two distinct stages, first being surface reactions controlled by chemical kinetics and second being gas-phase reactions controlled by CO_2 diffusion. The breaking of a thin protective film formed in the first stage played a crucial role in the ignition process, leading to the beginning of the gas-phase reactions and then to ignition.

During combustion, several flames appeared sporadically and intermittently over the swelling sample surface which was coated with a thick porous layer. The flames produced CO in the gas phase, while on the non-flame surface the reactions of Mg with both CO diffusing from the flames and CO_2/CO in the streams generated condensed MgO and C that built the porous layer. The pulsating manner of the flames can be explained in terms of the abrupt breaking of the layer followed by its blockage due to the surface reaction with CO.

Introduction

We have proposed a CO_2-breathing jet engine using metals as fuel for a future aircraft type vehicle in Mars atmosphere, consisting of CO_2.[1,2] One of the attractive metal fuels for the engine is Mg. We have performed an experimental study on the ignition and combustion of Mg disks in a CO_2 stream at 1 atm.[1] We found that Mg had easy ignitability and fast burning rate. During combustion intermittent flames appeared sporadically over the Mg surface to produce CO by gas-phase reactions. Recently, Shafirovich and Goldshleger[3] have made an experimental study on the combustion of Mg particles introduced into high temperature CO_2/CO mixtures at 1 atm. They concluded that the heterogeneous reaction of Mg with CO occurred during combustion in CO_2, in addition to the homogeneous reaction of Mg with CO_2. However,detailed experimental knowledge of the ignition and combustion processes, such as the interactions between the homogeneous and heterogeneous reactions and the surface reaction layer structure, has been insufficient. Thus the ignition and combustion mechanism still remain obscure. In particular, since the atmospheric pressure of Mars is about 0.6 kPa[4], information about the ignition and combustion of Mg at low pressures will be required to develop a combustor of the engine.

The present experimental study was made to investigate and detail the ignition and combustion process of Mg and to reveal the effects of pressure on it. The experiment was carried out in a stagnation region of CO_2 streams, in the same manner as that of our previous studies for various metals.[1,5] In order to help understand the ignition and combustion mechanism in CO_2, CO_2/CO mixture streams were also used as oxidizers.

Experimental Apparatus and Procedure

Figure 1 shows a schematic of the experimental apparatus.[5] CO_2/CO mixtures at room temperature issued through a nozzle (25 mm diameter) into a combustion chamber. The mixtures impinged on a sample holder of BN (20 mm in diameter), which was situated 17 mm downstream of the nozzle exit. A machined Mg sample (9.5 mm in diameter, 5 mm in height, purity: 99.9 %) was mounted in a depression of the holder through a thin exchangeable receptacle and a crucible, both of which were made of stainless steel.

In CO_2/CO mixture streams, the experiments were performed at combustion chamber pressures P_m from 8 to 101 kPa, and at the nozzle exit velocities V_m from 0.5 to 5 m/s with CO_2 mole fractions X_{CO2} varying from 0 to 1. Before each test, the Mg sample was heated in an Ar stream at a predetermined pressure and flow rate. When the sample temperature reached a predetermined value, the test run was started by changing Ar to a CO_2/CO mixture of the same pressure.

The sample temperature was measured by a chromel-alumel thermocouple. Changes in sample appearances were continuously recorded by two video cameras as shown in Fig.1. Microphotographic observation was made of samples that were quenched during the ignition and combustion process by injecting Ar in the chamber. Condensed combustion products gathered after the experiment were analyzed chemically and by an X-ray diffractometer.

Experimental Results

Critical Ignition Temperature:

In this study, ignition was defined as a first appearance of a flame on the sample surface. The ignition temperature T_{ig} was the sample temperature at ignition. To determine an ignition limit, the ignition delay time t_d, the interval to ignition after the gas replacement of Ar by a CO_2 stream had take place, was measured as a function of the initial sample temperature T_{in} defined as temperature at the gas replacement. Regarding the CO_2 streams was found that at higher pressures t_d hardly varied with decreasing T_{in} (3-10 sec), while at lower pressures t_d increased slightly with decreasing T_{in} (up to about 50 sec). When T_{in} reached a critical value, ignition did not occur even after an interval of ten minutes. The critical ignition temperature T_{cr} was defined as the lowest T_{in} above which a sample heated up by itself to ignition.

Figure 2-1 shows the variation of T_{cr} with P_m in CO_2 streams at V_m=1.5 m/s. T_{cr} decreased with a decrease in P_m. This tendency was different from that in O_2 and air atmospheres.[6,7] Even at the lowest pressure, T_{cr} in CO_2 was higher than that in air of 1 atm.[6-8] Mg in CO_2 streams, as well as in O_2 and air atmospheres,[9,10] could burn at sufficiently low pressures. Figure 2-2 shows the effect of V_m on the T_{cr} at P_m=41.3 kPa in CO_2. T_{cr} varied little with V_m. This result suggests that the ignition process of Mg in CO_2 is not controlled by CO_2 diffusion through the boundary layer over the sample surface, but by chemical kinetics. It also suggests that a heat loss variation with V_m has only a slight influence on T_{cr}. Figure 2-3 shows T_{cr} plotted against X_{CO2} at P_m=21.3 kPa and V_m=1.5 m/s. T_{cr} hardly varied when X_{CO2} decreased from 1.0 to 0.5, but rapidly increased with decreasing X_{CO2} from 0.5 to 0.1. Below X_{CO2}=0.1, the sample could not ignite even at the boiling point of Mg (937 °C[11]) at P_m=21.3 kPa. The dependency of T_{cr} on X_{CO2} was substantially the same over the P_m range tested, and when X_{CO2} remained constant, T_{cr} decreased with decreasing P_m.

Ignition and Combustion Process:

i) Sample appearance and temperature behavior

Figure 3 shows a typical example of the time variation of the sample temperature after the gas replacement at P_m=41.3 kPa. The sample appearances corresponding to (a)-(d) are also shown in the figure. In an Ar stream, the Mg surface was clean and actively ejected Mg vapor. As soon as the sample was exposed to the CO_2 stream, the surface was uniformly covered with a reaction film which stopped the vapor ejection. Simultaneously, the sample temperature began to increase and the surface as a whole became red hot at about (a). As time proceeded, many "bubbles" appeared over the surface at about (b). They further increased size and number with a corresponding rapid increase in the sample temperature at "transition", where the heat release mechanism drastically changed. Finally, when one or more of "bubbles" burst at (c), incandescent flamelets appeared locally, indicating ignition at T_{ig}=888 °C. However, when T_{in} was lower than T_{cr}, the temperature increased just after the gas replacement, but this was not followed by the appearance of "bubbles" and the sample temperature decreased with time, resulting in non-ignition.

