AN EXPERIMENTAL INVESTIGATION
RELATING TO THE COMBUSTION MECHANISM
OF AMMONIUM PERCHLORATE COMPOSITE PROPELLANTS

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RELATING TO THE COMBUSTION MECHANISM OF
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LIST OF SYMBOLS

A  area
AP  ammonium perchlorate
B  dimensionless burning rate for Emmons problem
C  flow coefficient for orifice
CC  copper chromite
Cp  specific heat at constant pressure
CTPB  carboxy terminated polybutadiene
d  diameter
f  lens aperture
F  focal length of lens
IL  rated current
IO  iron oxide
\( \lambda \)  binder height
L  heat of gasification per unit mass of fuel
\( \dot{m} \)  mass flow rate
M  magnification
NL  power
P  pressure
PBAA  polybutadiene acrylic acid
PS  polysulfide
PU  polyurethane
PPS  pictures per second
q  heat lost from surface per unit mass of fuel gasified
LIST OF SYMBOLS (Continued)

\( \dot{q} \) heat flow rate per unit area

\( \hat{q} \) term used in Emmons problem (see Appendix F)

\( Q \) heat of reaction

\( r \) burn rate

\( R \) gas constant

\( R_L \) internal resistance

\( T \) absolute temperature

\( u \) distance from front principal plane of lens to object

\( U_G \) base voltage

\( U_L \) operating voltage at rated current

\( v \) distance from rear principal plane of lens to film plane

\( V \) mean flow velocity

\( W \) molecular weight

\( x \) distance in x-direction

\( Y \) net expansion factor for compressible flow through orifice

\( \gamma_{o} \) mass fraction of oxidizer

\( \gamma_{o} \) term used in Emmons problem (see Appendix F)

\( \gamma_{I} \) term used in Emmons problem (see Appendix F)

\( \delta \) 1/2 binder thickness

\( \Delta \) differential

\( \lambda \) thermal conductivity

\( \mu \) absolute viscosity or microns

\( v' \) stoichiometric coefficient for species appearing as a reactant

\( v'' \) stoichiometric coefficient for species appearing as a product

\( \rho \) density
LIST OF SYMBOLS (Continued)

\( \omega \) mass flow rate per unit area

\( \dot{\omega} \) total mass flow rate

Subscripts

AP ammonium perchlorate
b binder
c combustion
DF diffusion flame
g gas
i initial
s surface of binder
O orifice or oxidizer
l orifice inlet
\( \infty \) free stream

Superscripts

\( ^\circ \) standard reference or degrees
SUMMARY

Results are presented of an experimental motion picture study designed to provide observations of composite solid propellant combustion. This study adds greater insight into the combustion mechanism of composite solid propellants, increases and complements the observations of previous quench testing and provides a background of catalytic effects for use in future experimentation.

Two-dimensional sandwiches composed of pressed ammonium perchlorate (AP) sections laminated together with thin layers of four currently used binders -- polysulfide (PS), polyurethane (PU), polybutadiene acrylic acid (PBAA) and carboxy terminated polybutadiene (CTPB) -- were burned in a window bomb at pressures up to 3200 psig. High speed motion pictures of the combustion were taken at frame rates up to 4000 pictures per second using magnifications of 1:1 to 4:1 on the film plane. The resulting film was carefully reviewed to provide basic physical characteristics of the combustion.

An exploratory program involving catalytic effects was then undertaken using a similar approach. Catalysts investigated were copper chromite (CC) and iron oxide (IO).

Cinephotomacrographic results are not included here due to space and format considerations. Edited films are available on loan.

The general results of the investigation may be summarized by the following conclusions:

1. A flowable binder melt exists for PU, PBAA and CTPB during
combustion. A similar melt may exist for PS, but evidence of the melt was not present in this investigation.

2. An AP surface melt is present for PS sandwiches at pressures where the pure AP burn rate is decreasing. This possibly explains why propellant burn rates are so much greater than AP burn rates at these pressures.

3. The assumed sandwich combustion model with an AP monopropellant flame, a base flame and a diffusion flame aided greatly in sandwich deflagration interpretation.

4. Based on this motion picture study, there is no conclusive evidence that heterogeneous interfacial reactions occur for the non-catalyzed sandwiches. This is in agreement with the findings of Varney [13] and casts doubt on models employing this type reaction such as the model due to Hermance [14]. For the catalyzed sandwiches, leading edge regression was obtained in the area of the interface, but the resolution in this study was not great enough to determine if it was occurring exactly at the interface.

5. The results of this investigation indicate that the sandwich model of Nachbar [20] is not sufficiently detailed to account for the actual physical processes which are occurring in the sandwich combustion.

6. A realistic model for binder/AP composite propellant combustion would consist of:

   a. Binder melt flows onto the AP surface.
   
   b. AP melts, even in the pressure range where the AP burn rate is decreasing with increasing pressure.
   
   c. Three basic flames: AP flame and base flame, both of
which are elliptic in character, and a diffusion flame between the AP combustion products and the binder decomposition products which serves to decompose the binder.

7. Copper chromite catalyst augments the AP deflagration process at both 600 psig and 2000 psig, the augmentation increasing with pressure increase.

8. Copper chromite possibly catalyzes the AP flame and base flame gas phase reactions at both 600 psig and 2000 psig, increasing the heat release rate of the reactions.

9. Copper chromite catalyst possibly promotes gas phase reactions by its presence in crevices between solid fuel and solid AP (at the interface).

10. Copper chromite catalyst does not promote heterogeneous reactions of gases with the solid fuel binder and does not modify the pyrolysis mechanism of the solid fuel binder.

11. Iron oxide catalyst inhibits the AP deflagration process at 600 psig, but augments it at 2000 psig.

12. Iron oxide possibly catalyzes the base flame gas phase reactions at both 600 psig and 2000 psig, increasing the heat release rate of the reactions.

13. Iron oxide catalyst possibly promotes heterogeneous reactions by its presence in crevices between solid fuel and solid AP (at the interface).

14. Iron oxide catalyst does not promote heterogeneous reactions of gases with the solid fuel binder and does not modify the pyrolysis mechanism of the solid fuel binder.
15. The preceding conclusions concerning CC and IO catalysts indicate that some of the theories concerning the catalytic mechanisms of CC and IO with AP/binder composite propellants are justified whereas others are not. The theories of Levy and Friedman [32], Pearson [37], Pittman [36], and Steinz, Stang and Summerfield [12] concerning various gas phase mechanisms are shown to be feasible. The validity of the theories of Pearson [37] and Pearson and Sutton [38] concerning the solid fuel binder is shown to be doubtful for these catalysts.

16. Copper chromite is a more effective catalyst than iron oxide for AP/CTPB propellant systems.

17. With the exception of IO at low pressures (600 psig), the addition of CC and IO in the AP is the most effective method of increasing sandwich burn rate, their addition at the AP/binder interface is less effective and their addition in the binder does not appear to be very effective at all in increasing burn rates, at least not for the sandwich configuration.

18. It is recommended that a detailed experimental program of composite propellant sandwiches using various catalysts, binders and oxidizers be initiated with use of both high speed cinephotomacrography and quench testing.
CHAPTER I

INTRODUCTION

Background and Literature Review

Solid propellants have a variety of applications. They are used in intercontinental ballistic missiles, tactical weapons and various space applications. Therefore it is desirable that analytical methods for predicting their performance be devised.

Two types of solid propellant are distinguished, according to the distribution of fuel and oxidant. If fuel and oxidant are mixed at the molecular level, the propellant is called homogeneous or double-base. Heterogeneous mixtures of oxidizer crystals in an organic rubber or plastic-like fuel binder are called composite propellants. Of the two types, composite propellants are of greater current interest because they are often capable of liberating a larger amount of chemical energy per unit mass and are safer to handle.

The heterogeneous nature of composite propellants results in a very complicated combustion process. The physicochemical processes of propellant combustion are so complex that mathematically tractable models are generally of only restricted relevance. This complexity leads to the design and study of models that represent as close as possible the real conditions, but are easier to analyze, and which will permit elucidation of the fundamental processes. The type of composite solid propellant receiving the most research and development effort contains ammonium
perchlorate as the oxidizer mixed with a variety of fuel-binders and additives.

Surveys of solid propellant combustion have been presented by Geckler [1] in 1954, and by Gutman [2] in 1960. Geckler stated that no theoretical studies of composite propellant combustion had been published at that time and also that "A great deal of study of solid propellant combustion appears to have been expended unnecessarily because of inadequate experimental facts." Gutman stated that the lack of knowledge of the fundamental mechanisms of burning was the basic reason for being unable to theoretically describe the composite propellant burning process. This was observed to be the case even though substantial experimental and analytical effort had been expended on composite propellant combustion in the interval between the two surveys. Gutman also recommended that detailed photographic scrutiny with color moving pictures of flame profiles in combustion bombs be continued.

The essential distinction between the combustion processes involved in the burning of homogeneous double-base propellants and composite propellants was stressed by Rice [3] as early as 1945. Rice emphasized that the time required for mixing by diffusion of the gaseous fuel and oxidizer streams, which were generated at the surface of the burning propellant, entered as an essential step into the overall conversion rate and might, in fact, be of dominant importance for composite propellants. Rice proposed a columnar diffusion flame model, which was the first of the diffusion controlled models of composite propellant burning. Although the model gives the correct qualitative dependence of burning rate on oxidizer particle size, neglect of finite reaction times and the assumption
of a columnar flame geometry combine to cause the burning rate to be insensitive to pressure, which is contrary to experimental observations.

In 1950, Wilfong, Penner, and Daniels [4] introduced the idea that the rate-controlling reaction in the thermal decomposition of a double-base propellant occurs in the monomolecular surface layer exposed to the flame zone. Schultz and Dekker [5] applied this concept to the combustion of composite solid propellants in the form of a "Two-Temperature" postulate. This postulate states that the decomposition of the solid oxidizer and binder are essentially independent processes when viewed on a microscopic scale. This leads to the result that the mean surface temperatures of the oxidizer and binder during burning of a composite solid propellant are usually different. Anderson, et al. [6] proposed a model for the combustion of ammonium nitrate (AN) composite propellants which is based upon the "Two-Temperature" theory and kinetic data for the pyrolysis of propellant oxidizer and binder. It is postulated that the oxidizer gasifies first and its gaseous products undergo an exothermic redox* reaction near the surface to establish an adiabatic flame which governs the pyrolysis of the propellant binder. The binder pyrolysis products then react with the gaseous products of the oxidizer to form a diffusion flame at a relatively large distance from the surface. Their statements that, in general, changes in the chemical structure of the binder (or even complete absence of binder) have only a minor influence on the burning characteristics, along with the large distance of the

*Every reaction accompanied by a transfer of electrons is a unity of two opposite processes, namely oxidation and reduction. That is why all such reactions are called oxidation-reduction or redox reactions.
final diffusion flame from the surface, lead to their conclusion that the binder-oxidizer diffusion flame has insignificant influence on the processes occurring at the oxidizer surface. Chaiken [7] uses the "Two-Temperature" postulate and the model postulated by Anderson, et al., as the basis for his "Thermal Layer" theory. The thermal layer postulated is the exothermic reaction of the oxidizer decomposition products. This is taken to be the rate controlling step for AN type propellants and the agreement between the theory and available data on AN propellants is reasonable. The fact that the theory predicts that burning rate is linearly dependent upon pressure and insensitive to fuel type and fuel-oxidant mixture ratio renders it invalid for AP type propellants. Chaiken and Anderson [8] later considered the role of the binder in composite propellant combustion and expanded the "Thermal Layer" theory to AP type propellants. Several possibilities were presented for the interaction mechanisms of the binder gases with the oxidizer. A heuristic case was examined in which the binder gases diffused into the thermal layer and reacted at a rate which is slow compared to the oxidizer redox reaction. This necessitated the introduction of two variable mixing factors, the values of which were not derived from fundamental principles and no burning rate predictions were possible.

An analytical theory for the combustion of AP type propellants based on diffusion flames was proposed by Sutherland [9]. He considered diffusion flames over the fuel pockets as comprising the major source of heat release to support the combustion process. The analysis contains no explicit dependence of burning rate and flame structure on pressure. However, the fuel surface temperature appears as an important parameter in
the development and as it may vary with pressure, pressure dependence may enter in this manner. The theory has no procedure for the determination of the fuel surface temperature and therefore must be considered incomplete. Summerfield, et al. [10] expanded Sutherland's work to include the effects of pressure, oxidizer particle size and binder-oxidizer mixture ratio. They proposed a physicochemical model based on experimental observations and hypotheses and entitled the theory the "Granular Diffusion Flame" (GDF) model. The GDF model is a one-dimensional model in which it is assumed that the gasification process at the solid regressing surface is driven by conductive heat feedback from a two-stage flame occurring in the gas phase. This two-stage flame is composed of the gaseous constituents released by either surface pyrolysis or sublimation (both endothermic) from a dry surface. It is assumed that the flow is laminar. A premixed flame (exothermic) is postulated to exist adjacent to the AP surface with ammonia and perchloric acid (A/PA flame) as the premixed reactants. The products of this premixed reaction which are rich in oxygen and oxygen-containing compounds, then serve as a reactant along with the gaseous fuel constituents to form a diffusion flame (O/F flame) at a much greater distance from the surface than the first stage A/PA flame. The fuel gases are presumed to enter the O/F flame zone in the form of tiny gas pockets whose mass is independent of pressure. Both the rates of diffusional mixing and chemical reaction determine the overall reaction rate of the O/F flame. An expression for burning rate is obtained in which chemical reaction is rate controlling at low pressures while diffusion is rate controlling at high pressures. This theoretical burning rate law contains two parameters which may not be calculated directly due
to insufficient basic information on parameters involved in their calculation. One parameter is called the "Gas-Phase Reaction Time Parameter" as it varies inversely as the chemical reaction rate in the gas phase. This parameter is related to the binder-oxidizer ratio of the propellant. The other parameter is called the "Diffusion Time Parameter" as it varies inversely as the chemical reaction rate in the gas phase. This parameter is related to the average oxidizer particle size. In order to test the GDF theory more decisively, a series of experiments were performed by Blair, et al. [11]. The GDF theory was later extended by Steinz, Stang, and Summerfield [12] to subatmospheric pressures. Experimental burning rates for unmetallized propellants at pressures from subatmospheric to 100 atmospheres are correlated quite well by the GDF theory as long as oxidizer loading is sufficiently high, oxidizer particles are of medium size and the fuel is of the type that does not melt readily. When these conditions are not met abnormal burning in the form of plateaus, mesas or extinctions occurs. However, burning rates of fuels such as PS and PBAA are correlated by the GDF theory, although Varney [13] observed that these fuels form a flowable melt during the combustion process.

Hermance [14] recently proposed a model of composite propellant combustion which incorporates the heterogeneity of the burning surface of composite propellants and a mechanism for substantial energy release at the burning surface. This model includes fuel pyrolysis, AP decomposition, heterogeneous reaction between fuel and decomposed oxidizer along the fuel-oxidizer interfaces, and gas phase combustion of the final fuel and oxidizer decomposition products. This model gives quite good agreement between theory and experiment concerning the burning rate-pressure
relationship over a wide pressure range, qualitatively predicts the
effect of various particle size distributions and predicts surface tem­
peratures and surface heat generation reasonably well. Rather unrealistic
values are obtained for the flame standoff distances. Actually, there is
little or no evidence which unequivocally indicates the existence of the
heterogeneous reaction which is postulated to occur at the interfaces
and this is one of the major assumptions of the model. In addition, the
model ignores the diffusion processes in the gas phase which should be
important, at least at high pressures.

A qualitative description for combustion of composite propellants
based on flame structure was postulated by Fenn [15]. He envisions the
burning process to be driven by a gas phase fuel-oxidant diffusion flame
situated over the interfacial area between the solid fuel surface and the
solid oxidizer surface. A small premixed flame is assumed to occur at
the leading edge of the overall flame and this premixed flame is said to
lead the attack on the interfacial area. The effects of pressure, binder-
oxidizer ratio and oxidizer particle size are qualitatively predicted by
this model.

A simple, one-dimensional, diagnostic aid to understanding the
burning of composite propellants was presented by Culick and Dehority
[16]. They use an inverse computation in which the distribution of com­
bustion in the gas phase and the burning rate-pressure curve are specified
and then calculations are made to obtain values for rate of heat release,
flame thickness and standoff distance. This approach is useful for exam­
ining the gross effects of some of the parameters.

The most realistic model to date for AP type propellants has
recently been developed by Beckstead, Derr, and Price [17]. This model is realistic since it allows for a strong exothermic reaction taking place at the propellant surface as could occur due to the AP partially decomposing exothermically in the thin surface melt of AP reported by Boggs [18]. Three separate flame zones are considered: (1) a primary diffusion flame between the decomposition products of the binder and the oxidizer, (2) a premixed oxidizer flame, and (3) a final diffusion flame between the products of the other two flames. The oxidizer decomposition is taken as being the over-all controlling factor in the combustion process. The results obtained by this model show that the calculated surface temperature and the effect of oxidizer concentration predicted by the model are in agreement with observed experimental trends. The predicted effect of particle size is somewhat greater than observed experimentally while the effect due to initially heating or cooling the solid propellant is in excellent agreement with experimental data.

As a compromise between the complexity of the three-dimensional combustion zone and the oversimplification of a one-dimensional approximation, Schultz, Green and Penner [19] suggested a two-dimensional sandwich burner model. Nachbar [20], in 1960, postulated a two-dimensional theory for a sandwich burner model. This model assumes decomposition of fuel and oxidizer directly into gaseous reactants and makes use of the Shvab-Zeldovich procedure [21,22]. The solution obtained by the Shvab-Zeldovich procedure is independent of gas-phase reactions due to the neglect of finite gas phase kinetics and so, when coupled with pressure-independent surface pyrolysis laws for the rates of decomposition of each
of the solid constituents, it will yield burning rates which are independent of pressure. As the burning rates are actually pressure dependent, this model must be considered incomplete. Nachbar stated that no experiments with two-dimensional sandwich geometry had been reported as of 1960.

Several experiments involving two-dimensional sandwiches have since been reported. Hightower and Price [23] and [24] ran quench studies, along with high speed motion picture photography, of pure crystal AP type sandwiches with PBAA binder in a nitrogen flushed combustion bomb. An earlier study involving only pure crystals of AP [25] provided basic experimental techniques and base cases of pure AP deflagration. The sandwich study covered a pressure range from 100 to 1200 psig. No indication of preferential interfacial reaction was noted and this casts significant doubt on theories based on interfacial reactions such as Hermance's [14].

Nadaud [26] experimentally burned sandwich-like samples consisting of two slabs of polybutadiene pressed against a slab of AP. Various bonding agents between the AP and polybutadiene were tested and the results showed that interfacial propagation during combustion depended greatly on the bonding method used. Actual combustion tests were then run using no bonding agents, but just tightly pressing the two slabs of polybutadiene against the slab of AP. Tests were also conducted with polyurethane taking the place of the polybutadiene. The test results indicate that in the pressure domain from atmospheric to 300 psi Fenn's theory [15], in which a symmetrical gasification of the fuel and of the oxidizer is assumed, may be utilized. The leading edge of the regressing
surface was noted to be at the interface between the AP and the fuel. This is contradictory to the results of Hightower and Price [23]. This discrepancy evidently results due to the fact that the slabs were only pressed tightly together for Nadaud's tests, whereas much better bonding was achieved by Hightower and Price by curing of the fuel-binder while in place against the AP.