The first flamelets at ignition did not spread over the whole sample surface and quenched quickly (about 0.1 sec). However, many flamelets continued to appear sporadically and intermittently over the surface with "bubbles", repeating the process from ignition to quenching. When the frequency of the appearance increased substantially, stable combustion of the sample was attained at about (d). This behavior is different from that in the air stream, in which an one-dimensional flame appeared over the Mg sample surface without a reaction film.[8] Figure 3 shows that during stable combustion the sample temperature remained almost constant at a value close to the boiling point of Mg (1003 °C[11]) at the ambient pressure, in agreement with results of Mg in the air stream[8]. Figure 4 shows a sequence of the developing process of a typical intermittent flamelet which formed during combustion at P_m=41.3 kPa. Figure 4-(a) represents a red hot "bubble" which appeared near the top of the surface. As soon as the "bubble" began to rapidly increase in luminosity, it burst to eject white smoke into the open space, generating an incandescent flamelet like a jet diffusion flame (Fig.4-(b) and (c)). After the flamelet had continued to burn for a while, it was quenched just after (d). The corresponding spectrum of the flamelets showed that Mg lines and MgO bands were visible, in agreement with observations of Mg in O_2 and air atmospheres[8,10]. CO was measured in the gaseous combustion products. It is of interest that the non-flamelet surface area continued to be heated red hot during combustion. These results indicate that gas-phase reactions of the flamelets produced CO, while surface reactions also occurred on the non-flamelet surface areas. After the stable combustion continued for a few minutes, flamelets vanished and the surface with "bubbles" became dark, resulting in quenching of a whole sample. A thick black and white shell remained on a quenched sample.

ii) Effects of pressure and velocity

The above mentioned ignition and combustion process in pure CO_2 streams was found to be essentially similar over the range of pressure tested except for the case at P_m=8 kPa. However, pressure has effects on the following characteristics. T_{ig} scattered in the range of 870-920°C seems to decrease slightly with decreasing pressure. The averaged heat release rate q was estimated from the temperature increase rate of the sample between "transition" and stable combustion by assuming that the sample mass and the reaction surface area were kept constant at original values. q was about 3-12 J/(cm^2·s), and decreased with decreasing pressure from

101 to 21.3 kPa as shown in Fig.5-(1). Corresponding to this, the total burning time from ignition to quenching increased greatly with pressure; about 50-80 sec for 101 kPa and about 100-160 sec for 21.3 kPa. *q* also increased with increasing V_m as shown in Fig.5-(2), which implies that after ignition CO_2 diffusion through the boundary layer became the rate limiting step. With a decrease in pressure, the frequency of flamelet appearance decreased, but the scale of "bubbles" and the height of the swelling surface increased. Individual flamelets continued to burn for about 0.1-0.2 sec, and this was observed to be insensitive to the ambient pressure. At P_m=8 kPa, ignition occurred when a surface film burst extensively without generating "bubbles" and surface swelling, resulting in a jet diffusion flames.

iii) Effect of CO concentration

Addition of CO to CO_2 led to noticeable changes in the ignition and combustion process. Decreasing X_{CO2} from 1.0 to 0.25 caused the "bubbles" to become bigger, and the sample swelled significantly higher. In the range of X_{CO2} below 0.5, t_d at the ignition limits became much longer than that in CO_2. At lower X_{CO2}, individual flamelets became to burn slowly and weakly for a few seconds. Corresponding to this, *q* was smaller than that in CO_2 streams. The total burning time of the whole sample remarkably increased with decreasing X_{CO2}; for example at P_m=21.3 kPa and V_m=1.5 m/s, about 100-160 sec at X_{CO2}=1.0 and about 170-300 sec at X_{CO2}=0.5. These results suggest that the reaction rate of Mg with CO is considerably slower than that with CO_2. It is noteworthy that pulsating combustion of the whole sample, such as that reported by Shafirovich and Goldshleger,[3] was observed over the wide range of X_{CO2} from 1.0 to 0.5 in the present experiment.

When X_{CO2} was less than 0.25, the behavior of the sample appearance changed drastically. Whenever leading to ignition at lower pressures, an abrupt bursting occurred just after gas replacement, and was followed by a large jet flame of Mg vapor without any flamelets.

Combustion Products and Reaction Film Structure:

Figure 6 shows a typical example of the cross sections of quenched samples at certain intervals after ignition at 101 kPa in a CO_2 stream. During the ignition stage, a thin gray reaction film adhering to the Mg surface formed, having about 0.1 mm in thickness (Fig.6-(a)). At the next stage, a black product layer was generated over the thin film. It increased in thickness with time, while the thin film maintained an almost constant thickness. As time went on, the unevenness and thickness of the black product layer increased and many protruding parts, like "teeth," filled with Mg appeared on the rough surface (Fig.6-(b)). It should be noted that their bottoms attached to the thin gray film and the black layer was covered with white powder products in spots. All the samples quenched at this stage had cavities inside. They became larger in diameter with proceeding combustion. This observation supports the fact that the sample was boiling during combustion. As combustion continued, the black product layer became more complicated and much thicker. Eventually several stratified protrusions like "cabbages" formed (Fig.6-(c)). They consisted of alternatively arranged thin metallic layers and thin gray layers, and had paths from the Mg sample. The layer number of a stratified protrusion was nearly equal to the pulse number of the corresponding flamelet. This suggests that the protrusions were alternatively stratified by Mg and combustion products. The growing of the black product layer indicates that it was appreciably porous and non-protective, thus allowing further gas-phase reactions occurring close to or inside it. After combustion, the black product layer existed almost entirely clear of white products, remaining as a shell covering the receptacle.

When the ambient pressure was lower, the layer structure was essentially the same as that at high pressures. The differences were that the initial thin film decreased in thickness, and that the black product layer, including stratified protrusions, became more complicated and much thicker, for example, up to about 5 mm at P_m=41.3 kPa. On the other hand, adding CO in the CO_2 streams made the layer both more complicated and thicker. In the quenched layers, stratified protrusions were not observed, but Mg metal existed in a coral formation.

After the test run, the shell of the black product layer was analyzed chemically. It was confirmed that the shell was composed of combustion products, MgO and carbon, and of unburned Mg. Mole fraction of carbon in the combustion products, $X_C=mol_C/(mol_C+mol_{MgO})$, was about 0.15 in CO_2 streams. One possible explanation for the carbon formation is surface reactions, as the gas-phase reactions between Mg vapor and CO_2 produce only CO.[1] Figure 7 shows the variation of X_C with X_{CO2} in CO_2/CO streams at P_m=21.3 kPa. X_C increased with increasing CO concentration. This tendency of X_C on X_{CO2} remained similar over the range P_m tested. The ST1 curve represents the stoichiometric values for the case when only CO in the mixtures of CO_2/CO is perfectly reduced to C through the heterogeneous reaction $Mg(l)+CO=MgO(c)+C(c)$. The ST2 curve represents the case in which all CO and CO_2 in the mixtures are stoichiometrically converted to C through the heterogeneous reactions $Mg(l)+CO_2/2=MgO+C/2$ and the above reaction. The difference between the experimental values and the ST1 curve is considered to be equal to the amount of carbon formed from the reaction with CO_2. This consideration may imply that below approximately $X_{CO2}=0.5$, CO_2 in the mixtures did not take part in the surface reactions to produce carbon. In addition, the amount of carbon corresponding to the difference between ST2 and the experimental values was probably generated as CO without conversion to carbon.

Discussion

Reaction Kinetics:

A proper understanding of the Mg-CO_2/CO reactions will be necessary to make the ignition and combustion process of Mg clear. Possible overall kinetics between Mg and CO_2/CO are as follows:

Reaction 1: $Mg+CO_2 = MgO+CO$ $\Delta H=-318.9$ [kJ],

Reaction 2: $Mg+CO_2/2= MgO+C/2$ $\Delta H=-404.7$ [kJ],

Reaction 3: $Mg+CO = MgO+C$ $\Delta H=-490.9$ [kJ].

Figure 8 plots the standard Gibbs free energy change against temperature for Reactions 1-3 at 101 kPa. It is known that if the value is negative the reaction can proceed spontaneously, and that the lower the position of the line the more stable the product is. At the flame temperature of Mg with CO_2 at stoichiometric ratio (2952 K[1]), only Reaction 1 can take place, but Reactions 2 and 3 can not. However, below about 2100 K, close to the flame temperature of Mg with CO (2040 K[12]), CO can react with Mg vapor to form carbon. However, the present experimental results related to T_{cr}, the combustion products and the burning time suggest that the reaction rate of CO with Mg was not as fast as that of CO_2. It is interesting to note that on the burning Mg surface at about 1000 °C Reaction 2 and 3 can occur, producing carbon.

Ignition and Combustion Process:

From the present experimental results and the above considerations, we postulated an ignition and combustion mechanism of Mg in

CO_2 as follows. As soon as CO_2 is impinged on a clean Mg surface, Reaction 1 occurs in the gas phase to form CO. Part of the CO can diffuse back to the surface to produce MgO and C on it because the rate of Reaction 3 is slow in the gas phase. These products form a somewhat protective initial film, inhibiting further gas-phase oxidation. Since the rate of the surface reaction through the film is considered to be much slower than gas diffusion, a kinetic process may control the early ignition stage. As time goes on, at "transition," the film will become non-protective and allow passing of liquid or gaseous Mg. This may be due to be significantly high pressure of Mg. Since the film is not perfectly broken, Reaction 1 and 3 occur only close to the film surface. Thus the reactions look as if they are pure surface reactions, and produce a porous layer, composed of MgO and C, filled with Mg. When the liquid Mg vaporizes vigorously inside the layer due to heat caused by the reactions, the unevenness of the layer increases, in particular it grows into "bubbles." If the vapor pressure in a bubble is increased by local heating, the vapor will be ejected into the open space through a hole to form a luminous jet diffusion flame, leading to ignition.

Figure 9 explains schematically the burning of Mg in CO_2. As the vapor pressure of Mg in the "bubble" decreases, part of CO produced in the flamelet can diffuse back again to clog its hole with reaction products MgO and C, resulting in quenching. However, at the other non-flamelet parts of the surface, the gas-phase and surface reactions continue to occur in the vacant space near the outer edge of the layer. This is followed by the occurrence of next flamelets over the surface, leading to the appearance of intermittent and sporadic flamelets. If a flame forms intermittently at the same place, the layer stratifies and the flame appears as pulsating combustion. During combustion, consequently,

CO_2 diffusion through the boundary layer over the sample surface becomes a rate limiting step because it is much slower than the gas-phase reactions.

Effects of Ambient Pressure and Impinging Velocity:

We proposed that the rate limiting step is changed from chemical kinetics to CO_2 diffusion in the ignition process. The chemically controlled mechanism, supported by the result of Fig.2-(2), leads to the following explanation of the dependency of T_{cr} on P_m. It is known that the oxidation of metals without significant scale cracking proceeds at rates proportional to the (1/n)th power of the oxidizer pressure.[13] Generally, n is positive. Thus the lower the ambient pressure is decreased, the thinner is the film that forms. This tendency is confirmed by our present observation. If "transition" from a protective film to a porous one occurs by cracking of the film due to the pressure difference between the external ambient pressure and the internal vapor pressure, a thinner film is more likely to break, and to begin the gas-phase reactions. This consideration could explain how a decrease of P_m facilitates ignition at lower T_{in}. In this case, the heat loss dependent on P_m has only a minor effect because its order is the same as that of V_m shown in Fig.2-(2).

On the other hand, increasing V_m and P_m causes an increase in mass transfer in the stagnation region due to a decrease in thickness of the boundary layer over the surface. In the diffusion controlled regime, the decrease effects an increase in the burning rate of Mg, resulting in higher *q* as shown in Fig.5

Effect of CO Concentration:

The experimental fact concerning to the slow gas-phase reaction rate of Mg with CO may allow a preferential reaction of CO_2

with Mg. Thus the ignition process, controlled by chemical kinetics, should be essentially insensitive to CO concentration. This is supported by the independency of T_{cr} on X_{CO2} in the range from 1.0 to 0.5. Of course, during combustion the increased CO concentration leads to the decreased Mg vapor ejection from the surface due to the additional surface reaction with CO, generating a thicker surface layer. Its growth mechanism is a subject that merits further studies. On the other hand, at higher CO concentrations, probably below $X_{CO2}=0.5$, the mass flow rate of CO_2 to the surface decreases substantially. Thus, the gas-phase reactions close to the surface occur inactively, resulting in a higher ignition temperature.

It is interesting to note that when X_{CO2} was in the range 1.0 to 0.5, the whole sample frequently burned in a pulsating manner. In pure CO, however, the sample could not ignite. These results differed from Shafirovich and Goldshelger's experimental observations[3], carried out in CO_2/CO mixtures at the temperature of 1000 K and 1 atm. This contradiction seems to be attributed to the difference of the ambient temperatures. However, our results propose that the pulsating combustion is a fundamental phenomenon attendant on the system in which the gas-phase reactions occur simultaneously with the surface reactions producing condensed product layer.

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Captions

Fig.1. Schematic of experimental apparatus.

Fig.2. Variation of critical ignition temperature: (1) with ambient pressure in pure CO_2 streams at Vm=1.5 m/s, (2) with CO_2 stream velocity at P_m=41.3 kPa, (3) with CO concentration in CO_2/CO streams at 21.3 kPa and V_m=1.5 m/s.