The deflagration of pure crystals of AP was later studied in even greater detail, using similar techniques to Hightower and Price, by Boggs [18]. Boggs' study covered a pressure range from 275 to 6000 psia. The burning rate-pressure curve of pure AP, Figure 1, is shown to have four different regimes: (1) Regime I is between 300 and 800 psia in which the curve has an equation of the form \( r = cp^n \) with \( n \) approximately 0.77, (2) Regime II is from 800 to 2000 psia in which the slope of the curve is positive, but decreasing, (3) Regime III is between 2000 and 4000 psia and the slope of the curve is negative, (4) Regime IV is above 4000 psia and the slope of the curve is rapidly increasing. The detailed nature of the combustion zone is said to be qualitatively different in each of these regions. Therefore the burning behavior of two-dimensional sandwiches of AP with a thin binder layer should exhibit distinct differences in these four different regions.

The most realistic model of AP deflagration to date has recently been developed by Guirao and Williams [27]. They employed an AP surface melt, as observed by Boggs in Regimes I and II, as one of the major aspects of the combustion process. A one-dimensional model for AP deflagration between 20 and 100 atmospheres was established in which exothermic condensed-phase reactions occurring in this AP surface melt provide the
Figure 1. Deflagration Rate of Ammonium Perchlorate in $N_2$. 
major portion of the heat release to the surface. A very good agreement with experimental burning rates is obtained in the pressure range 300 to 1500 psia.

Experimental sandwich studies have also been reported by Powling [26], Austin [29] and Varney [13]. Varney used sandwiches composed of a thin binder layer (PS, PU, PBAA or CTPB) laminated between two slabs of compacted polycrystalline sheets of AP. Quench tests were performed over a pressure range of 300 to 2400 psig and the quenched samples were investigated for surface characteristics and profile shapes. The compacted slabs of AP were found to match results of single crystal AP deflagrating as a monopropellant and as a sandwich constituent. Varney observed smooth contours at the binder-oxidizer interface which were similar to those observed by Hightower and Price [22]. Also binder melts were observed to cover portions of the AP surface for all binders tested. Varney postulated burning mechanisms for the deflagration of AP sandwiches with the different binders, but since he was unable to observe the combustion process these are highly hypothetical. Varney concludes that because of the uncertainties involved in sandwich regression time history and the actual flame structure and location, it is imperative that high speed motion pictures be taken of the sandwich combustion in order to obtain details unobtainable using quenching techniques.

Although much work has been done, very little knowledge exists concerning the details of catalyst effects in composite solid propellant combustion. Several mechanisms have been postulated for catalytic action on AP and on composite propellants. The basic premise of these mechanisms is that catalysts enhance the heat release rate and raise the
surface temperature of the propellant. This is postulated to occur in a variety of ways:

1. Through accelerating the gas phase reaction.
2. Through exothermic reactions of gases on the surface of catalyst particles imbedded in the propellant (heterogeneous or not).
3. Through heterogeneous reactions of gases with solid fuel.
4. Through modifying the pyrolysis mechanism of the solid fuel.
5. Through gas phase or heterogeneous reactions promoted by the presence of catalyst particles in crevices between solid fuel and solid AP (at the interface).

In 1957, Friedman, et al. [30] reported on experiments involving a weighed quantity of finely powdered catalyst, thoroughly mixed with as-received AP powder, the mixture then being pressed at 100,000 psi. The greatest increase in deflagration rate was produced by copper chromite (analyzing 85 per cent by weight CuO and 15 per cent by weight Cr₂O₃). Another catalyst investigated, iron III oxide (Fe₂O₃), was found to decrease the burning rate below that of pure AP at low pressures (below 1700 psi) and to increase the burning rate at high pressures (above 1700 psi). Copper chromite was observed to either augment the deflagration rate of pure AP or completely prevent burning, depending on the concentration and pressure. At 3000 psi the deflagration rate appears to increase monotonically with copper chromite concentration, from zero per cent up to a saturation point between 3 and 5 per cent. However, at a lower pressure, say 1500 psi, any copper chromite addition in excess of perhaps 2 per cent will augment the rate, while slightly smaller additions prevent burning. Deflagration of the pressed AP was found to have an
upper-pressure limit which could be slightly raised by preheating. This upper-pressure limit of approximately 4500 psi has more recently been shown to be nonexistent through the experiments of Boggs [18] and Bobolev, et al. [31]. Boggs used pure crystal AP and achieved deflagration up to 6000 psi while Bobolev and associates used clear pressed pellets and achieved deflagration up to 10,000 psi. The upper-pressure limit was evidently either apparatus or sample dependent. The addition of small percentages of catalysts was found to either eliminate this upper-pressure limit or shift it above the pressure limits of the apparatus. Some hypotheses stated concerning this upper-pressure limit are: (1) The mechanism for raising this upper-pressure limit might be that catalysts raise the surface temperature. Therefore, if this proposed mechanism is correct, copper chromite addition must be far more efficient than preheating in raising the surface temperature. (2) The reaction may be supposed to occur in two distinct stages. In the first stage, the solid decomposes to give intermediates. In the second stage, these intermediates diffuse to various active sites on the surface where they react further to give the final products. The first stage is rate-controlling unless the pressure is so high that diffusion is too slow; hence, above a critical pressure, an abrupt change of rate would occur. The catalytic mechanism might be that catalysts elevate the upper-pressure limit by providing additional sites which catalyze the second stage. However, this seems to be inconsistent with the finding that catalyst addition augments the deflagration rate at low as well as high pressures. (3) A final model involves a rate-controlling gas phase reaction from which heat is transferred to the surface. The exothermic gas reaction might well be of the
chain-branching type, with competition between second-order branching and third-order breaking processes. At a sufficiently high pressure, the chain-breaking processes overtake the chain-branching processes. Catalyst additions are assumed to promote gas phase chain-branching and, accordingly, to postpone or eliminate the upper limit.

Levy and Friedman [32] discussed further studies in 1962, of catalyst effect on AP deflagration. They stated that copper chromite definitely had an effect on the gas-phase reaction, as experiments indicated that the catalyst evidently catalyzed the conversion of nitrous oxide to nitrogen in the product gases. This is evidence that the catalyst did affect reactions in the gas phase, whatever its other functions may have been.

In 1967, Jacobs and Russell-Jones [33] presented data concerning kinetic data for AP pellets containing copper chromite. They stated that despite the early discovery [30] of copper chromite being an efficient burning rate catalyst, little attention has been paid to the study of its effect on the thermal decomposition of AP. They hoped to correlate their kinetic data with data on the sublimation and decomposition of pure AP and data on the deflagration of AP + copper chromite thus leading to enlightenment of the various mechanisms which had been proposed for the decomposition process. Using a thermogravimetric type apparatus they determined that the reaction rate constant increases with increasing concentration of copper chromite. The activation energy was found to increase from 31 to 48 kcal/mole with catalyst addition. They proposed the following model for catalytic action effect of AP: The primary step is the dissociation of AP into ammonia and perchloric acid on the surfaces
of the pellet granules. The molecules of ammonia and perchloric acid diffuse over the surface of the granules becoming adsorbed by the catalyst particles where decomposition of perchloric acid and oxidation of ammonia proceed. The rate-determining step seems likely to be the heterogeneous decomposition of perchloric acid since the measured activation energy is close to that required to decompose perchloric acid. Oxidation of ammonia commences on the catalyst surface, thus by-passing the activation energy required by the uncatalyzed reaction, and is continued in the gas phase. Heat from the exothermic gas phase reactions is transmitted back to the surface of the decomposing solid by conduction.

Waesche [34] and [35] performed differential scanning calorimetry decomposition tests of AP and composite propellants mixed with the catalysts copper chromite and iron oxide. He found that the catalysts accelerated AP decomposition at one atmosphere pressure, with copper chromite the more effective of the two materials. These effects also occurred for AP decomposition at 250 psi with the exception that iron oxide was nearly as effective as copper chromite at this pressure. In both cases, the effectiveness of the catalyst was seen to increase with increasing concentration. Propellants of AP with PS, PU and PBAA were tested with the same two catalysts and both decomposition and burning rate were found to increase with increasing catalyst concentration. It was noted that iron oxide was a more effective catalyst in propellants than in pure AP. Burning rate changes in those propellants containing AP and binder only resulted from changes in the pre-exponential factor of the reaction-rate expression, while the activation energy remained unchanged. The addition of the catalysts iron oxide and copper chromite on the other hand, caused increases
in both activation energy and pre-exponential factor. Pressure increases had only slight effects on propellant decomposition kinetics. The effectiveness of the two catalysts in increasing the pre-exponential factor was decreased as pressure increased. Waesche stated that the addition of iron oxide or copper chromite to propellants increased the heat flux requirement of the surface. He said that the required increase in heat transfer from the gas phase would occur as a result of the acceleration of gas-phase reactions accomplished by the additives.

Nadaud [26] examined the effect of copper chromite on his sandwich experiments at pressures from 5 to 80 atmospheres using 1 per cent of copper chromite in the fuel, oxidizer or both. He found that the maximum burning rate for his sandwiches occurred when the catalyst was present in the fuel. Lower burning rates were obtained when the copper chromite was present in the AP or in both the AP and the fuel.

Pittman [36] described a series of strand burning experiments using CTPB propellants and an iron containing catalyst. These experiments were aimed at determining the site of catalytic action and the mechanism of burning rate catalysis. His conclusions were that: (1) The effectiveness of mixing catalyst into the binder and of coating the interface between the AP and the binder with catalyst is the same. (2) Catalyst mixed into AP does not increase the effectiveness of the catalyst compared with mixing it into the binder. (3) The burning rate ratio of catalyzed to uncatalyzed propellants is unchanged if the oxidizer is modified by encapsulation in a Viton-A shell. This indicates that catalysts do not operate beneath the burning surface to enhance AP decomposition or to catalyze oxidizer-binder reactions. (4) Catalysts probably act in
the gas phase (including heterogeneous surface reactions) to increase
the reaction rate of perchloric acid and its initial decomposition prod­
ucts.

Pearson [37] conducted thermogravimetric analysis and differential
thermal analysis of the catalyst copper chromite. In addition he per­
formed studies on the effect of reactive gases on copper chromite. He
discussed the relevance of his results to the mechanisms of composite
propellant catalysis. His work showed that copper chromite was effective,
at temperatures close to those prevailing on the propellant surface, in
promoting the reaction of ammonia or olefins \( (C_{n}H_{2n}) \) with perchloric acid
or oxygen. Under the same experimental conditions, a paraffin hydrocarbon
\( (C_{n}H_{2n+2}) \), methane, was unaffected. Pronounced heat release was observed
both when the catalyst was exposed to fuel vapor and when it was exposed
to perchloric acid or oxygen. It was postulated that under the hetero­
geneous conditions prevailing on the surface of the composite propellants
it was possible that the catalyst particles are participating in a redox
cycle which was associated with exothermic reactions in both the reduc­
tion and oxidation stages of the cycle. In this redox reaction it was
suggested that the fuels react with the oxygen contained in the catalyst,
which was simultaneously reduced, and that this heat release depended on the
nature of the fuel. On the other hand, the effect of oxygen would be to
reoxidize the catalyst from its reduced state and this heat release should
be independent of the nature of the fuel previously introduced. It was
concluded that the enhanced heat release from such reactions on catalyst
particles on the surface of the propellant constitutes one route by which
the burning rate could be enhanced.
Pearson also postulated three other possible catalytic mechanisms for copper chromite with composite propellants: (1) Increased surface heat release might also enhance the burning rate through a second effect in which the gas-phase burning velocity was increased. Support for this argument is the subject of a paper by Hall and Pearson which is to be published. (2) The catalyst might promote the reaction of perchloric acid vapor with solid fuel. Pearson and Sutton [38] found that the ignition of mixtures of perchloric acid vapor and ammonia was accelerated by the addition of copper chromate as a catalyst. They also found that the ignition of gaseous fuels with perchloric acid vapor in the absence of a surface was considerably slower than that of the condensed fuel which would form these gaseous fuels by pyrolysis. Consequently, this suggested that heterogeneous reactions are the important ones leading to ignition in a propellant containing ammonium perchlorate. They proposed a reaction for an ammonium perchlorate solid propellant with a catalyst as follows: As the temperature rises, the ammonium perchlorate would dissociatively vaporize into ammonia and perchloric acid, and the fuel would pyrolyze into gaseous products. The perchloric acid and ammonia (or fuel products) were then suggested to react in the presence of the catalytic surface thus igniting the mixture. This reaction included the heterogeneous decomposition of perchloric acid vapor into chlorine oxides, which might subsequently react either homogeneously or heterogeneously with fuel. (3) The catalyst might modify the pyrolysis mechanism of the solid fuel. No support was given for this argument.

Catalysts which accelerate the high temperature decomposition of AP such as copper chromite are commented on by Steinz, Stang, and Summerfield
They state that two possible modes of catalytic activity exist for the normal rocket pressure range: (1) One or more of the O/F flame reactants react heterogeneously with the surface of the catalyst particles while they are still embedded in the propellant surface, and (2) The existence of gas-phase or heterogeneous reactions in crevices between the solid fuel and solid AP is promoted by the presence of catalyst particles at the interface between the solid AP and solid fuel. They feel that the latter possibility is more credible. To help clarify the mechanism of catalytic action they suggest an interesting set of tests which have not yet been done: Investigate (1) a propellant without catalyst, (2) the same propellant with fine catalyst particles mixed uniformly in with the fuel and (3) the same propellant with the same amount of the same catalyst, present now as a uniformly dispersed inclusion in the AP crystal structure.

Hightower and Price [23] and [24] conducted quench tests using copper chromite catalyst mixed with the PBAA binder of AP/PBAA sandwiches. Their only conclusion was that the pressure dependent surface pattern of the quenched sandwich was virtually unaffected by the addition of copper chromite.

**Purpose of the Research**

It is evident from the preceding literature survey that a wide variety of views exist regarding the mechanism of composite solid propellant combustion. Many of the theories thus far advanced are based on highly hypothetical cases in order to make the mathematical models more tractable. In order to achieve more realistic analytical models a more
thorough knowledge of the combustion process is required.

The purpose of this investigation will be to further the understanding of the basic combustion mechanisms of composite propellant combustion through experimental investigations.
CHAPTER II

INSTRUMENTATION AND EQUIPMENT

The experimental investigation was conducted in the Georgia Institute of Technology Aerospace Engineering Propulsion Laboratory. Principal aspects of the facility are a high pressure, variable flow combustion rig equipped for photography, a high speed movie camera, a high intensity light source, a motion analyzer (movie projector) and associated laboratory apparatus required for sample preparation and data analysis. A layout of the basis apparatus is shown in Figure 2.

Combustion Rig

The combustion rig, shown schematically in Figure 3, consists of a nitrogen supply, an orifice for flow measurement, a window bomb in which the sample is ignited and miscellaneous valves, plumbing, gauges and controls.

Nitrogen is supplied to the rig from three pressurized cylinders. One to three cylinders may be used depending on the flow requirements. Medium and low pressure runs were performed using high purity, dry nitrogen (Linde) supplied in cylinders containing 300 standard cubic feet at 2600 psig. High pressure runs, up to 3200 psig combustion pressure, were performed using prepurified nitrogen (Matheson) supplied in cylinders containing 338 standard cubic feet at 3500 psig. The rig is designed for an ultimate capability of 6000 psig. For such high pressures prepurified nitrogen (Matheson) supplied in cylinders containing 494 standard cubic
Figure 2. Basic System Layout.
Figure 3. System Flow Schematic
feet at 6000 psig may be used with only slight modifications to the existing rig as mentioned below.

The three nitrogen cylinders feed into a three-station, single row brass manifold supplied by the Matheson Company. The manifold, rated for 6000 psig safe working pressure, is wall mounted on the combustion system enclosure. This manifold is now set up for use with either the 2600 psig or 3500 psig cylinders. The 6000 psig cylinders may be used with this manifold by changing the cylinder connectors from CGA type 580 to CGA type 67U.

The nitrogen is admitted to the system from the manifold through the System Supply Valve, a Hoke Roto-Ball, type 303 stainless steel valve (P/N 7223F85). This type valve is also used as the Safety Vent Valve which provides a nitrogen bleed capability in case of a regulator lock-up or other unforeseen difficulty in the pressurization system.

Manifold supply pressure is monitored on a Marsh Type 210 pressure gauge. This gauge is constructed of type 403 stainless steel and has a pressure range of zero to 8000 psig. Accuracy is ± 20 psi and readability is 50 psi.

Nitrogen flow is monitored using a sharp-edged flow orifice furnished by Vickery-Simms, Inc. The orifice meter tube assembly is rated at ASA 1500#/ (good up to 3500 psig, should be removed for higher pressure runs) and is made of type 316 stainless steel. Schedule 80, 3/4 inch pipe is used upstream and downstream of the orifice plate. The orifice plate is 1/8 inch thick and the orifice hole is 0.136 inches in diameter. The orifice meter tube assembly and the orifice plate conform to guidelines laid down by the ASME [39] and by the American Gas
Association [40]. Orifice inlet temperature was measured using an iron-
constantan thermocouple furnished by the Thermo Electric Company. Average 
inlet temperature during a run was noted to be 525°R. The thermocouple 
was removed from the system upon noting that this temperature did not 
vary appreciably during a run and also due to the fact that slight 
inlet temperature variations only slightly affect mass flow. Pressure 
drop across the orifice is measured using a zero to 100 psi ΔP gauge 
furnished by the ITT Barton Company. This gauge is a Barton Model 227 
made of type 316 stainless steel and is rated at 6000 psig SWP. Accuracy 
is ± 0.5 psi and readability is 1 psi. A sample flow calculation may be 
found in Appendix A along with ΔP tables for one foot per second nominal 
flow velocity past the sample. The greatest inaccuracy of this system 
is that $P_1$, the orifice inlet pressure, is not constant, but varies 
during the run. $P_1$ varies approximately 200 psig per run, thus actual 
flows are based on an average $P_1$ during the run. This flow measurement 
system is used to provide a rough estimate of nitrogen flow past the 
sample thus removing combustion products from the field of view of the 
camera. The system is considered adequate for this purpose even with 
the inaccuracies of using an average $P_1$, assumed inlet temperature and 
the inherent inaccuracies of a sharp-edged orifice.

The combustion pressure level is maintained constant during the 
run by a Victor Controls dome-loaded pressure regulator, Model GD 68. 
The Model GD 68, a diaphragm operated, balanced poppet regulator with 
type 316 stainless steel body and springs and "Viton" diaphragm and 
seals is rated for 10,000 psig service. This regulator is in turn con-
trolled by a Victor Controls LR series pressure reducing regulator which
maintains the proper dome loading for the model GD 68. The LR series regulator is a low flow, high pressure regulator for the control of pressure up to 10,000 psig. The LR regulator is self-relieving and has a hand wheel control.

Combustion pressure is monitored by a Marsh Type 210 pressure gauge. This gauge is constructed of type 403 stainless steel and has a pressure range of zero to 5000 psig. Accuracy is ± 12.5 psi and readability is 20 psi. This gauge must be replaced if the system is ever operated above 5000 psig combustion pressure.

A stainless steel pressure vessel equipped for photography was used for control of the combustion atmosphere. The combustion vessel, schematically shown in Figure 4, is a three piece assembly with provisions for sample ignition, lighting and photography:

1. The lower section provides an inlet for the nitrogen pressurization flow, houses the ignition circuitry and serves as a base for the sample holder assembly.

2. The center section serves as the combustion section and has quartz windows for lighting and photography. The windows are ground and polished General Electric Type 151 Clear Fused Quartz Discs, 1 1/2 in. in diameter and one inch thick. Appendix B contains prints used for the construction and assembly of this center section.

3. The top section provides an exit for the exhaust flow. The three piece combustion vessel is retained by upper and lower stainless steel, threaded collar rings on the O.D. of the combustion section and utilizes rubber "O"-rings for leak proof pressure control.