Fig.3. Time variation of sample temperature and video photographs in a CO_2 stream. T_{in}=873 C, P_m=41.3 kPa, V_m=1.5 m/s.

Fig.4. Video photographs of a flame developing process in a CO_2 stream. P_m=41.3 kPa, V_m=1.5 m/s.

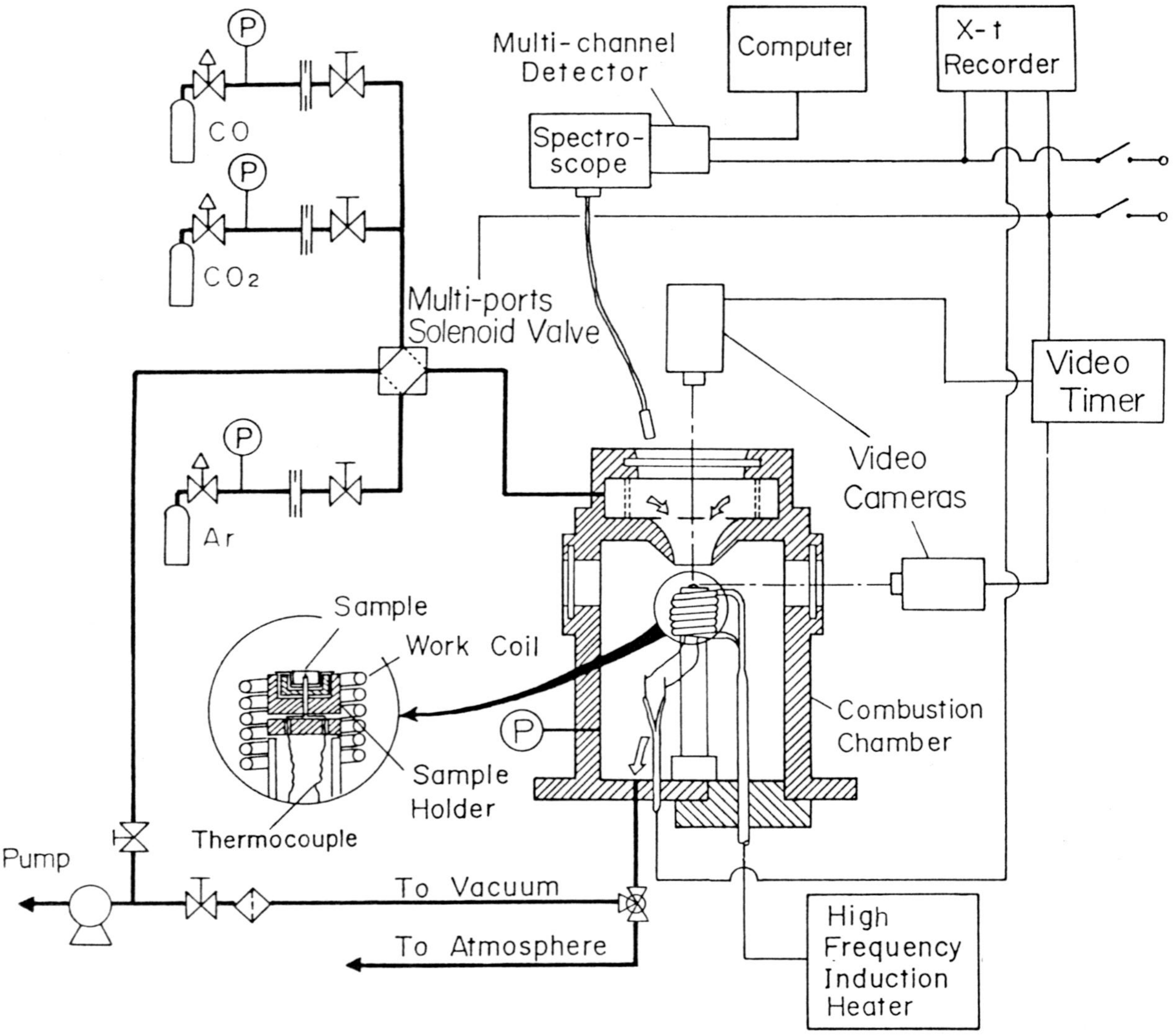
Fig.5. Variation of heat release rate in CO_2 streams: (1) with ambient pressure at V_m=1.5 m/s, (2) with stream velocity at P_m=41.3 kPa.

Fig.6. Cross section photographs of quenched samples in a CO_2 stream at P_m=101.3 kPa and V_m=1.5 m/s: (a) after ignition t=2.3 s, (b) t=9.4 s, (c) t=46.3 s. T_{in}=900 C.

Fig.7. Carbon mole fraction in combustion products against CO_2 mole fraction in CO_2/CO streams at P_m=21.3 kPa and V_m=1.5 m/s.

Fig.8. Standard free energy change as a function of temperature.

Fig.9. Schematic representation of a Mg-CO_2 combustion mechanism postulated in this study.


P
CO
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CO2
Multi-channel
Detector
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X-t
Recorder
Spectro-
scope
Multi-ports
Solenoid Valve
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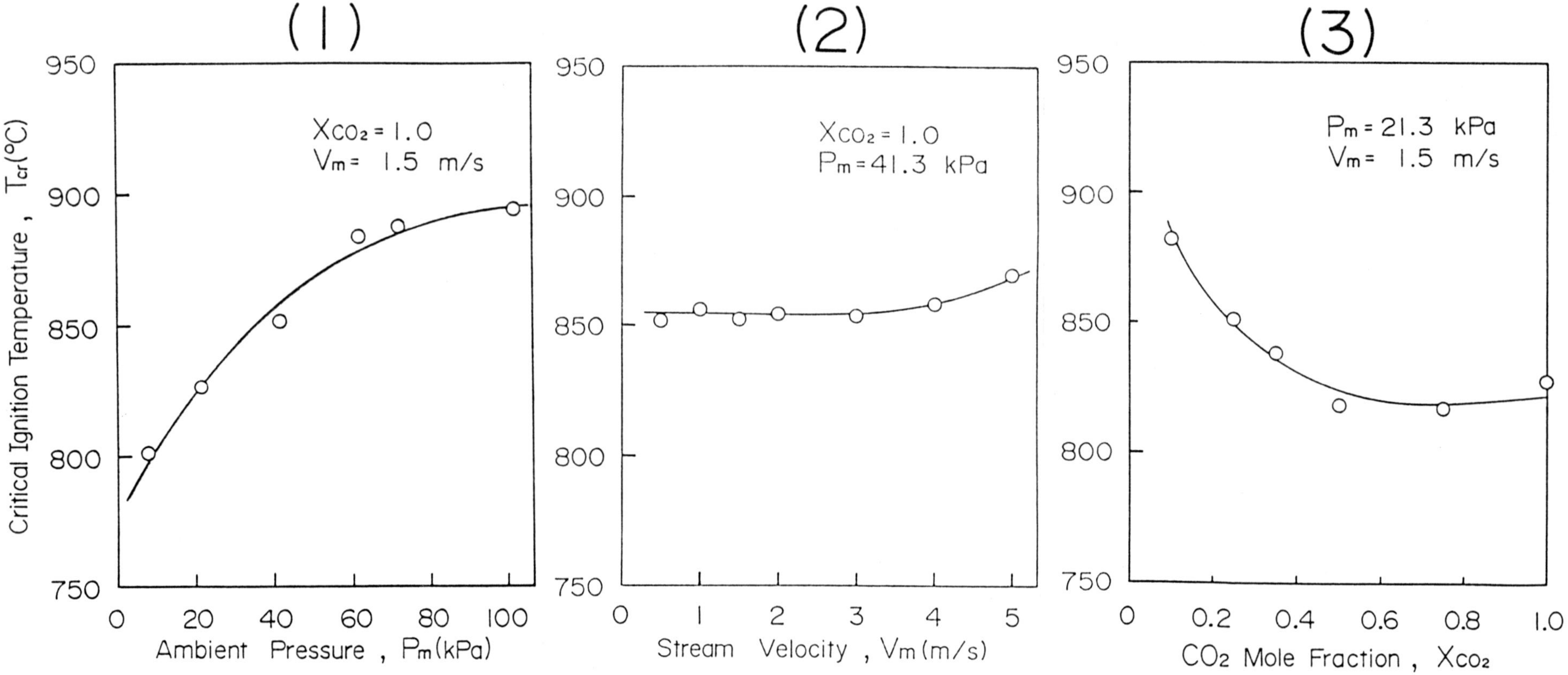
Video
Timer
Video
Cameras
Sample
Work Coil
Sample
Holder
Thermocouple
P
Combustion
Chamber
Pump
To Vacuum
To Atmosphere
High
Frequency
Induction
Heater

Fig.1. Schematic of experimental apparatus.



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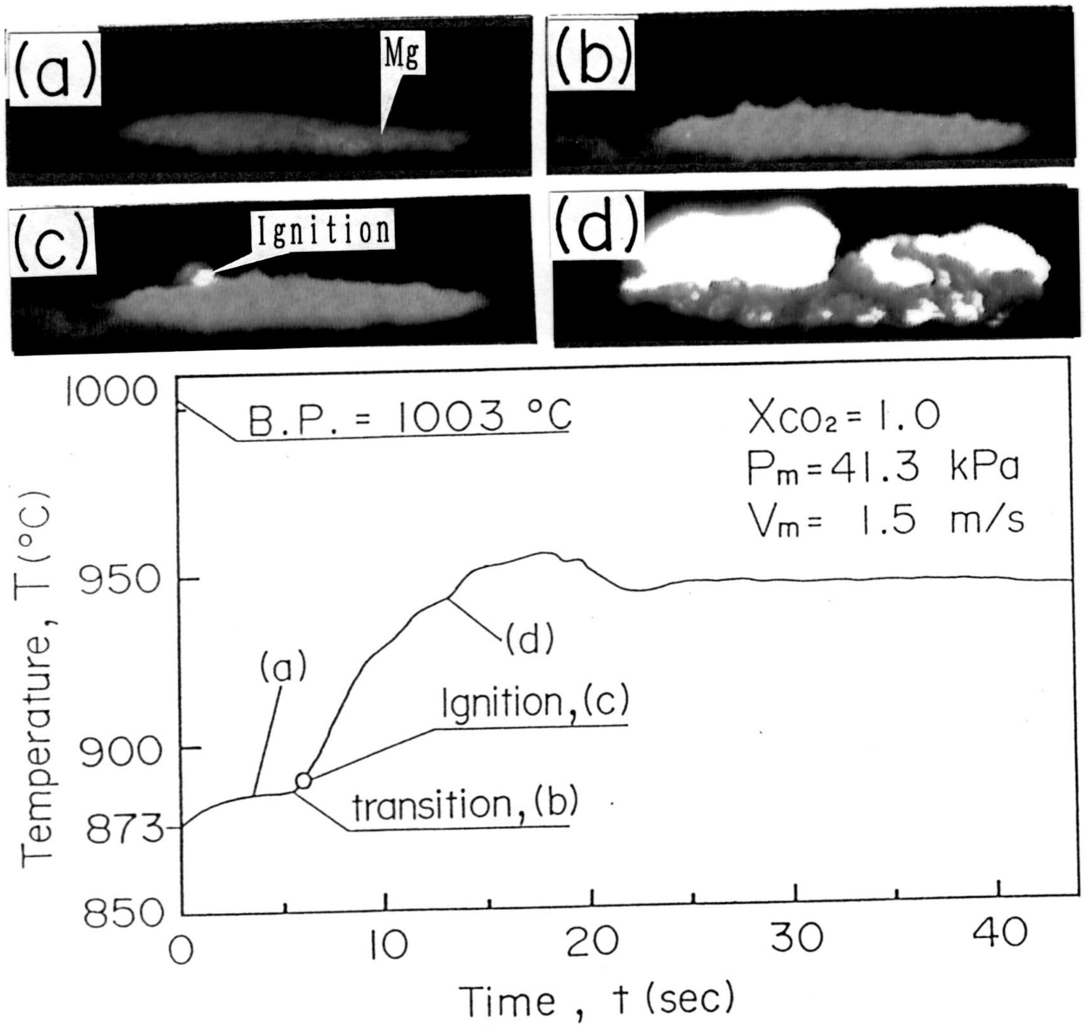

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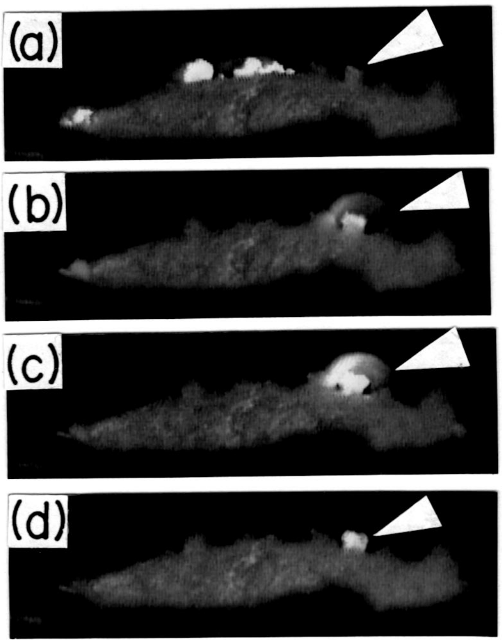


after the gas replacement

8.48 sec

8.54 sec

8.56 sec

8.68 sec

Fig.4. Video photographs of a flame developing process in a CO_2 stream. P_m=41.3 kPa. V_m=1.5 m/s.