The sample holder assembly, as shown in Figure 4, is attached to
Figure 4. Combustion Vessel Schematic.
the lower section of the combustion vessel and serves three functions:

1. Provides a mean for sample support in the combustion section.

2. Initiates smooth flow past the combustion sample via a five micron, sintered stainless steel porous plate which serves as the floor of the holder assembly.

3. Provides an extension of the ignition circuitry to the proximity of the combustion sample.

Sample ignition is accomplished by electrically heating a single strand of 10 mil nichrome wire positioned on the upper sandwich surface. The wire is positioned such that it will not interfere with the photography and also such that it lies along the binder layer. Ignition power is supplied by an American Monarch Company, 48 volt power supply. A variac is used to control the AC input to the power supply. With the ignition circuit open, the power supply is adjusted by the variac to an unloaded voltage of 36 vdc. During the run a microswitch in the movie camera is closed after a pre-set amount of film has been used. This in turn closes the ignition circuitry and applies approximately six volts DC and three amps to the ignition wire.

Smooth regulation of combustion vessel purge flow is provided by two Hoke 270 series needle valves arranged in parallel in the exhaust portion of the flow system.

Nitrogen flow lines are constructed with type 316 stainless steel tubing having either a 3/4 inch O.D. with a 0.120 inch wall thickness or a 1/4 inch O.D. with a 0.049 inch wall thickness and with type 304 stainless steel tubing having either a 3/4 inch O.D. with a 0.109 inch wall thickness or a 1/4 inch O.D. with a 0.049 inch wall thickness.
Leak proof connections are made using Crawford "Swagelok" type 316 stainless steel tube fittings.

The control console, where the test conductor stands during a run, is shielded from the pressurized combustion vessel by a shield consisting of 3/4 inch plywood and 1/4 inch carbon steel.

**High Speed Movie Camera and Associated Equipment**

High speed photography of the combustion of the sandwiches is accomplished with a Hycam 16 mm high speed motion picture camera, Model K 20S4E-115. This camera is supplied by Red Lake Laboratories, Inc. and has a film capacity of 400 feet of standard thickness film on day-lite spools. The frame rate is variable from 10 to 11,000 pictures per second.

The Hycam camera is mounted on a Palmgren No. 83 Rotary Table with Cross Slides which has adjustments for movement of the camera perpendicular and parallel to the sample as well as rotary motion in a horizontal plane. The rotary table is in turn mounted on a six foot Sheldon Machine Company lathe bed along with the combustion vessel. The combustion vessel is fixed in place on the lathe bed while the camera is free to slide perpendicularly to the combustion vessel and then be secured at the desired location.

Special features of the Hycam camera are a remote ON/OFF switch, a ground glass focusing gate, an event synchronizer and film speed timing lights. The remote ON/OFF switch allows operation of the camera by the test conductor while he is in place behind the safety shield during a run. The ground glass focusing gate is used for critical focusing.
It is placed in the location of the regular focusing gate during camera set-up and then must be removed for the actual filming process. The event synchronizer allows the camera operator to either start or stop an event when a pre-set amount of film has been used. This is used to close the ignition circuitry during a run. The ignition is set to occur close to the time when the camera has reached the selected film speed. Appendix C contains curves for selected film speeds versus time required or feet of film required to reach the selected film speed. Camera settings for achieving the desired film speed are also included in Appendix C.

The camera contains dual timing lights. One timing light may be used for determining film speed while the other is used as an event marker. The light used for timing is driven by a Red Lake Timing Light Generator, Milli-Mite Model TLG-3. This generator flashes the timing light at 10, 100 or 1000 cps. The light leaves marks along the side of the film thus allowing accurate film speed to be determined. Due to the instability noted in the timing frequency, a counter is used to note the frequency at the start and finish of a run. As the timing light generator develops 700 to 900 volts, the voltage to the counter must be lowered through the use of a series resistor. The other light was not used in this investigation.

The camera is equipped with an 85 mm, f/1.8 Vivitar Preset Lens. This lens along with a lens reversing adapter and proper extension tubes is used for magnifications of the samples from 1:1 to 4:1.

As an aid in determining proper exposures a Honeywell Pentax 1°/21° Exposure Meter is available. This meter may be used for either "spot" or "average" light meter readings. The meter calculator for this
meter is specifically set up for use with the Hycam camera, with pictures per second rather than shutter speed marked on the calculator. If it is desired to use this meter with a still camera, the shutter speed is \(1/(2.5 \times \text{PPS})\).

The majority of the runs were made with color film. Black and white film was used for checkout, pure AP runs and titles. The color film used was ASA 160 Kodak Ektachrome Daylight Film, Type 7241-EF430, 16 mm, 100 and 400 foot, for high speed cameras. The black and white film used was ASA 200 Daylight/ASA 160 Tungsten Tri-X Reversal Film, Type 7278-TXR430, 16 mm, 100 foot, for high speed cameras.

**High Intensity Light Source and Associated Equipment**

High intensity illumination of the sample is available through the use of a XeTron 2500 Watt xenon lamp system. The system consists of a power supply, a lamphouse and a lamp.

The power supply is a XeTron solid state power supply, Model N3-X95/140DM. This power supply requires 220 volt, 3 phase, 60 cycle AC input and delivers 28-40 volts DC at 75-140 amps output. This provides power to ignite the lamp initially as well as power to sustain the illumination.

The lamphouse is a XeTron Model 4000X, Type 25. The lamphouse contains mirrors for directing the light and holds the xenon lamp. Controls for focusing the light and moving it horizontally or vertically are in the lamphouse. A douser at the light exit of the lamphouse provides a means of containing the light within the lamphouse when illumination of the sample is not desired.
Both the power supply and the lamphouse are capable of using a 4000 watt xenon lamp. However, at present a 2500 watt xenon lamp is being used. This lamp is an Osram XBO 2500W lamp furnished by the Macbeth Sales Corporation. This lamp is rated at 2500 watts and operates at a mean DC voltage of 30 volts, a minimum current of 60 amps and a maximum current of 95 amps. Light from this lamp has practically the same spectral properties as daylight composed of mixed direct sunlight and indirect light from the sky. Therefore no color correction filter is needed for color photography when using daylight type film.

Cooling for the lamp is provided by a Grainger 7C037 Blower. Air at a rate of 300 cfm is pulled up through a filter in the base of the lamphouse, past the lamp and then out the top of the lamphouse into a flexible hose connected to the blower. The blower then expels the air into the laboratory.

In order to prevent unwanted infrared radiation from entering the combustion vessel an infrared absorbing, visible transmitting filter is installed over the illumination window of the combustion vessel. This filter is a Corning CS 1-75, two inches square.

Calculations for the amount of energy deposited on the sample by the xenon lamp during a run are included in Appendix D. The calculations, which are very conservative, show that the energy deposited on the sample by the lamp is roughly 30 per cent as much as is created by the combustion process or approximately 24 per cent of the total energy. This additional energy does not appear to influence the combustion process at all. Two similar tests were run with sandwiches, one with the xenon lamp and the other without, and there was no observable difference in any aspect of the combustion process.
Motion Analyzer

An L-W Model 900 Motion Analyzer is used for data analysis. The 16 mm film may be viewed with this machine using rear screen projection or normal type projection. The film speed is variable from one to eight frames per second, 16 frames per second or 24 frames per second. The projector may be operated in either the forward or the reverse mode and single frames may be stopped for as long as desired.

Associated Laboratory Apparatus

Associated laboratory apparatus required for sample preparation and data analysis are described below.

Sample Preparation Apparatus

A heavy glass mortar and pestle are used to thoroughly grind crystalline AP into a fine powder. AP polycrystalline compaction is achieved using a six piece mold in a Carver Laboratory Hydraulic Press as shown in Figure 21 of Varney [13].

All precision weighings required in sample formulation are made using a Mettler H6T Digital Balance. The Mettler H6T is a single pan, beam balance using synthetic sapphire knife-edged bearing planes for beam support. Weight compensation is accomplished by a combination of built-in ring weights. Readout is in digital form and is read optically. The weighing range of the H6T is zero to 160 grams with a standard deviation precision of ± 0.05 milligrams.

Binder samples are cured in an Acme Laboratory Vacuum Oven, Model 43573, which has a temperature range of 35-260°C maintained within ± 1°C of the selected level. Vacuum conditions in excess of 28 inches of
mercury are provided by a Welch two-stage Duo-Seal vacuum pump driven by a Craftsman one-half horsepower electric motor. The binder curing apparatus is shown in Figure 3 of Varney [13].

Microscopic determination of the sandwich binder thickness is achieved using a Bausch and Lomb Dynazoom Laboratory Microscope. The microscope permits fixed magnifications ranging from 40X to 1000X with a continuously variable 1X to 2X zoom magnification within each fixed setting. Basic illumination is achieved by a base mounted, variable output illuminator coupled with an Abbe condenser system. Photomicrographs can easily be made with the Dynazoom microscope which incorporates provisions for several camera assemblies. For this investigation a piece of ground glass with a marked scale on it was used at the film plane to determine binder thickness in microns. Adequate illumination to form the image on the ground glass requires external illumination. A Fish-Schurman "Zirconarc" photomicrographic lamp and power supply are used to provide this external illumination. The "Zirconarc" is basically an arc lamp, but its electrodes are fixed and sealed within a glass bulb containing an inert atmosphere. The power supply and lamp starter are a separate, self-enclosed unit operating from 110 volt AC power.

Data Analysis Apparatus

A Finescale Magnifying Comparator is available for use in measuring distances from a 16 mm film frame. Two scales are available, one with white markings and the other with black, which can be read to within 0.005 inch.
CHAPTER III

PROCEDURES

Ammonium Perchlorate Compaction

The pressed polycrystalline AP discs were prepared using the procedure developed by Varney [13].

Binder Preparation

Binders were formulated and mixed as outlined by Varney [13].

Catalyst Addition

The catalysts, Harshaw Catalyst Cu O2O2 (copper chromite - CC) and Fe2O3 (iron III oxide - 10), were introduced into the sandwiches by four methods:

1. Type 2, 2 per cent by weight in the AP alone.

2. Type 4, same volumetric loading in the binder alone as occurred in the AP of Type 2.

3. Type 5, same volumetric loading in both the AP and binder as in Type 2.

4. Type 6, a paint of catalyst on one interface of the AP before curing the binder between the AP slabs.

The manufacturing of the above sandwiches is straightforward with the exception of the Type 6 sandwiches. The Type 6 sandwiches were manufactured by making a paint using the catalyst and methyl alcohol. This paint was then spread evenly over one surface of a compacted polycrys-
talline AP disc. A sandwich was then made using the coated AP disc and positioning the coated side toward the binder layer. The per cent by weight of catalyst was not controlled for this type sandwich.

**Sandwich Preparation**

Sandwiches were prepared as outlined by Varney [13] with the exception that the sandwich edges were smoothed with a single-edged razor blade in order to ensure a flat top surface for even ignition and to ensure a smooth edge toward the camera thus improving the focus. Also two small lines were scratched into the camera edge of the sandwiches, the first approximately 1/10 inch from the top and the second 1/10 inch below the first. These lines were then made more visible by the use of a black ball point pen. The lines were used to aid in the framing and focusing of the sandwich and to provide a quick estimate of the burning rate of the sandwich. The sandwiches were mounted on 10-32 UNF x 1/2 inch binding head machine screws.

**Binder Thickness Determination**

The binder thicknesses were determined after the sandwiches were mounted on the machine screws. The Baush and Lomb Dynazoom Laboratory Microscope with a piece of ground glass at the film plane and with the Fish-Schurman "Zirconarc" photomicrographic lamp and power supply were used in determining the binder thicknesses.

The sandwich was placed with the camera edge up on a glass slide and was held in place with kneaded rubber. The sandwich was aligned carefully so that the binder layer was perpendicular to the glass slide and the camera edge was parallel to the glass slide. The glass slide
was then placed on the mechanical stage of the microscope. The sand­
wich edge was illuminated externally using the "Zirconarc" lamp. The
microscope was focused on the binder layer by looking through the eye­
pieces and using the 10X objective lens and the zoom lens set at 1X.
The image was then switched to the ground glass, being magnified by
another 10X lens in the camera system. Thus the image appeared on the
ground glass at 100X magnification. The ground glass was marked with a
scale graduated in millimeters. The binder thickness was now measured
using the fact that one millimeter on the ground glass scale was equiva­
lent to ten microns of binder thickness.

**Ignition Wiring**

A schematic of the ignition system is shown in Figure 5. The
ignition was accomplished by closing the microswitch in the camera, thus
applying a voltage of 36 volts DC (unloaded) to the 10 mil nichrome
ignition wire. The microswitch in the camera was closed after a pre-set
amount of film had been expended, therefore allowing the camera to get
up to speed prior to ignition. The open circuit voltage of the power
supply was regulated by a variac which controlled the AC input voltage
to the power supply, this in turn controlling the power supply's DC
output voltage. With the unloaded voltage of the power supply set at 36
volts DC, approximately 3 amps and 6 volts (18 watts) was applied to
the 1-1 1/2 inch piece of 10 mil nichrome wire which was in place on
top of the sample.

The 10 mil nichrome wire was placed carefully on top of the
sample so that ignition was insured and the ignition occurred either
Figure 5. Ignition Wiring Schematic
evenly along the top of the binder layer or at the camera edge of the sample. To aid in placing the wire properly the samples were slightly notched at the top along the binder. This helped to hold the wire in place against the binder. The wire was also fitted tightly enough against the binder so that it was not displaced upward away from the binder by the purge flow. No energetic igniter pastes were used to help promote ignition for any of the sandwich runs. However, a small droplet of tris-(2-methyl-1-aziridinal) phosphine oxide (MAPO) was applied to the top edge of the samples for tests involving AP only.

**Magnification of Sample**

It was desired to magnify the sample from 1:1 to 4:1 on the motion picture film in order to enlarge the details of the combustion process. Photographing objects using magnifications of these magnitudes is called photomacrography or for motion pictures cinephotomacrography. Information concerning this subject may be found in an Eastman Kodak technical publication [41] and in Cox [42].

For photomacrography a lens of short focal length with a bellows or extension tube are normally used. Regular camera lenses are designed so that the front of the lens faces the object and the focal length of the lens is less than the object distance. The distance from the film plane to the rear principal plane of the lens is equivalent to the focal length. This provides good definition in ordinary photography. However, in photomacrography the object distance is less than the focal length while the distance from the film plane to the rear principal plane of the lens is greater than the focal length. Therefore the lens should
be reversed for photomacrography in order to obtain the best definition. Reversing the lens also generally decreases the length of extension tube required for a particular magnification. The principal planes of a lens are planes from which the object and image distances for the lens are measured. If a ray of light enters a lens at a certain angle with the axis and then leaves at an equal angle, the position where the entering ray cuts the lens axis is called the front nodal point and the position on the axis from which the emergent ray appears to come is called the rear nodal point. The principal planes are planes at right angles to the lens axis which pass through these two nodal points.

The distance, \( u \), from the front principal plane of a lens to the object may be found by the formula,

\[
u = F(1 + 1/M)\]

where \( F \) is the focal length of the lens and \( M \) is the magnification. Since 4:1 magnification was desired and the minimum value of \( u \) was roughly 7.6 centimeters, \( u \) being the distance from the center of the combustion vessel to outside the window, the minimum focal length lens for our purposes was found to be 61 mm. This was not a short focal length lens, as was previously noted to be desired, but the system legislated that a short focal length lens could not be used.

The extension tube length (image distance) required may be found by the formula,

\[
v = F(1 + M)\]
where \( v \) is the distance from the rear principal plane of the lens to the film plane.

It is difficult to determine the location of the principal planes of a lens. They do not align with the physical front and rear of the lens, but are usually inside the lens somewhere. Most manufacturers of regular camera lenses do not publish this information, but it may be obtained for a particular lens by using a nodal slide on an optical bench, or a manufacturer may obtain it for a fee.

Extending the lens away from the film plane also results in a loss of light. A new "effective aperture" for any magnification can be determined using the formula,

\[
\text{Effective f-number} = f(M + 1)
\]

where \( f \) is the actual lens aperture.

Several problems occur in photomacrography. The main difficulties are:

1. Extremely shallow depth-of-field. At close working distances, regardless of the lens used, depth-of-field is reduced to fractions of an inch.

2. Extreme sensitivity to camera movement.

3. Exposure compensation. Extending the lens away from the film plane results in a loss of light.

For our purposes an 85 mm focal length lens was obtained and used in the reversed position. This meets the minimum focal length requirement which was previously discussed. Lenses made specifically for high
magnification work were not available except in short focal length lenses.

Since the location of the principal planes for this lens were not available, magnifications were determined as follows:

1. A small scale was mounted vertically on the sample post of the combustion vessel.

2. Different extension tube lengths between the lens and camera were tried until the marks visible on the film frame were equivalent to the vertical size of the frame (0.3 inches) divided by the magnification desired. The total extension length was actually longer than the tubes used since a lens reversing adapter was used for purposes previously described.

**Focusing**

The depth-of-field, as previously noted, is extremely shallow for photomacrography. Therefore accurately focusing the sample image on the film was imperative, especially at the higher magnification levels used. Focusing should be done with the lens closed down to the aperture to be used in filming since some lenses change their plane of sharpest focus when they are stopped down. The near depth-of-field is always slightly less than the far depth. Slightly out of focus detail is less obtrusive when it appears near the back of the subject than at the front. This indicates that detail near the camera should be favored slightly in focusing. An effect that may be confused with poor focus is the blurring caused by either camera vibration or subject drift during exposure.
Focusing was accomplished as follows:

1. The lower film gate of the Hycam camera was replaced by the ground glass focusing gate.

2. The sample was illuminated with high intensity illumination, using either the "Zirconarc" lamp or a General Electric Reflector Photolamp.

3. The camera was moved along the lathe bed until a rough image of the sample was obtained and was then secured in place by tightening the two bolts at the base of the camera mount.

4. Movement of the camera parallel and perpendicular to the sample and rotation in the horizontal plane was controlled using the rotary table incorporated in the mount. Movement of the camera in the vertical direction, both parallel to the sample and rotation in the vertical plane, was accomplished using spacers and by adjusting the two bolts at the base of the camera.

5. The lens was set at the aperture to be used during the test.

6. The sample was then centered on the ground glass and fine focus was achieved using the rotary table adjustment perpendicular to the sample. The camera was moved toward the sample until it was slightly past the sharpest point of focus and then moved back away from the sample until it was either slightly past or at the sharpest point of focus.

The high pressure runs (2000 psig and above) presented a difficult focusing problem. At these high pressures, slight movement of the sample toward the camera was evident which moved the front edge of the sample out of the field of sharp focus. Since no correction could be determined which would stop this slight movement, the camera was adjusted
to sharp focus as noted above with the system unpressurized and was then moved away from the sample an extra 10-20° of turn on the rotary table adjustment knob.

**Exposure Determination**

The correct exposure for a particular camera speed, lighting and magnification was determined by running a series of tests. For these tests, various apertures were used at a particular camera speed, lighting and magnification. Tests were made for various xenon lamp settings and at various magnifications. These tests showed that the xenon lamp set at its minimum operating current (approximately 65 amps) provided sufficient illumination. The camera speed effect was found to act as expected; by doubling the camera speed the aperture had to be opened up one stop for correct exposure at constant lighting and magnification. Tests were made at the different magnifications to be used during the experimental program in order to more accurately determine proper exposures.