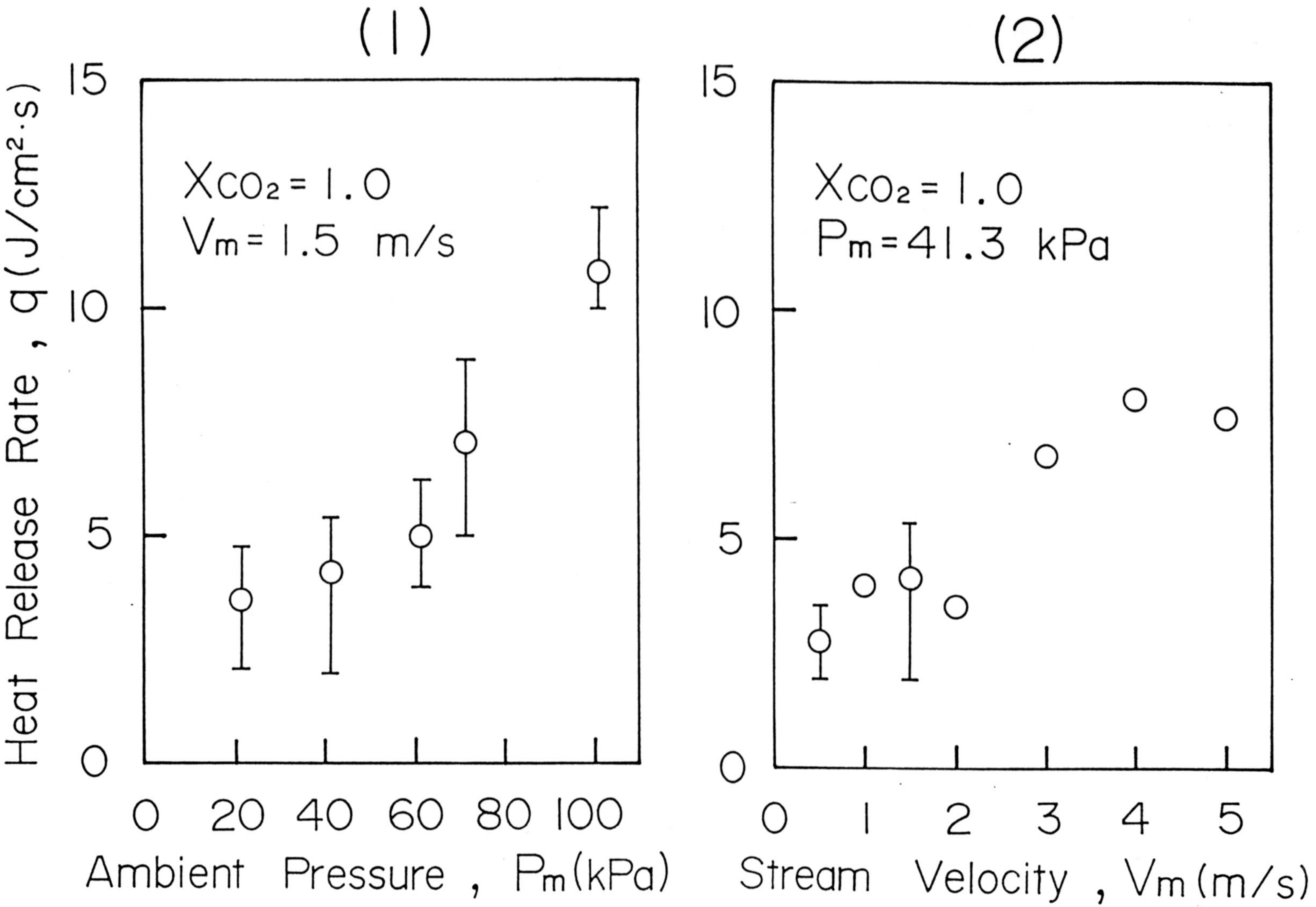


Fig.5. Variation of heat release rate in CO_2 streams:
(1) with ambient pressure at V_m=1.5 m/s,
(2) with stream velocity at P_m=41.3 kPa.

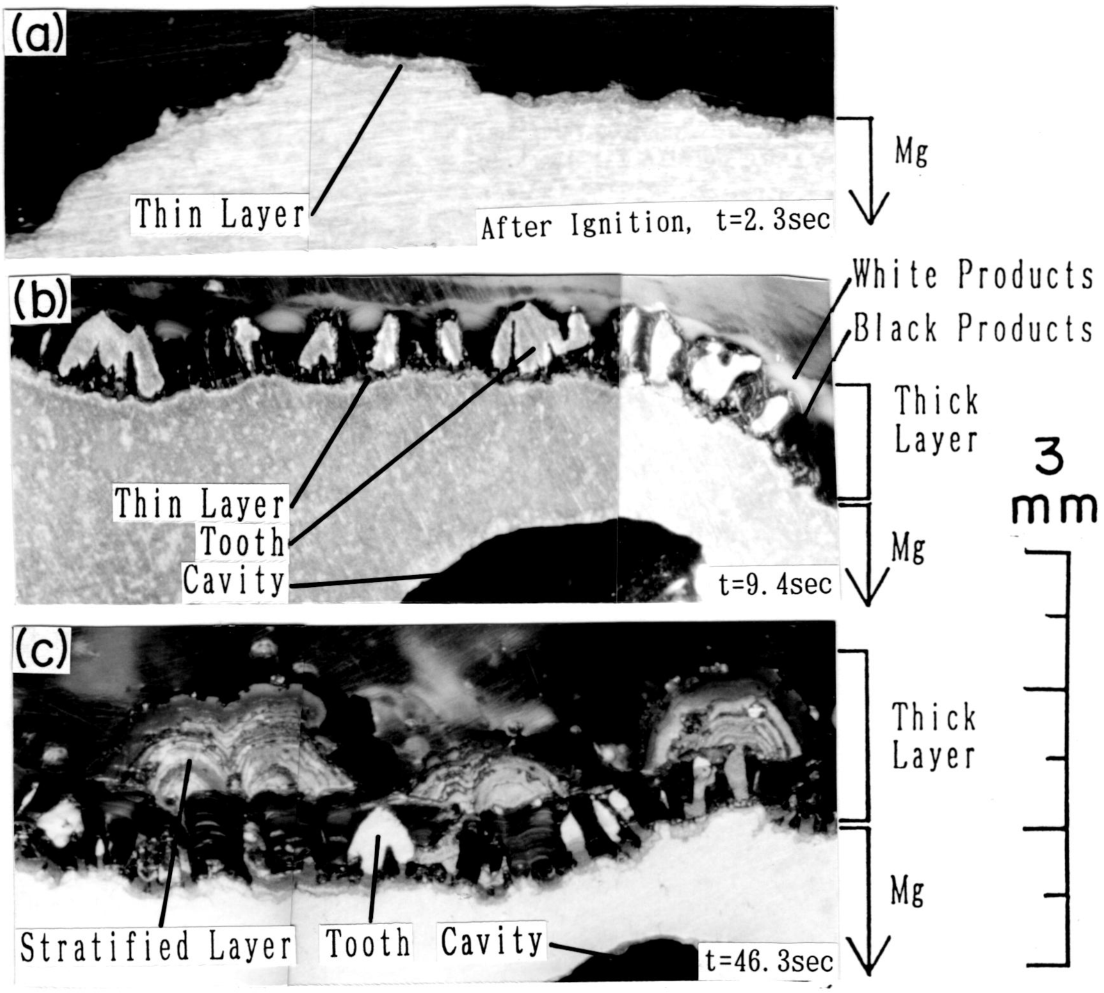
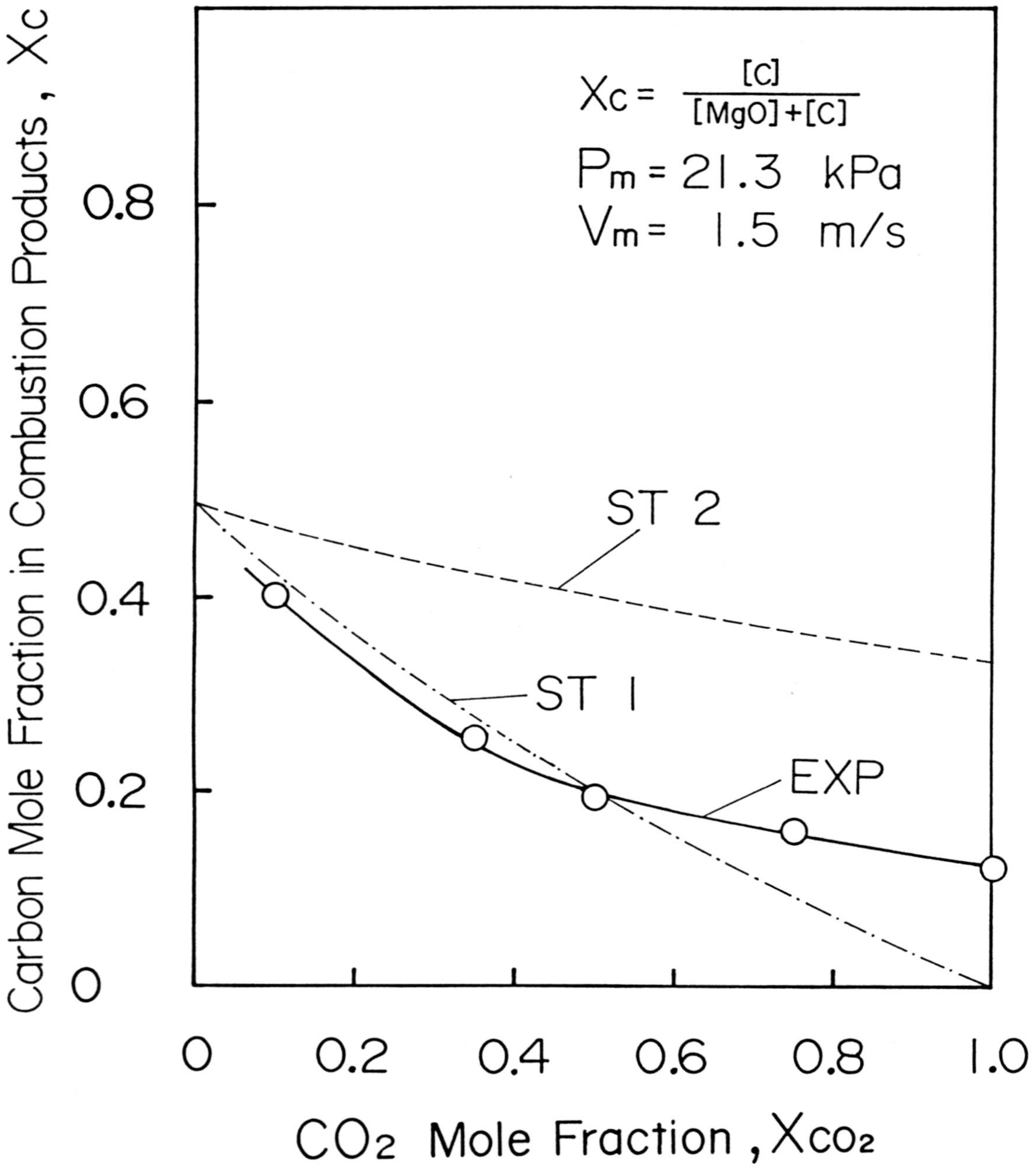

(a)
Thin Layer
After Ignition, t=2.3sec
Mg
(b)
White Products
Black Products
Thick Layer
Thin Layer
Tooth
Cavity
Mg
t=9.4sec
3 mm
(c)
Thick Layer
Mg
Stratified Layer
Tooth
Cavity
t=46.3sec

Fig.6. Cross section photographs of quenched samples in a CO_2 stream at P_m=101.3 kPa and V_m=1.5 m/s: (a) after ignition t=2.3 s, (b) t=9.4 s, (c) t=46.3 s. T_{in}=900 C.