Settings which gave good results with the xenon lamp operating at its minimum amperage, the light passing through the infrared absorbing filter and using Ektachrome film were as follows:

1. Magnification = 1:1; 800 pps, f/9.5.
2. Magnification = 2:1; 1600 pps, f/6.7 to f/8.

Normal extrapolation sufficed in determining different camera speeds and corresponding apertures.
Smoke Control

In order to determine the proper nitrogen purge flow required to remove the combustion products from the camera's field of view, a series of tests were conducted. Various flow rates, from no-flow up to three feet per second flow velocity past the sample, were observed at different pressure levels. These tests showed that approximately one foot per second flow velocity past the sample was sufficient to remove the exhaust products for the pressure range covered in this experiment. This flow velocity was also used by Hightower and Price [24] in their sandwich work. Higher velocities past the sample also removed the combustion products adequately, but since the results seemed to be no better than at one foot per second, a nominal flow velocity of one foot per second was chosen for use in the experimental program. Early in the program an attempt was made to match the nitrogen flow velocity with the combustion gas velocity. This also did not appear to remove the exhaust products any better than the one foot per second nominal flow velocity.

Run Procedures

A step by step Pre-Run Procedure and Run Procedure are included in Appendix E. Run Data Sheets for all of the runs attempted in this experimental program are also included in Appendix E. A typical run had a duration of approximately three seconds. One to two seconds were required to ensure the camera reached its pre-selected speed (1600 pps for most runs) and the actual combustion process then required 1/2 to one second. The majority of the runs were filmed at a frame rate of 1600 pps and a magnification of 2:1. A frame rate of 1600 pps was
Chosen since this speed seemed to slow the motion sufficiently for observation of the combustion process. Tests run at higher frame rates, up to 4000 pps, did not appear to provide any more detail than the tests run at 1600 pps. Another reason for choosing 1600 pps was that a 100 ft. roll of film lasts approximately three seconds, the typical run duration. The 2:1 magnification was chosen since it provided much better detail than 1:1 magnification. Magnifications higher than 2:1 were not used extensively due to the focusing and depth of field problems previously discussed.

Data Analysis

Procedures involving the utilization of the motion picture film after it was developed are described below.

The motion pictures of each test were carefully scrutinized. A Film Analysis Form was completed for each test. A sample filled out form is shown in Figure 6. Also tracings were made of the instantaneous sandwich profile during combustion.

The burn rate portion of the Film Analysis Form was obtained using two different methods. The first, and most accurate, method was to place the film on a light table and use the Finescale Magnifying Comparator. A portion of the film where the combustion appeared to be steady was chosen and the camera speed, magnification and amount of the sandwich burned for a number of frames were determined. The camera speed was determined by counting the timing marks on the film over the number of frames selected for the burning rate determination and using the average cps of the timing light during the run. The magnification
**SAMPLE - 100 µ PS\)**

**PRESSURE - 1000 psig\)**

**RUN NUMBER - 51\)**

**MAGNIFICATION - 2:1\)**

**FILM SPEED - 1600 pps\)**

**QUALITY - Very Good\)**

1. **BURN RATE**
   \[ r_1 = 0.3 \text{ in/sec}; \quad r_2 = 0.32 \text{ in/sec} \]

2. **PROFILES**

   ![Flamelets out over AP](image)

   ![Large AP bubble](image)

3. **DESCRIPTION OF FLAME STRUCTURE & PROFILES**

   Profile appears similar to the other 1000 psig runs. The flame structure shows flamelets out over the AP surface on left side.

4. **BINDER & AP MELTS**

   No visible binder melt on edge although flamelets indicate that a binder melt is present. Good AP melt visible on right side.

5. **BINDER HEIGHT**

   \[ t = 0.021 \text{ inch} \]

6. **GENERAL COMMENTS**

   Very good quality movie.

---

**Figure 6. Film Analysis Form**
was determined by measuring the sandwich cross-section on the film using the magnifying comparator and comparing this to the actual cross-section as noted on the Run Data Sheet. The amount of sandwich burned for the number of frames selected was determined by using the magnifying comparator. The burn rate was then calculated. The sandwich burn rates determined in this experiment are based on the leading edge of the steadily regressing sandwich surface. Thus they are the maximum steady state burn rates.

The second method of determining burn rates was only used for a rough comparison with the first method. The film was placed in the motion analyzer and run until the top burn rate mark on the sandwich was reached by the regressing surface. The analyzer was stopped here and its frame counter set to zero. The film was then run until the regressing surface reached the lower burn rate mark. Burn rate was then determined knowing that the sandwich had burned approximately 1/10 inch during the number of frames shown on the motion analyzer's counter. The camera speed was assumed to be constant at the setting listed on the Run Data Sheet. The burn rate was then calculated.

The tracings were made using the motion analyzer in the stop mode along with its small table top viewing screen. The analyzer was focused directly on the viewing screen opposite the analyzer. The 1:1 magnification tests were focused on the screen so that the cross-section width of the sandwich was approximately 2 7/8 inches. Selected sandwich profiles were then traced by following the outline visible on the tracing paper. The 2:1 magnification tests were focused so that the sandwich width was approximately 4 1/2 inches. These tracings were then reduced to 65 per
cent size, which left them approximately the size of the 1:1 magnification tracings. The tracings were then compared to each other and to a theoretical monopropellant profile. The theoretical monopropellant profile resulted from assuming ignition at the center of the sample, the profile then developing as a circle of increasing radius. A slab of pure, compacted polycrystalline AP approximately the same size as a sandwich was burned at 1000 psig. Figure 7 shows how well the theoretical profiles agreed with the actual experimental profiles.
1 THROUGH 4 ARE THEORETICAL MONOPROPELLANT PROFILES.

Figure 7. Comparison of Compacted Polycrystalline AP Profiles at 1000 psig with Theoretical Monopropellant Profiles.
CHAPTER IV

HIGH SPEED MOTION PICTURE STUDY OF UNCATALYZED AP/BINDER SANDWICHES

Two-dimensional propellant sandwiches were observed during combustion through the use of high speed motion pictures. The sandwiches were composed of pressed ammonium perchlorate (AP) sections laminated together with thin layers of four currently used binders: polysulfide (PS), polyurethane (PU), polybutadiene acrylic acid (PBAAS) and carboxy terminated polybutadiene (CTPB). Sandwiches were burned in a combustion vessel at pressures from 300 psig to 3200 psig. System pressurization and purge flow for smoke removal were provided by dry nitrogen which flowed vertically upward past the sample at a nominal velocity of one foot per second. High speed motion pictures of the combustion were taken using color film at frame rates up to 4000 pictures per second and magnifications of 1:1 to 4:1. Illumination for the motion pictures was provided by a 2500 watt high intensity xenon lamp. Two-dimensional propellant sandwiches are less complex than three-dimensional propellants and permit viewing of the flame structure and the surface profile in great detail while providing some heterogeneous structure.

The potential of simplifying the study of composite solid propellant combustion by utilizing two-dimensional sandwiches has been explored theoretically by Nachbar [20] and Fenn [15] and experimentally by several investigators, primarily Hightower and Price [24], Nadaud [26], Powling [28], Austin [29] and Varney [13].
The major goals for this portion of the investigation were to raise the pressure for experimental sandwich combustion to 3200 psig and to answer questions arising from Varney's investigation. In order to achieve these objectives, several tests were conducted and the resulting film was carefully reviewed thus providing basic physical characteristics of the combustion processes. The data obtained are composed of many high speed motion pictures showing the combustion of AP/binder sandwiches. Space and format considerations preclude the presentation of the cinephotomacrography results. However, edited films are available on loan from:

School of Aerospace Engineering
Georgia Institute of Technology
Atlanta, Georgia 30332
Attn: Prof. Warren C. Strahle

Several items of interest were investigated in this uncatalyzed sandwich portion of the investigation. The items investigated were:

1. Pressure effects on the PS sandwich combustion.
2. Differences in the flame structures of the four binders.
3. Evidence of binder melts.
4. Evidence of AP surface melts.
5. Attainment of steady state combustion.
6. Reasons for asymmetries.
7. Measurement of binder heights above the AP surface.
8. Peculiar AP plateaus observed by Varney.

The data obtained in the investigation, consisting of motion pictures showing the sandwiches during combustion, were carefully analyzed by measuring burn rates and by observing sandwich surface profiles, flame
structures and general aspects of the combustion process. The burn rates were obtained for the leading edge of regression of the sandwich surface. The results of this analysis were further analyzed using theoretical monopropellant profiles, kinematics and an assumed combustion model.

The theoretical monopropellant profiles were obtained by assuming ignition at the center of the sample, with the profile then developing as a circle of increasing radius. Sample profiles are shown in Figure 8. The monopropellant profiles are unsteady, with the local burn rate normal to the propellant surface, $r_m$, constant. The vertical burn rate, $r_v$, varies from equivalent to $r_m$ at the center of the sample to greater than $r_m$ at the sides. The maximum $r_v$ in the section of the sample utilized for analysis, occurs at the top edge of the sample. This maximum $r_v$ is approximately 10 per cent greater than $r_m$ and is approximately 8 per cent greater than the maximum $r_v$ at the lower edge of the section. Thus within the accuracy of the experimental observations, this section has approximately steady state regression. These monopropellant profiles provide a visual comparison with sandwich profiles thus providing a rough indication of which regions of the sandwich are regressing similarly to a monopropellant.

A slightly different analysis was used for the sandwich configuration. This is shown in Figure 9. For the sandwich, the local burning rates normal to the surface, $r_s$, are not constant, but vary along the profile. The majority of the sandwiches appear to the eye to have a steady state profile through the analysis section. The vertical burn rate, $r_v$, is then constant for steady state regression. This vertical burn rate is referred to as the sandwich burn rate in the following
1. \(1\) THROUGH \(4\) ARE THEORETICAL MONOPROPELLANT PROFILES.

2. FOR PROFILE \(2\) : \(r_v = r_m / \cos \theta\), WHERE \(r_m\) IS CONSTANT
   AND \(r_v\) VARIES WITH THE PROFILE SLOPE.

Figure 8. Theoretical Monopropellant Profiles.
$r = \frac{r_s}{\cos \theta}$

$\theta_1 = \theta \Rightarrow r_{v_1} = \frac{r_m}{\cos \theta_m}$, WHERE $m$ DESIGNATES MONOPROPELLANT.

$r_{v_2} = \frac{r_{s_2}}{\cos \theta_2} = r_{s_2}$ SINCE $\theta_2 = 0$

$r_{v_3} = \frac{r_{s_3}}{\cos \theta_3}$ WHERE $\theta_3 > \theta_1$

$\frac{r_m}{\cos \theta_m} = r_{s_2} = \frac{r_{s_3}}{\cos \theta_3} \Rightarrow r_{s_2} > r_m > r_{s_3}$

Figure 9. Sandwich Regression Analysis.
discussion. The sandwich burn rates should never be less than the pure AP monopropellant burn rate unless the AP regression itself is in some way hindered. Away from the center of the sandwich and the influence of the binder, the surface should regress with a monopropellant type profile. A detailed description of this type analysis is given by Varney [13]. This type analysis is beneficial since it can be used to compare local burn rates normal to the surface of points along the profile and thus determine the relative rates at which the points are regressing. Also in the case of catalyzed sandwiches the burn rate, $r_s$, of the AP may be determined using the vertical burn rate, $r_v$, of the sandwich obtained from the burn rate measurements. This can then be compared to the pure AP burn rate and whether the AP regression is accelerated by the catalyst, retarded or not affected can be determined.

Several flames probably make up the complicated AP composite propellant combustion process. These include:

1. AP monopropellant flame.
2. Diffusion flame between AP decomposition products and binder pyrolysis products.
3. Diffusion flame between AP combustion products and binder pyrolysis products.
4. Ignition region at the base of all diffusion flames.
5. Premixed flame caused by binder melt flows onto the oxidizer.

In addition to these possible flames there is apparently an AP surface melt, as observed experimentally by Boggs [18] and utilized analytically by Guirao and Williams [27], in which exothermic condensed-phase reactions occur.
A simplified model of combustion was assumed as an aid in interpreting the experimental results. The major heat sources involved in the combustion process were assumed to be the AP monopropellant flame, the AP surface melt, the diffusion flame formed by the AP combustion products and the binder pyrolysis products, and the ignition region at the base of the diffusion flame. The AP monopropellant flame and the AP surface melt were assumed in the model to be a single heat source and were called the AP flame. The diffusion flame mentioned above was assumed to be divided into two parts. The upper portion of the diffusion flame which provides negligible heat feedback to the sandwich surface was called the diffusion flame for the model. The ignition region at the base of the diffusion flame and the lower portion of the diffusion flame were called the base flame for the model. Thus the model consisted of three flames: an AP flame, a diffusion flame and a base flame. The model is shown schematically in Figure 10. The base flame is elliptic in character as opposed to the parabolic character of the diffusion flame. It was assumed that this base flame is one of the major sources of energy transfer to the sandwich surface for the sandwich configuration. The AP flame was also assumed to be a major energy source. The diffusion flame was assumed to have no effect on the sandwich surface. This simplified model is similar to the "Phalanx Flame Model" of Fenn [15]. Through the use of this simplified model some qualitative results of the sandwich combustion process were obtained. The base flame and the AP monopropellant flame were assumed to be affected by pressure changes to a greater extent than the diffusion flame. For a Burke-Schumann type diffusion flame the height of the flame is known to be invariant with
Figure 10. Analytical Sandwich Combustion Model.
pressure for constant mass flow of fuel and oxidant. By definition in this model, the diffusion flame can not account for the pressure dependence of burning rate. However, the base flame and AP monopropellant flame, being elliptic in character, were assumed to account for the pressure dependence of burning rate. As the pressure is increased the reaction rates increase and the flames are moved closer to the sandwich surface. Being closer to the surface, heat transfer is enhanced from the reaction zone to the surface and the burning rate is increased.

Also shown in Figure 10 are typical corner effects created by the cooling of the nitrogen purge flow. These corner effects are characteristic of the experimental apparatus and techniques used and are not considered to represent realistic combustion phenomena. These effects appear to be confined to a small portion of the sandwich near the edges or corners and do not seem to affect the sandwich combustion characteristics elsewhere. The AP flame affects the sandwich surface to a depth of only 10-20 microns (see Boggs [18]). Thus the AP flame should lose heat to the nitrogen purge flow only near the sandwich edges.

The visible flame for sandwich combustion is made up of luminous particles of the AP/binder reactions. The reaction zone of the diffusion flame is not visible. Thus the flame structure shown in the figures is not really indicative of the diffusion flame structure. The extent or height of the visible flame increases with an increase in binder thickness. The flame height is not accurate for some of the figures. The sandwich surface must regress in planes which are parallel to the lens axis in order for the flame height to be accurately portrayed on the film. Many of the tests were made with the surface at an angle
to the lens axis. For such tests, the flame height is inaccurate. In fact, the transparent flame of the PS sandwiches was not visible at all in views which were at such an angle that the sandwich surface could be observed.

Binder thicknesses ranging from 20 to 300 microns were employed in these experiments. The binder thickness in all cases is much less than the AP thickness. The binder only affects the area of the sandwich near it and the portions of the AP surface away from the interfacial area are not affected. Thus these different thickness sandwiches can be compared to each other, keeping in mind that a thick binder will provide a larger heat sink than a thin binder of the same type.

The term interfacial regression used in this study refers to regression in the immediate vicinity of the interface, but does not necessarily imply regression at the interface itself.

**Combustion Pressure Effect**

In order to determine the effect of combustion pressure on burn rate, surface profile and flame structure, a series of tests were conducted using eight pressure levels and 50-150 \( \mu \) binder thickness PS sandwiches. The pressure levels were 300, 600, 1000, 1500, 2000, 2400, 2800 and 3200 psig. This series of tests raised the maximum pressure investigated for sandwiches to 3200 psig. Varney [13] had an upper pressure limit of 2400 psig. Hightower and Price [24] used PBAA and had an upper pressure limit of 1200 psia. The tests covered combustion in three of the four AP zones noted by Boggs [18] (refer to Figure 1). The motion pictures of Hightower and Price only covered the first two
zones. Polysulfide was chosen for this series of tests since Varney found it to be the least erratic of the four binders he tested. He found that the burning rate for PS sandwiches was increased over that of pure AP; this increase being uniform with pressure increase and with binder lamination thickness increase. Actually, no change in burning rate should be observed if the binder is consumed by a diffusion flame mechanism.

Burn rate results for PS sandwiches are shown in Figure 11. Included are Varney's results obtained through quench testing, Bastress, et al. [43] results for PS propellant and Boggs' results for pure AP crystals. As shown in the curve, the binder thickness has no appreciable effect on the burn rate as determined by the motion pictures and the pictures show that the binder is consumed by a diffusion flame mechanism. Thus it is seen that the quench testing, in which Varney calculated burn rates using the mass loss and burn time, both of which could not be determined very accurately, introduced an anomaly which has been removed by the motion picture study. The PS sandwich burn rates obtained through this experiment agree fairly well with the AP/PS propellant burn rates of Bastress, et al.

**PS Sandwich Deflagration at 300 psig**

The burn rate of the PS sandwich is less than the burn rate measured by Boggs for pure AP crystals, but is approximately the same as measured by Varney for PS sandwiches. Using the sandwich profile analysis previously described, the AP was found to be regressing at approximately 0.07 in/sec normal to the surface as compared to a pure AP monopropellant rate of 0.13 in/sec. This suggests that the AP in the sandwich is
Figure 11. PS Sandwich Burn Rates.
not regressing similarly to pure AP at this pressure. This possibly results from the fact that this pressure level is very near the lower pressure limit of pure AP deflagration and therefore the data are of questionable accuracy. A typical profile for a PS sandwich deflagrating at 300 psig is shown in Figure 12a. This profile has a much steeper slope than the monopropellant profile and as mentioned above, the AP burn rate has been retarded. Using the simplified model of combustion previously described, this profile may be possibly explained by assuming that the base flame is the major heat source for the combustion process. This must be the case since the AP regression rate is retarded and therefore the AP flame is not adding its normal heat release. The diffusion flame itself, being parabolic in character, aids in pyrolyzing the binder, but probably adds no heat to the sandwich surface. The profile is different from that obtained by Varney. Varney's profile is similar near the binder, but is rounded off at the outer edges. Evidently large pieces of AP at the outer edges are removed during the quench process. In fact, the motion pictures show what appear to be thermal stress cracks propagating out into the AP ahead of the leading edge of regression and large pieces of the AP at the outer edges breaking off and being carried away by the nitrogen flow. Thus it would be very easy to lose the outer details of the profile during the quench process. The flame is hardly visible for PS. It is very faintly luminous and has a bluish color. There are a few traces of orange flame adjacent to the binder. Flickers of this orange flame are visible along the entire length of the binder, moving back and forth and from side to side along the binder. Varney noted that PS sandwiches had much less
Figure 12. Surface Profiles of PS Sandwiches at 300 psig and 600 psig.
carbonaceous residue out over the AP surface following a quench than did the other three binders tested. The fact that PS sandwiches burn with such a clean flame with very little apparent soot evidently accounts for his observations.

**PS Sandwich Deflagration at 600 psig**

The burn rate of the PS sandwich is slightly higher than Boggs' results for pure AP crystals, but less than Varney's results for similar thickness PS sandwiches. Using the sandwich profile analysis previously described, the AP regression rate normal to the surface of the sandwich is found to be approximately the same as for pure AP crystals. Thus the AP in the sandwich is evidently regressing similarly to pure AP. A typical profile for a PS sandwich deflagrating at 600 psig is shown in Figure 12b. The profile is similar to that obtained by Varney near the binder, but slightly higher corners were observed in the motion pictures. This again, as at 300 psig, suggests that portions of the sandwich were expelled during the quench process. The slope is slightly steeper than for the monopropellant profile, but due to the slightly higher burning rate, as mentioned above, the AP surface is regressing as pure AP. This indicates that the base flame is possible adding a small amount of energy which results in the slightly higher vertical burn rate, but that the AP flame is controlling the majority of the sandwich regression. Again, as at 300 psig, the diffusion flame probably decomposes the binder, but adds no energy to the sandwich surface. There is a much more visible flame than at 300 psig. The bluish flame as well as the orange flame are now clearly visible. The orange flame is much more prominent and stable than at 300 psig.
PS Sandwich Deflagration at 1000 psig

The burn rate of the PS sandwich is approximately the same as the burn rate measured by Boggs for pure AP crystals, but is lower than measured by Varney for PS sandwiches. The AP is regressing similarly to pure AP except at the corners where a slight corner effect is observed. The regression of the corners is probably inhibited by a combination of the convective cooling of the nitrogen and the corner heat transfer phenomena. A typical profile for a PS sandwich deflagrating at 1000 psig is shown in Figure 13a. The profile is similar to that observed by Varney. The profile is similar to the monopropellant profile except at the corners. This possibly indicates that the AP flame is controlling the sandwich regression. The base flame and diffusion flame are probably providing heat for binder decomposition, but not adding any significant amount of heat to the sandwich surface. The flame is now more visible. The larger portion of the flame is bluish in color while orange flame is visible adjacent to the binder. An AP surface melt is visible at this pressure level. This melt was possibly also present at the lower pressure levels although it was not visible in the motion pictures. The lower pressure level runs were photographed at 1:1 magnification whereas a 2:1 magnification motion picture was made at 1000 psig. The details of the sandwich surface are much more visible at the 2:1 magnification, so this possibly explains why AP surface melts were not observed at the lower pressure levels.