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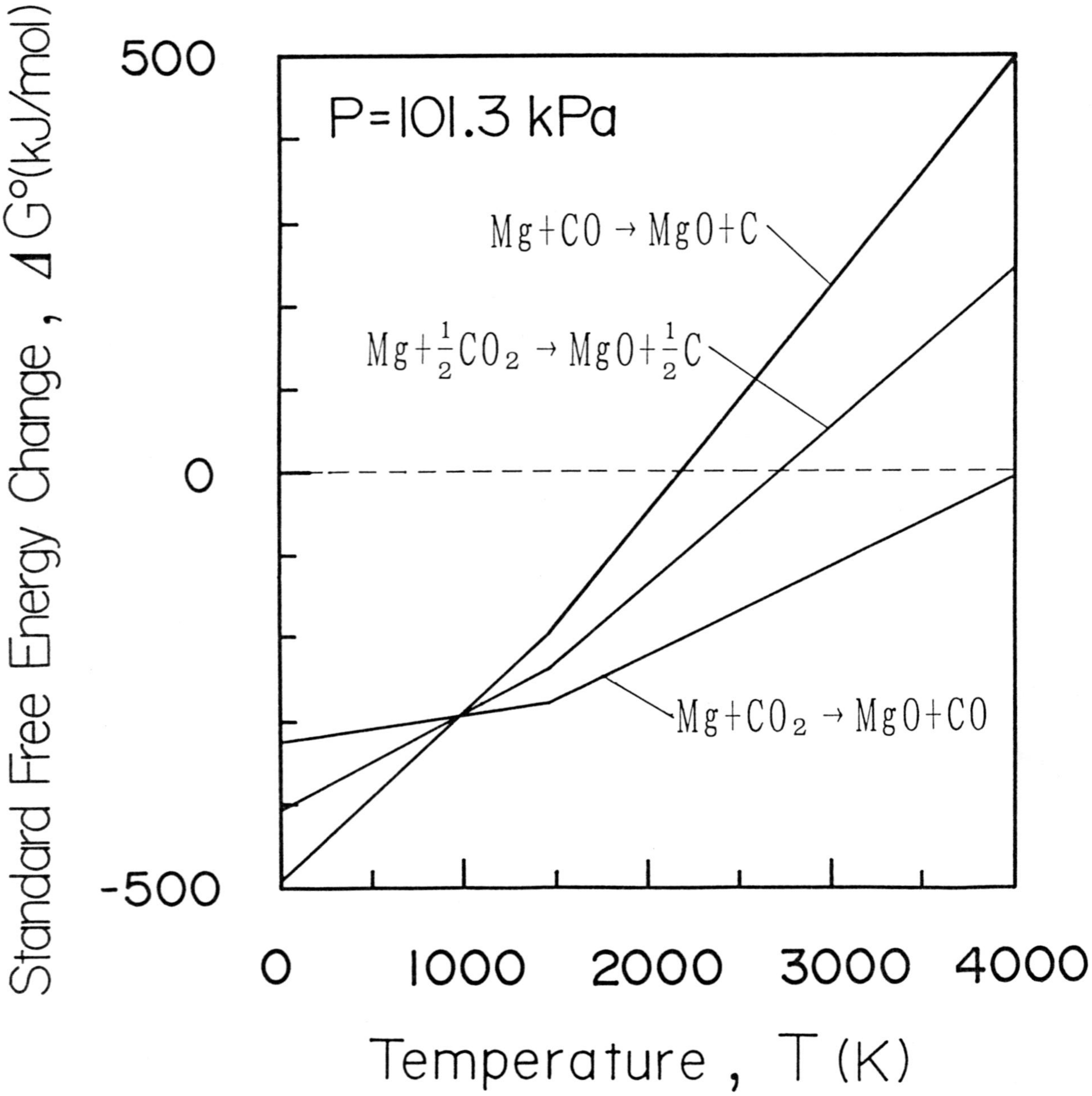

Fig.8. Standard free energy change as a function of temperature.

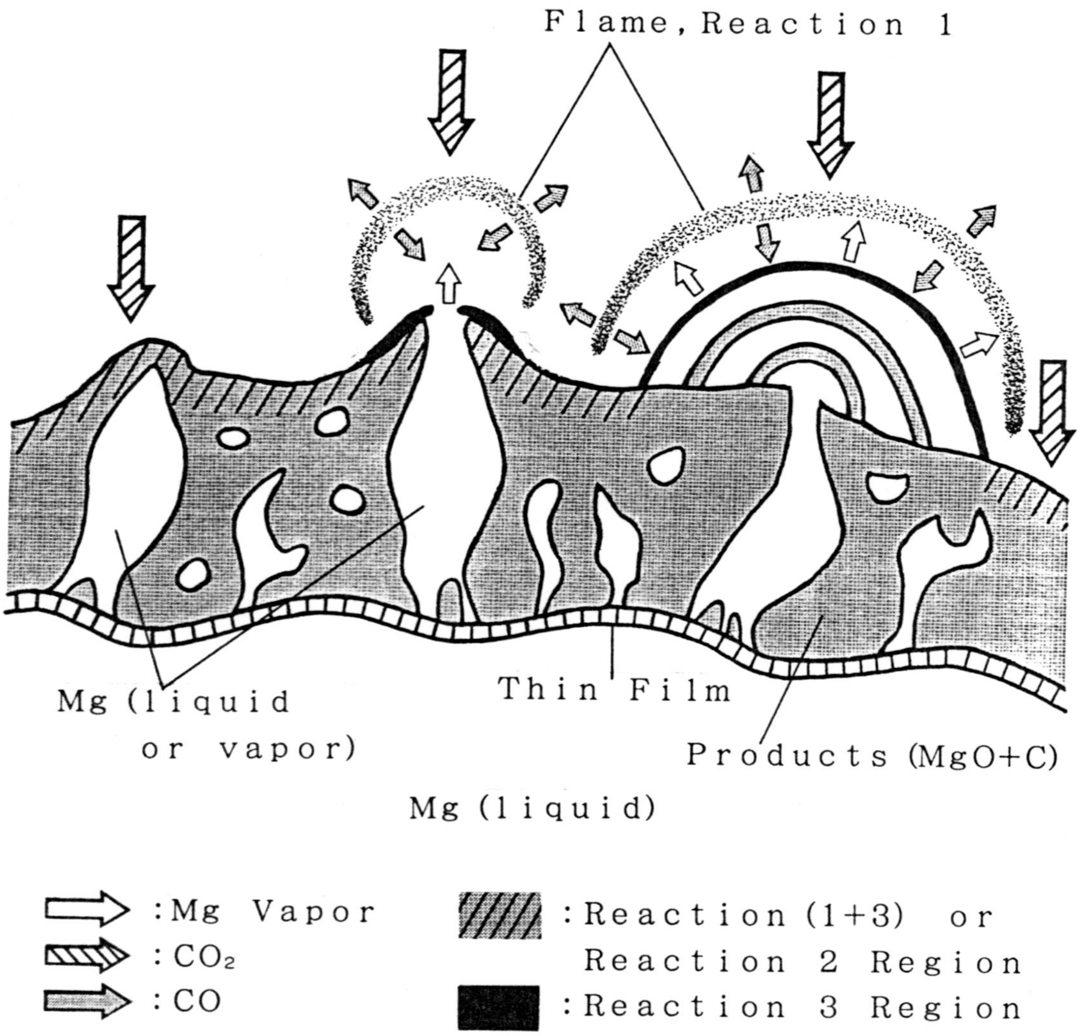
Flame, Reaction 1
Mg (liquid or vapor)
Thin Film
Products (MgO+C)
Mg (liquid)
:Mg Vapor
:CO_2
:CO
:Reaction (1+3) or Reaction 2 Region
:Reaction 3 Region

Fig.9. Schematic representation of a Mg-CO_2 combustion mechanism postulated in this study.