PS Sandwich Deflagration at 1500 psig

The burn rate of the PS sandwich is similar to Boggs’ results for pure AP crystals, but is lower than the burn rates obtained by Varney
Figure 13. Surface Profiles of PS Sandwiches at 1000 psig and 1500 psig.
for PS sandwiches. A typical profile for a PS sandwich deflagrating at 1500 psig is shown in Figure 13b. This profile is similar to the monopropellant profile except at the corners which peak above the monopropellant profile similarly to the corners observed at the lower pressures. The profile is similar to the PS profile observed by Varney at 1500 psig with the exception that the corners extend more above the AP surface than they did for the quenched profile. As at 1000 psig, the AP flame is probably controlling the sandwich regression, while the base flame and diffusion flame are only decomposing the binder. The visible flame is similar to the 1000 psig flame. No AP surface melt was observed and as previously noted, this was probably due to the 1:1 magnification used at 1500 psig.

**PS Sandwich Deflagration at 2000 psig**

The burn rate of the PS sandwich is similar to that measured by Boggs for pure AP crystals, but less than that obtained by Varney for PS sandwiches. A typical profile for a PS sandwich deflagrating at 2000 psig is shown in Figure 14a. This profile is similar to both the monopropellant profile and to Varney's quenched PS sandwich profile at 2000 psig. Thus the AP flame is probably controlling the sandwich regression, while the base flame and diffusion flame serve only to decompose the binder. The visible flame is similar in appearance to those at 1000 and 1500 psig. No AP surface melt was observed since this run was photographed at 1:1 magnification.

**PS Sandwich Deflagration at 2400 psig**

The burn rate of the PS sandwich is much greater than Boggs' burn rate for pure AP crystals, but is less than burn rates obtained by Varney
Figure 14. Surface Profiles of PS Sandwiches at 2000 psig and 2400 psig.
for PS sandwiches. As Figure 1 shows, this pressure is in Boggs' Zone III where the burn rate for pure crystal AP is decreasing with increasing pressure. The burn rate for pure crystal AP is 0.18 in/sec at 2400 psig, however, the burn rate normal to the AP surface, calculated using the sandwich profile analysis previously described, is approximately 0.5 in/sec. Thus the AP regression rate is much greater in the sandwich configuration than in the monopropellant state at 2400 psig. A typical profile for a PS sandwich deflagrating at 2400 psig is shown in Figure 14b. The profile is similar to both the monopropellant profile and to the profile observed by Varney for a PS sandwich at 2400 psig with the exception that the corners extend well above the AP surface for the profile obtained through this study. A possible explanation for this profile and the burn rates described above will be given at the end of this section in General Discussion. The visible flame description is similar to the flame description at 1000, 1500 and 2000 psig. No AP surface melt was observed since this run was photographed at 1:1 magnification.

PS Sandwich Deflagration at 2800 psig

The burn rate of the PS sandwich is much greater than Boggs' results for pure AP crystals. Varney only tested to 2400 psig so no comparison can be made to his results. This pressure is in Boggs' Zone III where the burn rate for pure AP crystals is decreasing with increasing pressure. The burn rate for pure AP crystals is 0.14 in/sec at 2800 psig, however the burn rate normal to the AP surface, calculated using the sandwich profile analysis previously described, is approximately 0.5 in/sec. Thus the AP regression rate is much greater in the sandwich configuration than in the monopropellant state at 2800 psig. A typical
profile for a PS sandwich deflagrating at 2800 psig is shown in Figure 15a. The profile has a slightly steeper slope than the monopropellant profile as well as corner effects extending well above the AP surface. It appears that the influence of the base flame on the profile is slightly greater than at 2400 psig. A possible explanation for this profile and the burn rates described above will be given at the end of this section in General Discussion. The visible flame has now become a more consistent mixture of bluish and orange flame than at lower pressures. This test was photographed at 2:1 magnification and an AP surface melt was observed.

**PS Sandwich Deflagration at 3200 psig**

The burn rate of the PS sandwich is much greater than Boggs' results for pure AP crystals. This pressure is greater than the 2400 psig limit of Varney so no comparison can be made to his results. This pressure is in Boggs' Zone III where the burn rate of pure AP crystals is decreasing with increasing pressure. The burn rate for pure AP crystals is 0.12 in/sec in 3200 psig, however, the burn rate normal to the AP surface, calculated using the sandwich profile analysis previously described, is approximately 0.4 (corresponding to \( r_v = 0.5 \) in/sec). Thus the AP regression rate is much greater in the sandwich configuration than in the monopropellant state at 3200 psig. The difference between the AP burn rates in the sandwich and in the monopropellant state, although still large, has decreased from the difference at 2400 and 2800 psig.

A typical profile for a PS sandwich deflagrating at 3200 psig is shown in Figure 15b. The profile has a much steeper slope than the monopropellant profile and now merges fairly smoothly with the corners. The base
Figure 15. Surface Profiles of PS Sandwiches at 2800 psig and 3200 psig.

(a) 70 µ PS Sandwich at 2800 psig.

(b) 80 µ PS Sandwich at 3200 psig.
flame seems to be adding a greater portion of the combustion energy than at 2400 or 2800 psig. A possible explanation for this profile and the burn rates described above will be given at the end of this section in General Discussion. The visible flame is very stable. It is orange at the base and then becomes bluish higher up. This test was photographed at 2:1 magnification and an AP surface melt was observed.

**General Discussion of Combustion Pressure Effect**

An AP surface melt was observed for runs photographed at 2:1 magnification; specifically at 1000, 2800 and 3200 psig. An AP surface melt was undoubtedly present at the other pressures tested since Boggs observed AP surface melts between 300 and 2000 psig and since the 2400 psig pressure is bracketed by melts at 2000 and 2800 psig. The fact that this melt was observed at such a high pressure level raises some interesting points. A recent paper by Guirao and Williams [27] has as its main premise that exothermic condensed-phase reactions occurring in a liquid layer at the surface of AP coupled with exothermic gas phase reactions, are responsible for the steady deflagration of the monopropellant in the pressure range from 300 to 1500 psi. They find that this liquid layer provides approximately 70 per cent of the heat release. They conclude that when the surface temperature of the AP drops below the melting point the condensed-phase reaction rates decrease greatly. Boggs [18] concludes that exothermic reactions occurring in a "Pseudo-Condensed" phase, coupled with gas-phase reactions, are responsible for the heat transfer to the deflagrating AP between 300 and 2000 psi. At higher pressures the surface is said to consist of closely packed solid AP "needles" and under these conditions only gas-phase reactions are
inferred to feed back energy to the surface. The fact that the AP surface melt is observed for sandwich combustion at pressures up to 3200 psig, which is in the "needle" region, indicates that the sandwiches deflagrate differently than does pure AP at these high pressures. Possibly the base flame provides sufficient energy to forestall the formation of the "needle" structure and to maintain the AP surface heating rate high enough to maintain an AP surface melt. Thus the exothermic condensed-phase reactions may continue to provide energy to aid the sandwich combustion at pressures above 2000 psi. This helps explain why the sandwich burn rate is so much greater than the pure AP burn rate at these high pressures. Another possible explanation is that possibly the compacted polycrystalline AP was contaminated with an impurity such as potassium. Boggs and Zurn [44] stated that very small amounts of impurities such as potassium greatly alter the burn rate and surface melt of AP. Between 2000 and 4000 psi, where the AP burn rate is decreasing with increasing pressure, potassium addition increases the burn rate enough to eliminate the dip in the curve (see Figure 1). The AP surface melt is found to be present at pressures up to 5000 psi, as opposed to ceasing at 2000 psi for pure AP. For both of these possibilities it seems that the surface melt is a major factor in increasing the burn rate in this pressure range. Thus it is plausible that this AP surface melt is present in this pressure range for sandwiches and three-dimensional propellants, regardless of how it is formed, and that this is the reason that burning rates higher than pure AP rates are obtained. The possibility of potassium impurities is questionable in this investigation since the sandwich burn rates obtained agreed with Boggs' pure
AP crystal burn rates rather than agreeing with the erratic burn rates of potassium doped AP at pressures below 2000 psi (see Boggs and Zurn [44]).

The surface profiles compare fairly well with those obtained through Varney's quench testing. It does appear that Varney lost all corner effects during the quench process. The surface profiles obtained indicate that near the low pressure deflagration limit of AP, at 300 psig, the base flame is the dominant heat source for the combustion process. In the mid-pressure range, 600 to 2000 psig, the AP flame appears to be the dominant heat source, while the base and diffusion flames' contribution is decomposition of the binder. The kinetics of the base flame are possibly not fast enough at these pressures to increase the sandwich regression near the interface. At higher pressures, 2000 to 3200 psig, the base flame again appears to be the dominant heat source. The kinetics of the base flame are greater at these higher pressures and thus the flame is moved closer to the AP surface. The increased burn rate for the AP in the sandwich configuration, as previously discussed, probably results from the AP surface melt observed at these pressures. The base flame possibly provides sufficient energy to forestall the formation of the AP "needle" structure and to maintain the surface heating rate high enough to maintain an AP surface melt. The AP melt seems to decrease slightly from 2400 to 3200 psig as the pressure is increased, based on the observed decrease in AP burn rate. The base flame seems to maintain a fairly constant vertical sandwich burn rate, but the effect of the base flame on the AP appears to decrease with increasing pressure.
Binder Melts

Varney [13] observed binder melts with sandwiches composed of all four binders under consideration. As his observations were only of the quenched sandwich surfaces, it was desirable that visual substantiation of these binder melts be obtained. A series of tests was performed at 1000 psig, the object being to visually locate binder melts during combustion. All four binders were investigated. The binder melts were judged to be present when they were visually observed and when the base of the visible flame appeared to emanate from the AP surface rather than from the binder/AP interface area. The latter situation would probably result if the binder melt flowed out over the AP surface and was then decomposed, the hot soot particles generated in the reaction then showing up as bright orange flamelets.

PS Binder Melt

Although Varney found through DSC testing that PS melts the most readily of the four binders and observed binder melt out over the AP surface for quenched PS sandwiches, little evidence of this melt was found in the motion picture study. Steinz, Stang and Summerfield [12] observed that extinguished PS propellant strands also exhibited a binder melt which flowed over the surface. A binder melt near the edge was present during a few of the PS tests, but this is believed to be an edge effect, with the binder not vaporizing due to the cool nitrogen flush. Views looking down on the sandwiches show no evidence of this melt back toward the interior of the samples. A possible PS binder melt was observed in one test and that was only for a brief moment. An orange flame was observed to move out from the visible flame, over the AP
surface. This indicates that possibly a small binder melt flowed out onto the AP surface and was then consumed. A typical flame structure for a PS sandwich is shown in Figure 16a. The base of the visible flame is located near the binder-oxidizer interface and no edge melt is visible. The lack of evidence supporting a PS binder melt flow in this experiment suggests that perhaps observations of a PS melt for extinguished samples resulted from quick cooling of the PS and flow out over the AP surface during the quench process.

**PU Binder Melt**

The PU sandwiches tested show evidence of a fairly substantial binder melt, although not nearly as substantial a binder melt as obtained by some other investigators [45]. A PU sandwich profile is shown in Figure 16b. An edge melt is present and the visible flame base is well out over the AP surface. This indicates that a binder melt flowed out over the AP. The visible flame of the PU sandwiches is bright orange in color and is not transparent as for PS. This evidently prevents observation of the binder melt using a top surface view, as the melt is covered by the orange flame. The orange color of the flame apparently results from hot soot particles generated by the reaction.

**PBAA Binder Melt**

A substantial binder melt is in evidence for the PBAA sandwiches. A PBAA sandwich profile is shown in Figure 17a. An edge binder melt is present and the base of the visible flame is well out over the AP surface on both sides of the binder. Thus a large binder melt is indicated, but observing the melt toward the interior of the sandwich is again impossible due to the bright orange PBAA flame.
Figure 16. Binder Melts for PS and PU Sandwiches.
(a) 100 $\mu$ PBAA Sandwich at 1000 psig.

(b) 90 $\mu$ CTPB Sandwich at 600 psig.

Figure 17. Binder Melts for PBAA and CTPB Sandwiches.
CTPB Binder Melt

Evidence of a binder melt for CTPB sandwiches is similar to that for PBAA sandwiches. A CTPB sandwich profile is shown in Figure 17b.

General Discussion of Binder Melts

Evidence of large binder melts is observed for PU, PBAA and CTPB. No appreciable binder melt evidence is found for PS. The fact that the base of the visible flame is well out over the AP surface, rather than at the binder-oxidizer interface area, appears to be rather conclusive proof of a binder melt for PU, PBAA and CTPB. The binder has to get out there in some way and the only feasible way is for it to flow as a liquid over the AP surface. The orange flame emanating from the AP surface appears to be several flamelets, rather than a single flame. Thus the binder melt seems to cause an unsteady combustion process where the binder flows out around particles of AP thus generating a series of small flamelets.

The large edge binder melts observed are sometimes consumed by the visible flame and at other times appear to hinder the AP regression under the melts. The majority of the large edge melts do not appear to affect the combustion process or to be affected by the combustion. As mentioned earlier, these large edge melts seem to be formed by the cool nitrogen gas inhibiting vaporization of the binder at the edges only as they do not appear toward the interior of the sandwiches. The fact that the melts do at times hinder the AP regression beneath them was also observed with PS propellant strands as noted by Steinz, Stang and Summerfield [12]. They observed that when anomalous burning behavior
was obtained that the propellant surface after extinguishment was covered with a molten fuel layer. These observations suggest that it is the molten fuel layer that causes the intermittent burning behavior; it would do so by covering the AP particles exposed at the regressing propellant surface, thereby depleting the gas phase flame of the necessary oxidizer gases.

Flame Structure

Flame structure was examined for all four binders. The results are indicated below.

PS Flame Structure

The PS sandwich visible flame is transparent. The color is bluish with traces of orange near the binder surface. At the same frame rate the flame appears to be unstable, just jumping around, near the AP low pressure deflagration limit, but becomes steady at higher pressures. The base of the visible flame emanates from near the interface between the binder and oxidizer except for isolated instances as discussed in the Binder Melt Section. The PS flame is a very clean flame with very little soot formation. The PS sandwiches were also noted by Varney as having very little carbonaceous residue on the AP surface following the quenching process.

PU Flame Structure

The PU sandwich visible flame is bright orange in color and is opaque. The bright orange color apparently results from soot particles which are generated by the reaction. Varney also noted that the AP surface of PU sandwiches was covered with a carbonaceous residue following
the quenching procedure. The PU flame is relatively short compared to PS, PBAA and CTPB flames, especially at higher pressures. A comparison between the flame height of a PU and a PBAA sandwich with equal thickness binder layers deflagrating at 2400 psig is shown in Figure 18. In some of the tests with PU the visible flame did not form a closed flame over the binder, but remained as two short, separate flames separated by the binder. This is shown in Figure 18a.

**PBAA Flame Structure**

The PBAA sandwich visible flame is a bright yellow-orange color and is opaque. The flame is taller than the PU flame as shown in Figure 18. Again the orange color apparently results from the hot soot particles and this creates the carbonaceous residue observed by Varney. The characteristic dip in the surface profile as obtained by Hightower and Price [24] for PBAA sandwiches is not as evident for PS sandwiches. A comparison may be seen in Figure 19. The difference in surface profiles for the two binders is evidently due to the fact that the PBAA has a much larger energy of decomposition than the PS [13] and therefore is a greater heat sink.

**CTPB Flame Structure**

The CTPB flame structure is similar to the PBAA flame structure.

**General Discussion of Flame Structure**

The only visible part of the sandwich flame structure for all four binders is the upper portion of the diffusion flame and luminous reaction particles. The base flame is not visually distinguishable from the upper portion of the diffusion flame and the AP flame is not visible at all. Orange flamelets, as discussed in the Binder Melt section, are
Figure 18. Comparison of PU and PBAA Flame Structure at 2400 psig.
Figure 19. Comparison of PS and PBAA Sandwich Profiles.
also visible when binder melts occur. Since the upper portion of the diffusion flame is the only visible flame and it is assumed not to provide heat for sandwich regression, the value of observing the flame structure becomes secondary to observing surface profiles and surface conditions. The upper portion of the diffusion flame does give some indication of the possible location of the base flame, but the surface profile gives a better indication. The flamelets provide an indication of binder melts and their extent. Another way in which observation of the flame structure may be beneficial is through the shape, coloration and character of the visible flame. For example, the visible flame could be short and rounded or tall and thin, tints of various colors could be observed with catalyst addition indicating effects on the gas phase reactions, and the flame could be stable or unstable. Some of these different effects might possibly be observed with catalyst addition as discussed in Chapter V.

**Additional Results**

Several asymmetric profiles were noted by Varney [13]. The majority of these asymmetric profiles occurred for thin binder layers, although one case of asymmetry was noted for a thick binder layer. Hightower and Price [24] noted that all thin binder layer PBAA sandwiches tested by them had asymmetry, whereas this asymmetry was not present for thick binder layer sandwiches. The lagging surface of the AP in the asymmetric case was observed to have a very thin binder layer on its surface. High speed motion pictures showed this asymmetry to be present during burning, with a visible flame distributed above the lagging surface. The general nature of the flame structure was unstable.
and the flame moved back and forth across the binder layer. Asymmetries have been observed in some of the motion picture runs of this experiment. Asymmetries appear to be due to no unsteadiness property of the combustion process, but seem to occur due to the following conditions:

1. Bad ignition - Sample profile remains steady, but asymmetric throughout run.

2. Sandwich flaw.

3. Large binder melt - Large binder melts appear at times to slow AP regression underneath the melt.

An exception to the above is the very thin binder sandwiches for which asymmetries developed due to unsteadiness. For these sandwiches the flame was unstable, moving from one side of the binder to the other. No explanation for this phenomena has been determined. This type of asymmetry is similar to that noted by Hightower and Price for very thin binder layers.

In many cases it is possible to estimate the binder height above the AP surface during combustion by observation of the motion pictures. At times, however, the actual binder height can not be obtained due to edge effects, where only a thin peak of binder at the edge extends above the normally regressing binder. The motion pictures indicate larger binder heights than were indicated by Varney's quench testing. Apparently much of the binder is ejected during the quench process, especially at the higher pressures. The Emmons Problem [46] was applied to the sandwich configuration in order to determine whether or not the simplified picture of a diffusion flame consuming the binder is valid. Appendix F contains details for the use of the Emmons problem. Data for the heat
of decomposition of the binders and binder surface temperatures were taken from the DSC results of Varney [13]. Table 1 shows the results of the computations. For both PS and PU binders the heights are predicted rather well (within the accuracy for which the binder height can be determined by the motion pictures). For PBAA, however, there is a large discrepancy believed to be due to the large heat of decomposition (1950 cal/g) obtained from Varney's DSC results.

Varney [13] noted that thin PBAA and CTPB quenched sandwiches developed a plateau of AP adjacent to the binder for tests run at pressures of 2000 and 2400 psig. This effect was examined with motion pictures using a 20 micron CTPB sandwich at 2400 psig. An AP plateau type profile for this sandwich is shown in Figure 20. Varney questioned whether this effect was due to an unsteady phenomena. The 20 micron CTPB sandwich appears to have no true steady state profile, the profile varying throughout the run. The profile begins with fairly planar regression, goes to an AP plateau type profile, develops large edge effects along with no AP plateau, then goes back to small edge effects with an AP plateau again. The visible flame appears to be unstable, jumping around and at times appearing only on one side of the binder. The flame is very short. Thus Varney's postulate of some unsteady phenomena causing this effect appears to possibly be the case. The unsteadiness possibly results from the thin binder layer causing an unstable flame. Perhaps this is a phenomenon similar to the thin binder asymmetry of Hightower and Price [24] previously discussed. As evident from the preceding possibilities postulated, this phenomenon is not really understood and remains a mystery.
Table 1. Results of Theoretical Binder Height Calculations Using the Emmons Problem

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<th>Type Binder</th>
<th>Binder Thickness (microns)</th>
<th>Test Pressure (psig)</th>
<th>Theoretical Binder Height (inches)</th>
<th>Experimental Binder Height (inches)</th>
<th>Binder Energy of Decomposition (cal/gm)</th>
<th>Ratio of Theoretical Height to Experimental Height</th>
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<td>PBAA</td>
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<td>1000</td>
<td>0.248</td>
<td>0.029</td>
<td>1950</td>
<td>8.55</td>
</tr>
</tbody>
</table>
20 μ CTPB Sandwich at 2400 psig.

Figure 20. AP Plateau.
Varney [13] questioned whether sandwiches achieved a steady state burning configuration and if so how quickly following ignition? The majority of the motion picture tests indicate that a semi-steady state is attained, usually within 0.1 inch from the ignition point. It is only semi-steady since the actual profile is not constant, but has small varying undulations; however, the average profile is essentially steady. An exception to this semi-steady state attainment is for thin binder layers, specifically at higher pressures as mentioned earlier in the AP Plateau discussion.

Additional Interpretation of Results

Results of this experimental investigation indicate that high speed cinephotomacrography of sandwich combustion is an effective research tool. Varney's [13] quench testing provided excellent results, but many findings were questionable due to the quench process. The unsteady effects of the quench process were unknown and Varney was unable to actually observe the combustion process. The quench testing does appear to be superior to photographic testing in obtaining interfacial and surface details. Although the basic sandwich profiles obtained with motion picture testing and quench testing are fairly similar, the quench testing loses portions of the AP surface and binder peak during the violent quench. Also, in the case of PS, quench testing appears to create a non-combustion process, binder melt flow, during the quench. The photographic testing provides details of the combustion process not obtainable with quench testing such as verifying the attainment of steady state, observing flame structures, finding reasons for asymmetry and more accurately measuring burn rates. It appears
that a test program composed of quench and photographic tests with sandwiches would be an excellent method of obtaining greater insight into the composite solid propellant combustion process.

Some of the more significant results of this experimental program are those involving binder melts, flame structure and AP melts. The results indicate that binder melts flow out over the AP surface during combustion, at least for PU, PBAA and CTPB. This flow significantly alters the flame structure. The reaction zone of the binder/AP diffusion flame is moved out from the binder over the AP surface. The possibility also exists for a pre-mixed AP melt/binder melt flame. The visible flame structure observed appears to be a binder/AP diffusion flame composed of binder pyrolysis products and AP combustion products. The region at the bottom of the diffusion flame, the base flame, as stated earlier probably controls the heat addition to the AP surface resulting from the presence of the fuel binder. The fact that no burning rate change occurs with binder thickness changes, as opposed to Varney's quench results, supplements the evidence that the binder is consumed by a diffusion flame mechanism. Also, at least for PS and PU (at 1000 psig), the results of applying the Emmons Problem indicate that a diffusion flame is consuming the binder. For PS, the base flame appears to play a significant role at low pressures near the lower deflagration limit of AP and at high pressures where the slope of the AP burn rate curve is negative. The AP flame appears to control the combustion process at other times. The diffusion flame appears to play no real major role, other than consuming the binder.
The observation of AP melts at pressures where the slope of the AP burn rate curve is negative and the surface pattern for pure AP is a needle-like structure indicates that the AP in sandwiches behaves differently from pure AP. The base flame possibly provides sufficient heat to delay the "needle" structure formation and to form the exothermic AP surface melt, thus maintaining the burn rate at a level above that for pure AP at these pressures.

The sandwich combustion model chosen for the analysis appears to provide logical answers for a majority of the combustion aspects. This, along with Varney's results, suggest that a realistic model for binder/AP composite propellant combustion would consist of:

1. Binder melt flows onto the AP surface.
2. AP melts, even in the pressure range where the AP burn rate is decreasing with increasing pressure.
3. Three basic flames; AP monopropellant flame, base flame beneath the diffusion flame which is elliptic in character and a diffusion flame between AP combustion products and binder decomposition products.
CHAPTER V

EXPLORATORY HIGH SPEED MOTION PICTURE STUDY OF
CATALYZED AP/BINDER SANDWICHES

The second half of this experimental effort consisted of an exploratory catalyst effect study. Catalysts investigated were Harshaw Catalyst Cu O202 (copper chromite - CC) and Fe2O3 (iron III oxide - IO). Both of these catalysts tend to increase the burn rate of catalyzed AP over that of pure AP. Friedman, et al. [30] noted that CC increases the burn rate for all pressure levels, whereas IO decreases the burn rate at low pressures while increasing it at high pressures. Therefore, both high and low pressure levels were investigated. These two catalysts were chosen because of the availability of unclassified literature concerning their use. The origins of the use of CC and IO in AP type composite propellants is not available in the unclassified literature. The binder chosen for this study was CTPB. This binder was chosen as it is the most advanced of the four being considered in this investigation. The oxidizer was compacted, polycrystalline AP as in the previous tests. Five types of catalyst addition were tested:

1. Type 1, no catalyst.
2. Type 2, 2 per cent by weight in the AP alone.
3. Type 4, some volumetric loading in the binder alone as occurred in the AP in Type 2.
4. Type 5, same volumetric loading in both the AP and binder as in Type 2.
5. Type 6, a paint of catalysts on one interface of the AP before curing the binder between the AP slabs. A Type 3, which contained 2 per cent by weight catalyst in the binder alone, was not investigated since the Type 4 addition was judged to be a more realistic comparison case between catalyst in the binder and a similar amount of catalyst in the AP. These various methods of catalyst addition are somewhat similar to those which Steinz, Stang and Summerfield [12] indicated would be beneficial to the understanding of catalytic mechanisms. Tests were conducted at 600 psig and 2000 psig in order to determine pressure effects. A frame rate of 1600 pictures per second and magnification of 2:1 were used. As stated in Chapter IV, edited films are available on loan if so desired since space and format considerations preclude the presentation of the cinematomacrography results.

The major goal of this portion of the investigation was to provide a foundation on which future catalyst experiments could be based. The motion pictures were examined for differences in burn rates, surface profiles and flame structures resulting from the different methods of catalyst addition. The analysis made use of the sandwich profile analysis, as described in Chapter IV, in order to determine catalytic effects on the AP in the sandwich configuration. The sandwich combustion model, described in Chapter IV, was also used as an aid in interpreting the results.

Various mechanisms have been postulated for catalytic action on AP and composite propellants. The basic premise of these mechanisms is that the catalysts enhance the heat release rate and raise the
surface temperature of the propellant. This is postulated to occur in a number of ways:

1. Through accelerating the gas phase reaction. This has been suggested by Levy and Friedman [32], Pittman [36] and Pearson [37].

2. Through exothermic reactions of gases on the surface of catalyst particles imbedded in the propellant (heterogeneous or not). This has been suggested by Pearson [37] and Steinz, Stang and Summerfield [12].

3. Through promoting heterogeneous reactions of gases with solid fuels. This has been suggested by Pearson [37] and Pearson and Sutton [38].

4. Through modifying the pyrolysis mechanism of the solid fuel. This has been suggested by Pearson [37].

5. Through gas phase or heterogeneous reactions promoted by the presence of catalyst particles in crevices between solid fuel and solid AP (at the interface). This has been suggested by Steinz, Stang and Summerfield [12].

The results of the analysis were reviewed, considering the various types of catalyst addition, and possible mechanisms for catalytic action were postulated. The justification for these possible mechanisms was reasoned as follows:

1. Acceleration of gas phase reactions -- The AP flame would be accelerated if the AP burn rate normal to the sandwich surface were accelerated, this acceleration varying with pressure. The base and diffusion flames would be accelerated if interfacial area regression were obtained. Also, changes in the color and character of the visible
flame would indicate that the catalyst was affecting the gas phase in some manner.

2. Exothermic reactions of gases on the surface of catalyst particles imbedded in the propellant -- This would be evidenced by an increase in AP or binder regression rate. Changes in pressure should produce changes in catalytic effectiveness.

3. Heterogeneous reactions of gases with solid fuels -- This should be observed more easily when catalyst particles are in the binder and have been exposed to the gas phase reactions. The height of the binder above the surface should decrease as compared to the non-catalyzed case. Pressure changes should have some effect.

4. Modification of the pyrolysis mechanism of the solid fuel -- Indication of this mechanism should be similar to item 3 above.

5. Gas phase or heterogeneous reactions promoted by the presence of catalyst particles in crevices between solid fuel and solid AP (at the interface) -- This would be indicated by interfacial regression. If gas phase, the pressure changes should affect the amount of interfacial regression more than if the reactions were heterogeneous.

Results for Copper Chromite

The burn rates determined for the various types of catalyzed sandwiches are shown in Figure 21 for CC addition. Included on this figure is a curve showing Boggs' [18] results for pure AP. These burn rates are for vertical regression and are based on the leading edge of regression.
Figure 21. Burn Rate for Copper Chromite Catalyzed Sandwiches.
Copper Chromite at 600 psig

**Type 1.** This non-catalyzed CTPB sandwich is used as a base case to which the various catalyzed sandwiches may be compared. The burn rate is approximately the same as the burn rate measured by Boggs for pure AP crystals. A representative surface profile for this sandwich is shown in Figure 22a. The profile is approximately the same as the theoretical monopropellant profile shown. The flame is a bright orange in color and is rather short. The visible flame edges are well out over the AP surface.

**Type 2.** The sandwich burn rate is increased approximately 50 per cent over the uncatalyzed Type 1 sandwich burn rate with the addition of CC to the AP. The catalyzed AP burn rate normal to the sandwich surface is found to be approximately 25 per cent greater than the pure AP crystal burn rate. The profile for this sandwich with CC addition in the AP is shown in Figure 22b. The profile is much steeper than the monopropellant profile. Interfacial regression is possibly indicated. The flame is much more violent and spread out over the AP surface than for the uncatalyzed Type 1 sandwich. The flame is still bright orange, but now has a purplish tint.

**Type 4.** The sandwich burn rate is similar to the Type 1 sandwich burn rate. Figure 23a shows a representative profile for this sandwich with CC in the CTPB. The right side of this sandwich regressed irregularly and is not considered a representative profile. The sandwich profile indicates sandwich regression exceeding pure AP, but within the accuracy of visually measuring the burn rate, no increase was noted. Near the interface the AP surface slope is very steep.
Figure 22. Surface Profiles for Copper Chromite Catalyzed CTPB Sandwiches, Types 1 and 2, at 600 psig.
Figure 23. Surface Profiles for Copper Chromite Catalyzed CTPB Sandwiches, Types 4 and 5, at 600 psig.
Interfacial regression is possibly indicated. The apparent binder peak height does not appear to be reduced from the uncatalyzed binder peak height. The flame is smoother (not as turbulent) than the uncatalyzed Type 1 sandwich. The color is bright orange as for Type 1.

Type 5. The burn rates, surface profile and flame description are similar to those for Type 2. A representative profile is shown in Figure 23b.

Type 6. The sandwich burn rate is similar to the Type 1 sandwich burn rate. This sandwich has a paint of CC catalyst at the left side interface. A representative profile is shown in Figure 24. The profile is similar to the monopropellant profile and to the Type 1 sandwich except at the catalyzed interface. At the catalyzed interface the regression rate appears to be greater than at other points. Interfacial regression is possibly indicated. The flame appears to be farther out over the AP surface on the catalyzed side. There is a slight purplish tint to the catalyzed side of the bright orange flame. The flame is much higher than for Type 1, but this is probably due to the thicker binder layer for this Type 6 sandwich.

Copper Chromite at 2000 psig

Type 1. This non-catalyzed CTPB sandwich is also used as a base case to which the various catalyzed sandwiches may be compared. The sandwich burn rate is similar to that measured by Boggs for pure AP crystals. A representative surface profile for this sandwich is shown in Figure 25a. The profile is similar to the monopropellant profile far from the binder near the sandwich edges. Near the binder the regression appears to be slowed. The flame is bright orange in color.
Figure 24. Surface Profile for a Copper Chromite Catalyzed CTPB Sandwich, Type 6, at 600 psig.
Figure 25. Surface Profiles for Copper Chromite Catalyzed CTPB Sandwiches, Types 1 and 2, at 2000 psig.
It is fairly turbulent and is not very tall.

**Type 2.** The sandwich burn rate is increased approximately 300 per cent over the Type 1 sandwich burn rate. The catalyzed AP burn rate is approximately 150 per cent greater than the pure crystal AP burn rate. Figure 25b shows a profile for this sandwich with CC in the AP. The entire profile is similar to the monopropellant profile. The flame is very tall and has the appearance of two laminar jets. The base of the flame is well out over the AP surface. The color of the flame is bright orange with purplish tints. The flame is much more stable than at 600 psig.

**Type 4.** The sandwich burn rate is similar to the Type 1 sandwich burn rate. The profile for this sandwich with CC in the binder appears in Figure 26a. The profile is similar to the Type 1 profile except the upslope near the binder has been considerably reduced. The apparent binder peak height does not appear to be different from the 600 psig case or the uncatalyzed case. The flame is fairly tall and is bright orange in color with some very faint purplish tints at times.

**Type 5.** The burn rates, surface profile and flame description are similar to those of Type 2. A representative profile is shown in Figure 26b.

**Type 6.** The sandwich burn rate at the catalyzed interface is approximately 50 per cent greater than the Type 1 sandwich burn rate. However, the AP burn rate normal to the sandwich surface is similar to the pure AP crystal burn rate. This sandwich has a paint of CC catalyst at the left side interface. A representative profile is shown in Figure 27. The right side AP surface, with no CC at the interface, is
(a) 90 μ Type 4 CC Sandwich.

(b) 100 μ Type 5 CC Sandwich.

Figure 26. Surface Profiles for Copper Chromite Catalyzed CTPB Sandwiches, Types 4 and 5, at 2000 psig.
170 μ Type 6 CC Sandwich.

Figure 27. Surface Profile for a Copper Chromite Catalyzed CTPB Sandwich, Type 6, at 2000 psig.
regressing similarly to pure AP, while the left side, with CC at the interface, indicates a greatly increased regression rate. Interfacial regression on the catalyzed side is possibly indicated. The flame is bright orange with a purplish tint on the catalyzed side. It is fairly straight up on the catalyzed side, but is fairly violent on the uncatalyzed side.

**Interpretation of Copper Chromite Results**

**Copper Chromite**

**Types 2 and 5.** The sandwich burn rates are greater than for the uncatalyzed sandwiches. Also the catalyzed AP burn rates normal to the sandwich surface are greater than for pure AP crystals. The increase in burn rates is greater at 2000 psig than at 600 psig. Interfacial regression is possibly indicated at 600 psig, but is not evident at 2000 psig. The flame at 600 psig is much more violent and spread out over the AP surface than for the uncatalyzed Type 1 sandwich. A purple tint has been added to the orange flame which was not visible in the uncatalyzed flame. The flame at 2000 psig has similar purple tints, but is more stable than the 600 psig flame. The 2000 psig flame is very tall and has the appearance of two laminar jets. Since the catalyzed AP burn rates normal to the surface are increased over the uncatalyzed rates, the AP burn rate is apparently augmented by the CC addition; the augmentation increasing with pressure. This possibly indicates that the AP flame is accelerated in the gas phase and/or that exothermic reactions of gases on the surface of catalyst particles imbedded in the AP are taking place. Acceleration of the base flame gas phase reactions,
at least at 600 psig, is indicated by the possible interfacial regression. This possible interfacial regression may also indicate gas phase or heterogeneous reactions promoted by the presence of catalyst particles at the interface. The purple tint and the different character of the flames, both with catalyst addition and with pressure change, indicate that the gas phase reactions have been affected in some manner.

**Type 4.** The sandwich burn rates are similar to the uncatalyzed sandwiches. Possible interfacial regression is observed at 600 psig while the upslope of AP near the binder is reduced at 2000 psig. The flame has faint purple tints and is smoother than the uncatalyzed flame at both pressures. The apparent binder height at both pressures did not appear to be different from the uncatalyzed case. The base flame gas phase reactions are possibly accelerated by the CC catalyst as evidenced by the regression near the interface as noted above. The fact that no appreciable change is observed in binder height possibly indicates that there are no heterogeneous reactions of gases with the solid fuel binder and no modifications of the pyrolysis mechanism of the solid fuel.

**Type 6.** The sandwich burn rate is similar to the uncatalyzed burn rate at 600 psig, but is increased at 2000 psig. However, the AP burn rate normal to the surface (away from the interfacial area) is similar to the pure AP crystal burn rate at both pressures. Interfacial regression is possibly indicated at both pressures. The flame is smoother than for the uncatalyzed case and has purple tints on the catalyzed side for both pressures. These results indicate that possibly
the base flame gas phase reactions are accelerated. Also gas phase reactions promoted by the presence of catalyst particles at the interface are possibly occurring. Gas phase, rather than heterogeneous, reactions are possibly evidenced by the large increase in interfacial regression with pressure increase.

For CC catalyzed sandwiches it is concluded that:

1. The CC augments the AP deflagration process at both 600 and 2000 psig, the augmentation increasing with pressure increase. This agrees with the results of Friedman, et al. [30]. This augmentation possibly results from acceleration of the AP flame gas phase reactions as suggested by Levy and Friedman [32] and by Pearson [37]. Another possibility is that exothermic reactions of gases on the surface of catalyst particles imbedded in the AP are taking place as suggested by Pearson [37] and by Steinz, Stang and Summerfield [12].

2. The CC does not promote heterogeneous reactions of gases with the solid fuel binder and does not modify the pyrolysis mechanism of the solid fuel binder. These two possibilities of CC catalytic action were suggested by Pearson [37] and Pearson and Sutton [38]. Thus this conclusion contradicts their suggestions.

3. The CC possibly promotes gas phase reactions by its presence in crevices between solid fuel and solid AP (at the interface) as suggested by Steinz, Stang and Summerfield [12].

4. The CC possibly accelerates the base flame gas phase reactions, thus increasing the heat release rate near the sandwich interface as suggested by Pearson [37].
Results for Iron Oxide

The burn rates determined for the various types of catalyzed sandwiches are shown in Figure 28 for 10 addition. Included on this figure is a curve showing Boggs' [18] results for pure AP. These burn rates are for vertical regression and are based on the leading edge of regression.

Iron Oxide at 600 psig

Type 2. The sandwich burn rate is slightly less than the uncatalyzed Type 1 sandwich burn rate. The catalyzed AP burn rate normal to the sandwich surface is approximately 30 per cent less than the pure AP crystal burn rate. Figure 29a shows a representative profile for this sandwich with 10 in the AP. The profile has a much steeper slope than the monopropellant profile. Interfacial regression is possibly indicated. The flame is very high (thick binder) and is bright orange in color. No observable difference in the flame color is noted between the Type 2 flame and the uncatalyzed Type 1 flame as opposed to Type 2 CC addition. The base of the flame emanates from the binder edge (near the interface). A catalyzed AP melt is observed which has a very sticky appearance. Many black spots are also evident on the AP surface.

Type 4. The sandwich burn rate is similar to the uncatalyzed Type 1 sandwich burn rate. The profile for this sandwich with 10 in the binder is shown in Figure 29b. The profile is similar to the monopropellant profile, which is in turn similar to the uncatalyzed Type 1
Figure 28. Burn Rate for Iron Oxide Catalyzed Sandwiches.
Figure 29. Surface Profiles for Iron Oxide Catalyzed CTPB Sandwiches, Types 2 and 4, at 600 psig.
profile. The apparent binder height is similar to the uncatalyzed binder height. The flame is fairly tall and is bright orange in color. It is fairly stable and emanates from the interfacial area. Occasionally it flashes out over the AP surface.

**Type 5.** The burn rates, surface profile and flame description are similar to those of the Type 2 sandwiches. A representative profile is shown in Figure 30a.

**Type 6.** The sandwich burn rate at the catalyzed interface is approximately 30 per cent greater than the uncatalyzed Type 1 sandwich burn rate. The AP burn rate normal to the sandwich surface is 50 per cent less than the pure AP crystal burn rate. This sandwich has a paint of catalyst at the left side interface. A representative profile is shown in Figure 30b. The right side AP surface, with no IO at the interface, is regressing similarly to pure AP, while the left side, with IO at the interface, indicates a greatly increased sandwich regression rate. Interfacial regression on the catalyzed side is possibly indicated. The flame is bright orange in color. The base of the flame is emanating from the interface on the catalyzed side and is out over the AP surface on the non-catalyzed side. The AP surface on the catalyzed side is covered with black spots. There is no AP melt visible.

**Iron Oxide at 2000 psig**

**Type 2.** The sandwich burn rate is approximately 100 per cent greater than the uncatalyzed Type 1 sandwich burn rate. The catalyzed AP burn rate normal to the AP surface is approximately 50 per cent
Figure 30. Surface Profiles for Iron Oxide Catalyzed CTPB Sandwiches, Types 5 and 6, at 600 psig.
greater than the pure AP crystal burn rate. Figure 31a shows a representative profile for this sandwich. The profile slope is steeper than the slope for the monopropellant profile. Interfacial regression is possibly indicated. The flame is bright orange in color. The base of the flame is anchored at the interface a majority of the time, but at times it moves out over the AP surface. A very sticky melt of AP and IO is observed.

Type 4. The sandwich burn rate is similar to the uncatalyzed Type 1 sandwich burn rate. The profile for this sandwich with IO in the binder is shown in Figure 31b. This profile is similar to the Type 1 profile except that the upslope near the binder is reduced. The apparent binder height is similar to the uncatalyzed binder height. The flame is slightly turbulent and is bright orange in color.

Type 5. The burn rate and flame description are similar to those of the Type 2 sandwich. A representative profile is shown in Figure 32a. This sandwich has a much thinner binder layer than the Type 2 at 2000 psig. This accounts for the much higher binder peak for the Type 2 sandwich.

Type 6. The sandwich burn rate at the catalyzed interface is approximately 30 per cent greater than the uncatalyzed Type 1 sandwich burn rate. This sandwich has a paint of IO catalyst at the left side interface. The AP burn rate normal to the surface on the catalyzed side is approximately 50 per cent less than the pure AP crystal burn rate. A representative profile is shown in Figure 32b. The right side AP surface, with no IO at the interface, is regressing similarly to pure AP near the binder (the outer edge of AP on this side did not
Figure 31. Surface Profiles for Iron Oxide Catalyzed CTPB Sandwiches, Types 2 and 4, at 2000 psig.
Figure 32. Surface Profiles for Iron Oxide Catalyzed CTPB Sandwiches, Types 5 and 6, at 2000 psig.
regress smoothly in this test and is not considered representative), while the left side, with IO at the interface, indicates a greatly increased sandwich regression rate. Interfacial regression on the catalyzed side is possibly indicated. The flame is bright orange in color. It moves down the interface very rapidly. This sandwich does not appear to achieve a true steady state during the observed portion of the test.

**Interpretation of Iron Oxide Results**

**Iron Oxide**

*Types 2 and 5.* The sandwich burn rate is retarded at 600 psig and accelerated at 2000 psig from the uncatalyzed burn rates. The catalyzed AP burn rates normal to the surface at both pressures behave similarly as compared to the pure AP crystal burn rates. Interfacial regression is possibly indicated at both pressures. The flame is bright orange in color with no tints as observed for CC addition. The base of the visible flame appears to emanate mainly from the interfacial area. A sticky melt of AP and IO is observed at both pressures and the AP surface has many black spots on it at 600 psig. At 600 psig the AP flame gas phase reaction is possibly hindered in some way by the catalyst, or the sticky melt of AP and IO observed possibly has exothermic reactions of smaller intensity than for the pure AP melt. At 2000 psig the AP flame gas phase reaction is possibly accelerated by the catalyst and it is possible that exothermic reactions of gases on the surface of catalyst particles imbedded in the AP are taking place. Unfortunately no explanation has been determined in this experiment for why the IO
affects the AP so differently at the two pressures. The possible interfacial regression indicates that the base flame gas phase reaction is accelerated at both pressure levels. Also the possibility exists for gas phase or heterogeneous reactions promoted by the presence of catalyst particles at the interface. The black spots appearing on the AP surface are possibly due to extensive carbonaceous residue from the diffusion flame or are possibly decomposition sites as observed for low temperature decomposition of AP [47].

Type 4. The sandwich burn rates are similar to the uncatalyzed burn rates. The upslope of AP adjacent to the binder is slightly reduced in the catalyzed case at 2000 psig. The apparent binder heights are unchanged from the uncatalyzed binder height. The base flame gas phase reactions are possibly accelerated, at least at 2000 psig, thus increasing the heat release rate near the interface. Based on the unchanged binder height for the catalyzed sandwiches there are probably no heterogeneous reactions of gases with the solid fuel binder and no modifications of the pyrolysis mechanism of the solid fuel binder.

Type 6. The sandwich burn rates at the catalyzed interface are greater than the uncatalyzed sandwich burn rate. The amount of increase does not seem to vary with pressure changes. The AP burn rates normal to the sandwich surface on the catalyzed side are less than the pure crystal burn rate and the amount of decrease does not seem to vary with pressure changes. Interfacial regression is possibly observed on the catalyzed side of the sandwiches. These results indicate that possibly the base flame gas phase reactions are accelerated. Also heterogeneous reactions promoted by the presence of catalyst
particles at the interface are possibly occurring. Heterogeneous, rather than gas phase, reactions are possibly evidenced by the burn rates not varying with pressure changes. The black spots appearing on the AP surface on the catalyzed side of the sandwich are possibly due to extensive carbonaceous residue from the diffusion flame or are possibly decomposition sites as observed for low temperature decomposition of AP [47]. There is a slight possibility that the methyl alcohol used in applying the catalyst layer possibly hinders the AP deflagration in some way. This is doubtful, however, since a sample of sandwich size, pure, pressed polycrystalline AP was coated with methyl alcohol and no differences in burn rate or surface profile were observed as compared to uncoated pure AP. Also, the AP burn rate normal to the sandwich surface on the catalyzed side at 600 psig was similar for Types 2 and 6 sandwiches and no methyl alcohol was used in Type 2. At both 600 and 2000 psig the AP slope is so very steep that the flow of gases from the interfacial area is flowing nearly parallel to the AP surface. This possibly carries IO into the AP flame zone and retards the reaction rates. An indication of this is that no AP melt is observed and that the black spots are similar to black spots observed for AP during low temperature decomposition.

For IO catalyzed sandwiches it is concluded that:

1. The IO augments the AP deflagration process at 2000 psig, but inhibits it at 600 psig. This agrees with the results of Friedman, et al. [30]. The augmentation possibly results from acceleration of the AP flame gas phase reactions as suggested by Pittman [36] and/or by exothermic reactions of gases on the surface of catalyst particles.
imbedded in the AP taking place. The inhibition may possibly result from the sticky type melt having exothermic reactions of less intensity than a normal AP melt.

2. The IO does not promote heterogeneous reactions of gases with the solid fuel binder and does not modify the pyrolysis mechanism of the solid fuel binder.

3. The IO possibly promotes heterogeneous reactions by its presence in crevices between solid fuel and solid AP (at the interface).

4. The IO possibly catalyzes the base flame gas phase reactions, thus increasing the heat release rate near the sandwich interface.

**Summary of Catalyst Effects**

Table 2 contains a summary of the effects of CC and IO on AP/CIPB sandwiches at 600 and 2000 psig.

The results of this experiment differ from those achieved by Nadaud [24] and Pittman [34]. Nadaud found that CC increased the burn rate more for his sandwiches when present in the binder than when present in the AP or in both the AP and binder. This study indicates that exactly opposite results occur. The inconsistency could possibly result from the fact that Nadaud's samples were not bonded together, but only pressed together. Pittman used an iron-containing catalyst and determined that:

1. The effectiveness of mixing catalyst into the binder and of coating the interface between the AP and binder with catalyst is the same. This study indicated that the catalyst is much more effective at the interface.
Table 2. Summary of Catalyst Effects for AP/CTPB Sandwiches

<table>
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<tr>
<th>Type Sandwich</th>
<th>Pressure psig</th>
<th>Sandwich Burn Rate in/sec</th>
<th>AP Burn Rate in/sec</th>
<th>Flame Description</th>
<th>Binder Height inches</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure AP</td>
<td>600</td>
<td>----</td>
<td>0.23</td>
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<td>----</td>
</tr>
<tr>
<td>90 μ Type 1</td>
<td>600</td>
<td>0.26</td>
<td>0.25</td>
<td>Turbulent and Orange, Out Over AP</td>
<td>0.11</td>
</tr>
<tr>
<td>150 μ Type 2 CC</td>
<td>600</td>
<td>0.38</td>
<td>0.29</td>
<td>Violent, Purple Tint, Out Over AP</td>
<td>0.026</td>
</tr>
<tr>
<td>120 μ Type 4 CC</td>
<td>600</td>
<td>0.26</td>
<td>0.23</td>
<td>Smooth and Orange</td>
<td>0.012</td>
</tr>
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<td>120 μ Type 5 CC</td>
<td>600</td>
<td>0.38</td>
<td>0.28</td>
<td>Similar to Type 2</td>
<td>0.012</td>
</tr>
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<td>180 μ Type 6 CC</td>
<td>600</td>
<td>0.24</td>
<td>0.22</td>
<td>Purple Tints on Cat. Side</td>
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<td>----</td>
<td>0.46</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>60 μ Type 1</td>
<td>2000</td>
<td>0.46</td>
<td>0.46</td>
<td>Turbulent and Orange</td>
<td>Can't Determine</td>
</tr>
<tr>
<td>120 μ Type 2 CC</td>
<td>2000</td>
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<td>1.24</td>
<td>Laminar and Orange with Purple Tints</td>
<td>Can't Determine</td>
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<tr>
<td>90 μ Type 4 CC</td>
<td>2000</td>
<td>0.45</td>
<td>0.45</td>
<td>Smoother than Type 1, Purple Tints</td>
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<tr>
<td>100 μ Type 5 CC</td>
<td>2000</td>
<td>1.38</td>
<td>1.32</td>
<td>Similar to Type 2</td>
<td>Can't Determine</td>
</tr>
<tr>
<td>170 μ Type 6 CC</td>
<td>2000</td>
<td>0.74</td>
<td>0.47</td>
<td>Purple Tints on Cat. Side</td>
<td>Can't Determine</td>
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(Continued)
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<th>Type Sandwich</th>
<th>Pressure psig</th>
<th>Sandwich Burn Rate in/sec</th>
<th>AP Burn Rate in/sec</th>
<th>Flame Description</th>
<th>Binder Height inches</th>
</tr>
</thead>
<tbody>
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<td>300 μ Type 2 10</td>
<td>600</td>
<td>0.20</td>
<td>0.15</td>
<td>Smooth and Orange</td>
<td>0.028</td>
</tr>
<tr>
<td>140 μ Type 4 10</td>
<td>600</td>
<td>0.22</td>
<td>0.22</td>
<td>Smooth and Orange</td>
<td>0.011</td>
</tr>
<tr>
<td>180 μ Type 5 10</td>
<td>600</td>
<td>0.24</td>
<td>0.17</td>
<td>Similar to Type 2</td>
<td>Can't Determine</td>
</tr>
<tr>
<td>150 μ Type 6 10</td>
<td>600</td>
<td>0.36</td>
<td>0.12</td>
<td>Orange</td>
<td>Can't Determine</td>
</tr>
<tr>
<td>300 μ Type 2 10</td>
<td>2000</td>
<td>0.84</td>
<td>0.69</td>
<td>Smooth and Orange</td>
<td>0.016</td>
</tr>
<tr>
<td>130 μ Type 4 10</td>
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<td>0.48</td>
<td>0.48</td>
<td>Slightly Turbulent</td>
<td>Can't Determine</td>
</tr>
<tr>
<td>80 μ Type 5 10</td>
<td>2000</td>
<td>0.92</td>
<td>0.84</td>
<td>Similar to Type 2</td>
<td>None</td>
</tr>
<tr>
<td>120 μ Type 6 10</td>
<td>2000</td>
<td>0.60</td>
<td>0.20</td>
<td>Orange</td>
<td>Can't Determine</td>
</tr>
</tbody>
</table>
2. Catalyst mixed into AP does not increase the effectiveness of the catalyst compared with mixing it into the binder. This study indicates that the effectiveness is much greater when the catalyst is mixed into the AP, at least at high pressures. Pittman used three-dimensional strands as compared to the two-dimensional sandwiches of this study. Although he used CTPB/AP as in this study, his catalyst was liquid n-butyl (bis) cyclopentadienyl iron (BBCI), while this study used IO. Therefore it is questionable as to whether the results may be realistically compared.

These results, conclusions and noted inconsistencies with previous works indicate that the work of this study was truly exploratory and that additional work would be beneficial. The possible cases of interfacial regression can only realistically be evaluated through quench testing, as this will provide much more interfacial detail (much greater magnification). It is evident that high speed cinephotomacrography is a useful research tool for composite propellant combustion and that the two-dimensional sandwich model allows viewing of details which would be obscured in three-dimensional propellants.
CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

The high speed cinephotomacrography has been shown to be an effective tool for investigating the burning mechanisms of AP composite propellants. The results of the present investigation may be summarized by the following conclusions:

1. A flowable binder melt exists for PU, PBAA and CTPB during combustion. A similar melt may exist for PS, but evidence of the melt was not present in this investigation.

2. An AP surface melt is present for PS sandwiches at pressures where the pure AP burn rate is decreasing. This possibly explains why propellant burn rates are so much greater than AP burn rates at these pressures.

3. The assumed sandwich combustion model with an AP flame, a base flame, and a diffusion flame aided in sandwich deflagration interpretation.

4. Based on the motion picture study, there is no conclusive evidence that heterogeneous interfacial reactions occur for the non-catalyzed sandwiches. This is in agreement with the findings of Varney [13] and casts doubt on models employing this type reaction such as the model due to Hermance [14]. For the catalyzed sandwiches, leading edge regression was obtained in the area of the interface, but the resolution in this study was not great enough to determine if it was occurring
exactly at the interface.

5. The results of this investigation indicate that the sandwich model of Nachbar [20] is not sufficiently detailed to account for the actual physical processes which are occurring in the sandwich combustion.

6. A realistic model for binder/AP composite propellant combustion would consist of:
   a. Binder melt flows onto the AP surface.
   b. AP melts, even in the pressure range where the AP burn rate is decreasing with increasing pressure.
   c. Three basic flames: AP flame and base flame, both of which are elliptic in character, and a diffusion flame between the AP combustion products and the binder decomposition products which serves to decompose the binder.

7. Copper chromite catalyst augments the AP deflagration process at both 600 psig and 2000 psig, the augmentation increasing with pressure increase.

8. Copper chromite possibly catalyzes the AP flame and base flame gas phase reactions at both 600 psig and 2000 psig, increasing the heat release rate of the reactions.

9. Copper chromite catalyst possibly promotes gas phase reactions by its presence in crevices between solid fuel and solid AP (at the interface).

10. Copper chromite catalyst does not promote heterogeneous reactions of gases with the solid fuel binder and does not modify the pyrolysis mechanism of the solid fuel binder.
11. Iron oxide catalyst inhibits the AP deflagration process at 600 psig, but augments it at 2000 psig.

12. Iron oxide possibly catalyzes the base flame gas phase reactions at both 600 psig and 2000 psig, increasing the heat release rate of the reactions.

13. Iron oxide catalyst possibly promotes heterogeneous reactions by its presence in crevices between solid fuel and solid AP (at the interface).

14. Iron oxide catalyst does not promote heterogeneous reactions of gases with the solid fuel binder and does not modify the pyrolysis mechanism of the solid fuel binder.

15. The preceding conclusions concerning CC and IO catalysts indicate that some of the theories concerning the catalytic mechanisms of CC and IO with AP/binder composite propellants are justified whereas others are not. The theories of Levy and Friedman [32], Pearson [37], Pittman [36], and Steinz, Stang and Summerfield [12] concerning various gas phase mechanisms are shown to be feasible. The validity of the theories of Pearson [37] and Pearson and Sutton [38] concerning the solid fuel binder is shown to be doubtful for these catalysts.

16. Copper chromite is a more effective catalyst than iron oxide for AP/CTPB propellant systems.

17. With the exception of IO at low pressures (600 psig), the addition of CC and IO in the AP is the most effective method of increasing sandwich burn rate, their addition at the AP/binder interface is less effective and their addition in the binder does not appear to be very effective at all in increasing burn rates, at least not for the
sandwich configuration.

18. It is recommended that a detailed experimental program of composite propellant sandwiches using various catalysts, binders and oxidizers be initiated with use of both high speed cinephotomacrography and quench testing.
APPENDICES
APPENDIX A

SAMPLE FLOW RATE CALCULATION
FOR SHARP EDGED ORIFICE

A flow of inert gas is required past the sample during combustion in order to remove combustion products which would otherwise obscure the camera's view of the sample. A sample flow calculation is included here for nitrogen gas. Using this type calculation, if a particular nitrogen flow velocity past the sample, $V_0$, is desired, the required orifice $\Delta P$ to produce this effect may be calculated.

The nitrogen flow area past the sample is roughly equal to the cross-sectional area of the combustion vessel's flow passage minus the area taken up by the sample and ignition posts. The flow passage is one inch in diameter, the sample is approximately 0.18 inch deep by 0.1 inch thick and the two ignition posts are 3/16 inch in diameter. Thus the flow area is approximately 0.7 in$^2$. This does not account for the enlargement of the flow passage by the three windows, but assumes the vessel has a constant one inch diameter flow passage. The average temperature of the nitrogen gas is assumed to be $65^\circ F$ or $525^\circ R$ and the gas constant is assumed to be 55.2 ft lb$^_/m^/lb^_/R$. Knowing the desired combustion pressure, $P_0$, the density of the nitrogen may be calculated. The nitrogen mass flow rate may be calculated using the one-dimensional continuity equation.
This mass flow rate is also the mass flow rate passing through the orifice. The ΔP required across the orifice to produce this mass flow rate may be determined using Crane [46]. The net expansion factor is assumed to be 0.995 and the flow coefficient is assumed to be 0.595. The orifice diameter is 0.136 inches. Incorporating the above, the equation for the ΔP across the orifice is:

$$\Delta P = \frac{3.54 \times 10^{-3} \frac{P_2^2 V_2^2}{c' c}}{P_1}$$

where $P_1$ is the nitrogen supply pressure.
<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Pressure (psi)</th>
<th>Temperature (°C)</th>
<th>Pressure (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>200</td>
<td>1.5</td>
<td>200</td>
</tr>
<tr>
<td>1.0</td>
<td>200</td>
<td>2.0</td>
<td>200</td>
</tr>
<tr>
<td>2.0</td>
<td>200</td>
<td>3.0</td>
<td>200</td>
</tr>
<tr>
<td>3.0</td>
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<td>200</td>
</tr>
<tr>
<td>4.0</td>
<td>200</td>
<td>5.0</td>
<td>200</td>
</tr>
<tr>
<td>5.0</td>
<td>200</td>
<td>6.0</td>
<td>200</td>
</tr>
<tr>
<td>6.0</td>
<td>200</td>
<td>7.0</td>
<td>200</td>
</tr>
</tbody>
</table>

*Note: The data represents average pressure readings at different temperatures.*

Constant Flow Rate Velocity Test Sample

Table A-1. Off-take AP Table for One Foot Per Second

133
Table A-1. (Continued)

<table>
<thead>
<tr>
<th>$P_c = 1500$ psig</th>
<th>$P_c = 2000$ psig</th>
<th>$P_c = 2400$ psig</th>
<th>$P_c = 2800$ psig</th>
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<tr>
<td>Avg. $P_1$</td>
<td>$\Delta P$</td>
<td>Avg. $P_1$</td>
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</tr>
<tr>
<td>(psig)</td>
<td>(psid)</td>
<td>(psig)</td>
<td>(psid)</td>
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<tr>
<td>1500</td>
<td>5.4</td>
<td>2200</td>
<td>3.7</td>
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<tr>
<td>1600</td>
<td>5.0</td>
<td>2300</td>
<td>3.5</td>
</tr>
<tr>
<td>1700</td>
<td>4.7</td>
<td>2400</td>
<td>3.4</td>
</tr>
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<td>1800</td>
<td>4.5</td>
<td>2500</td>
<td>3.2</td>
</tr>
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<td>4.3</td>
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</tr>
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<td>4.0</td>
<td>2700</td>
<td>3.0</td>
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<tr>
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<td>3.9</td>
<td>2800</td>
<td>2.9</td>
</tr>
<tr>
<td>2000</td>
<td>7.1</td>
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<td>5.1</td>
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<td>8.5</td>
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<tr>
<td>3000</td>
<td>9.3</td>
<td>3300</td>
<td>8.5</td>
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Table A-1. (Continued)

<table>
<thead>
<tr>
<th>Avg. $P_1$ (psig)</th>
<th>$\Delta P$ (psid)</th>
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</thead>
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<tr>
<td>3200</td>
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</tr>
<tr>
<td>3300</td>
<td>11.0</td>
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</tbody>
</table>

$P_c = 3200$ psig

<table>
<thead>
<tr>
<th>Avg. $P_1$ (psig)</th>
<th>$\Delta P$ (psid)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3400</td>
<td>10.7</td>
</tr>
<tr>
<td>3500</td>
<td>10.4</td>
</tr>
</tbody>
</table>
APPENDIX B

COMBUSTION VESSEL CENTER SECTION DRAWINGS

Drawings for the combustion vessel center section are shown in Figures B-1 through B-5.
Figure B-1. Center Section Assembly.

NOTES:
1. LUBRICATE ITEMS 2 AND 3 WITH ITEM 9 PRIOR TO ASSEMBLY.
Figure B-2. Quartz Window Retainer.
NOTES:
1. MATERIAL - TYPE 304 STAINLESS STEEL.
2. ALL DIMENSIONS IN INCHES.
3. TOLERANCES - UNLESS OTHERWISE SPECIFIED
   DECIMALS - ± .001 INCHES
   ANGLES - ± 0°3'
   SURFACE FINISH - ± 0.25
4. DO NOT SCALE THIS DRAWING.
5. PART SHALL BE FREE OF BURRS.

FiguRE B-3. Quartz Window Retainer.
Figure B-4. Center Section Detail.
NOTES:

1. MATERIAL - CLEAR FUSED QUARTZ, GENERAL ELECTRIC, TYPE 151, GROUND AND POLISHED.
2. ALL DIMENSIONS IN INCHES.
Table C-1 contains the Hycam camera settings required for the pictures per second desired. Figure C-1 contains the feet of film for the Hycam camera to stabilize at a particular pictures per second setting. This information is from the Hycam operating instructions [49].
### Table C-1. Hycam K20S4E Speed Setting Chart

<table>
<thead>
<tr>
<th>Pictures Per Second</th>
<th>Reel Size</th>
<th>Speed Multiplier</th>
<th>Servo Brake Tension</th>
<th>Follow Arm Compensator Brake</th>
<th>Drag Brake</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>100</td>
<td>L</td>
<td>X1</td>
<td>L</td>
<td>Extended</td>
</tr>
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<td>X1</td>
<td>L</td>
<td>Extended</td>
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<td>100</td>
<td>400</td>
<td>L</td>
<td>X1</td>
<td>L</td>
<td>Centered</td>
</tr>
<tr>
<td>to 200</td>
<td>200</td>
<td>H</td>
<td>X10</td>
<td>L</td>
<td>Extended</td>
</tr>
<tr>
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<td>400</td>
<td>H</td>
<td>X10</td>
<td>L</td>
<td>Centered</td>
</tr>
<tr>
<td>to 200</td>
<td>200</td>
<td>H</td>
<td>X100</td>
<td>L</td>
<td>Extended</td>
</tr>
<tr>
<td>2000</td>
<td>400</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Extended</td>
</tr>
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<td>to 400</td>
<td>200</td>
<td>H</td>
<td>X100</td>
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<td>400</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Extended</td>
</tr>
<tr>
<td>to 5000</td>
<td>200</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Centered</td>
</tr>
<tr>
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<td>400</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Extended</td>
</tr>
<tr>
<td>to 8000</td>
<td>200</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Centered</td>
</tr>
<tr>
<td>8000</td>
<td>400</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Extended</td>
</tr>
<tr>
<td>to 11000</td>
<td>400</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Centered</td>
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<tr>
<td></td>
<td>400Z</td>
<td>H</td>
<td>X100</td>
<td>H</td>
<td>Retracted</td>
</tr>
</tbody>
</table>

(3" Core)

*H = High Range, which is equal to No Speed Reducer.

**Start-Stop operation not possible at this frame rate.
Figure C-1. Hycam Speed Curves.
APPENDIX D

ESTIMATION OF ENERGY TRANSFERRED
TO SAMPLE BY XENON LAMP

The xenon lamp was operated at its minimum power setting of 65 amps for all runs. The operating voltage of the lamp is

\[ U_L = U_G + I_L \times R_L \]

where \( R_L \) is 0.11 ohms and the average \( U_G \) is 22.5 volts as stated in [50]. For \( I_L \) of 65 amps, \( U_L \) is 29.65 volts and the power developed is 1930 watts. Reference [51] states that 39 per cent of the power of the xenon lamp is converted into arc radiation. Thus if we assume that all of the arc radiation focuses down into a one inch diameter beam (actually the lamp will not focus down to the one inch diameter size of the window) the energy into the vessel is 35.9 cal/cm² sec.

Since there is an infrared absorbing filter over the window, not all of the energy into the window will get through to the interior of the vessel. Figure D-1 shows the per cent transmittance versus wavelength for the Corning 1-75 filter used. Based on this filter the following assumptions are made:

1. Approximately 85 per cent of the visible radiation is transmitted (400 to 700 nanometers wavelength).

2. Approximately 5 per cent of the infrared radiation is transmitted (800 to 1000 nanometers wavelength, which is the principal band of infrared radiation from the xenon lamp, Figure D-2).
Figure D-1. Corning 1-75 Infrared Absorbing, Visible Transmitting Filter.
Figure D-2. Distribution of Relative Spectral Radiance of 2500W Xenon Lamp.
Reference [51] states that the visible and ultraviolet radiation account for 17 per cent of the arc radiation. This is 43.6 per cent of the energy which enters the filter. The infrared accounts for 22 per cent of the arc radiation, which is 56.4 per cent of the energy which enters the filter. Now the amount of energy into the window which is actually passed through to the interior of the vessel is 13.3 cal/cm$^2$/sec from visible + ultraviolet radiation and 1 cal/cm$^2$/sec from infrared radiation. Thus 14.3 cal/cm$^2$/sec is the amount of energy which might be deposited on the sample. This assumes clean windows, no loss due to the tunnel between the window and the sample and that all radiation comes out of the lamp and goes into the one inch diameter window.

The total energy release for pure AP is approximately 330 cal/gm. A typical sandwich weighs approximately 0.25 grams, thus approximately 62.5 calories of heat is released for a piece of pure AP the size of our sandwiches during combustion. A sandwich would release more heat than the pure AP, thus the 62.5 calories is a lower limit for heat release.

If it is assumed that the lamp is focused on the sample for two seconds plus the time required for the sample to burn down at 0.7 cm/sec and that all the energy entering the vessel goes into the side of the sample (area of 0.5 cm$^2$) then the energy transferred to the sample by the xenon lamp is 19.4 cal. Thus the energy provided by the lamp is very roughly 30 per cent of the energy supplied by the combustion process.
This is a rough calculation and is very conservative based on the previously noted assumptions plus the fact that the distance from the lamp to the sample is not considered and the irradiance will vary as the inverse square of that distance.
APPENDIX E

RUN PROCEDURES

Pre-Run Procedure

1. Fill out Run Data Sheet.
2. Mount sandwich and position ignition wire.
3. Attach sample holder assembly to combustion vessel.
4. Assemble combustion vessel and tighten all connections.
5. Ensure that proper lens extension is being used for magnification desired.
6. Set camera lens at ∞ and f stop to be used during run.
7. Remove lens cap.
8. Replace camera’s lower film gate with the red ground glass focusing gate.
9. Rotate camera shutter until full frame is visible.
10. Focus eyepiece on cross-hair visible on the ground glass.
11. Provide illumination source for sample.
12. Slide camera on the lathe bed toward sample until rough sample image appears on ground glass.
13. Secure camera to lathe bed.
14. Focus camera on sample.
15. Close off camera eyepiece.
16. Remove sample illumination source.
17. Ensure switch on camera is in OFF position and that remote
switch is in OFF position.

18. Load film into camera; ensure that ON-OFF micro switch behind film is depressed and that perforations in film engage sprocket properly.

19. Adjust event micro switch to desired setting for ignition.

20. Check that ignition wires, power cord, remote cord and timing light cords are connected properly.

21. Place remote switch where it can be utilized during run.


23. Ensure that xenon douser is closed and that pulley cords are free to open and close douser.

24. Close safety vent valve (MOV-2), flow metering valves, and system supply valve (MOV-1).

25. Ensure that pressure regulator control is backed-off completely.

**Run Procedure**

1. Complete Pre-Run Procedure.

2. Put on hard hat and safety glasses.

3. Turn on timing light to desired cps and check with counter.

4. Enter timing light cps as read from counter on Run Data Sheet.

5. Turn on xenon lamp exhauster and xenon lamp power supply. Strike xenon lamp and adjust to desired amp setting.
6. Turn on ignition power supply and adjust variac to give 36 volts DC unloaded.

7. Open nitrogen cylinder valve(s).

8. Open manifold valve(s).

9. Note pressure indicated on supply pressure gauge and enter this pressure on Run Data Sheet.

10. Open system supply valve (MOV-1) slowly.

11. Adjust the pressure regulator control to give 300 psig in combustion vessel.

12. Leak check all connections which were tightened in Pre-Run Procedure.

13. If a leak is apparent, proceed to Step 20, complete Run Procedure from that step on and then repair leak. If no leak is apparent, proceed with Step 14.

14. Adjust pressure regulator control to the desired combustion pressure.

15. Open flow metering valve(s) and adjust orifice ΔP for flow desired.

16. Open xenon douser, then immediately switch camera ON.

17. After sufficient run time (approximately 2-3 seconds) switch camera OFF and close xenon douser.

18. Close flow metering valve(s).

19. Note pressure indicated on Supply Pressure Gauge and enter this pressure on Run Data Sheet.

20. Close nitrogen cylinder valve(s).
21. Open flow metering valve and bleed all pressure from system.
22. Close flow metering valve.
23. Close system supply valve (MOV-1).
24. Close manifold valve(s).
26. Enter timing light cps as read from counter on Run Data Sheet.
27. Turn off ignition power supply, timing light generator and xenon power supply.
28. Leave xenon exhauster running for approximately 10 minutes and then turn off.
29. Loosen camera and slide back away from combustion vessel.
30. Remove film from camera.
31. Place lens cap on lens.
32. Open combustion vessel.
<table>
<thead>
<tr>
<th>Run Number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Binder Thick.--microns</td>
<td>FU</td>
<td>PU</td>
<td>PU</td>
<td>CTPB</td>
<td>PBAA</td>
<td>PS</td>
<td>PS</td>
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<tr>
<td>Sample C/S--inches</td>
<td>--</td>
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<td>--</td>
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<tr>
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<td>1000</td>
<td>1000</td>
<td>1000</td>
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APPENDIX F

EMMONS PROBLEM APPLIED
TO SANDWICH CONFIGURATION

The Emmons Problem [46] is composed of a stationary semi-infinite flat plate, consisting of a solid or liquid fuel, placed parallel to a gaseous oxidizing stream with a uniform approach velocity $V_o$. Gaseous fuel pyrolyzes, sublimes or vaporizes from the plate and reacts with the oxidizer in the boundary layer. The products of the reaction are diffused and convected away. This application makes use of the Emmons Problem as outlined by Williams [52].

Williams presents the dimensionless burning rate for the Emmons Problem in graphical form. Entering the graph with the value calculated for \( \left( \frac{\alpha_{T,\infty}}{\alpha_{T,s}} - \alpha_{O,\infty} \right) / \hat{q} \) and obtaining the dimensionless burning rate, the theoretical total mass flow rate of the fuel leaving the surface may be calculated in terms of the flat plate length (binder height) \( l \). An experimental total mass flow rate can be obtained from the motion pictures. Equating the two total mass flow rates, the theoretical binder height can be obtained.

If we let the dimensionless burning rate of the graph be represented by \( B \) and assume that the product $\rho u$ is constant, the mass flow rate of fuel per unit area, $u_s$, is

\[
 u_s = \frac{B}{2} \left( \frac{\rho_s u_s V_{in}}{x} \right)^{\frac{1}{2}}
\]
Integrating this over the surface of the fuel layer, the theoretical total mass flow rate, \( \hat{\omega}_s \), is obtained.

\[
\hat{\omega}_s = B (\rho_s \mu_s V_s) \frac{1}{s}
\]

In order to initially obtain \( B \), the following parameters must be calculated:

\[
\alpha_{T,\infty} = \int_{T_\infty}^{T_\infty} \frac{C_p}{Q_{DF}} \, dT
\]

Thermal Enthalpy Per Unit Mass Of The Gas In The Free Stream

Heat Liberated In The Diffusion Flame Reaction

\[
\alpha_{T,s} = \int_{T_s}^{T_s} \frac{C_p}{Q_{DF}} \, dT
\]

Thermal Enthalpy Per Unit Mass Of The Gas At The Binder Surface

Heat Liberated In The Diffusion Flame Reaction

\[
\alpha_{\varphi,\infty} = \frac{\varphi_0}{\varphi_0 (\nu'' - \nu')}
\]

Denotes Chemical Energy Available For Diffusion Flame Reaction From Gas In The Free Stream

\[
\hat{Q} = \frac{L + q}{Q_{DF}}
\]

Heat Conducted Into The Surface Heat Liberated In The Diffusion Flame Reaction

For convenience \( T^0 \) is set equal to the binder surface temperature, \( T_s \).

A sample calculation for a 100 micron binder layer PS sandwich at 1000 psig appears below (test 51). Figure F-1 shows the sandwich configuration used.

For the diffusion flame reaction AP combustion products are the oxidizer and binder decomposition products are the fuel. The
Figure F-1. Half-Sandwich Configuration for Emmons Problem.
following reaction was used for the calculations (AF combustion products from Levy and Friedman [32]):

\[
0.265 \, N_2 + 0.12 \, N_2O + 0.23 \, NO + 1.015 \, O_2 + 1.62 \, H_2O + 0.76 \, HCl
\]
\[
+ 0.12 \, Cl_2 + 0.239 \, C_3.141H_6.58^1\,O_0.854^2\,O.869 = 0.75 \, CO_2 + 2.286 \, H_2O
\]
\[
+ HCl + 0.5 \, N_2 + 0.208 \, SO_2
\]

Using the above reaction and calculating the Heat of Reaction (used -69.5 kcal/mole for heat of formation of PS [53]), \( Q_{DF} = 120 \, \text{kcal} \).

Assuming \( C_p = 0.3 \, \text{cal/gm}^\text{o K} \), \( T^\circ = T^\circ = 641^\circ \text{K} \) [13] and \( T^\infty = 1400^\circ \text{K} \) [17], \( \mathcal{X}_{T^\infty} \) is 1.9 x 10^{-3}/gm and \( \mathcal{X}_{T^\circ} \) is zero. Assuming that the oxidizing species available for the diffusion flame reaction are \( N_2O, NO, O_2 \) and \( Cl_2 \), \( \mathcal{X}_{O^\circ} \) is -8.52 x 10^{-3}/gm.

The heat conducted into the binder, \( q \), is required to be in cal/gm for consistency of units. These units may be obtained using a rough order of magnitude analysis, the accuracy of which is later verified by the results of the calculation for PS. For \( q \) in the above units,

\[
q = \frac{\dot{q}_h}{m_0}
\]

where

\[
\dot{q} = \frac{\lambda(T^\circ - T^\circ)}{S}
\]
and using conservation of mass (inflow of binder = outflow of binder),

\[ m_b = \rho_b \delta r = \rho_b \ell b \]

or

\[ \dot{m}_b = \rho_b v_b = \frac{\rho_b \delta r}{\ell} \]

Thus the equation for \( q \) becomes:

\[ q = \frac{\lambda \ell (T_S - T_i)}{\rho_b \delta^2 r} \]

Assuming \( \lambda = 5 \times 10^{-4} \text{ cal/cm} \cdot \text{sec} \cdot \text{°K} \) [17], \( \rho_b = 1.3 \text{ gm/cm}^3 \) [12], \( T_i = 65 \text{ °F} = 292 \text{ °K} \), \( \ell = 0.0533 \text{ cm} \) (from the motion pictures), \( r = 0.71 \text{ cm/sec} \) (from the motion pictures) and \( \delta = 0.5 \times 10^{-2} \text{ cm} \) (from previously stated binder thickness) \( q \) is calculated to be 402 cal/gm. From Varney's DSC results [13], \( L = 66 \text{ cal/gm} \) for PS, thus \( \dot{q} \) is calculated to be \( 3.9 \times 10^{-3} \text{ cal/gm} \). Using the previously calculated values, \( \alpha_{T_i} - \alpha_{T_S} - \alpha_{0,\theta}/\dot{q} \) becomes 2.62 and from the graph in Williams, \( B \) is 0.64.

Using conservation of mass the free stream velocity, \( V_\infty \), is calculated to be 77 cm/sec.

Now assuming that \( \sigma_{\mu_S} = \sigma_{\mu_\infty} = \text{constant} \) and \( \mu_\infty = 9.25 \times 10^{-1} \text{ gm/cm} \cdot \text{sec} \), the theoretical binder total mass flow is calculated to be \( \dot{w}_b (\text{Theoretical}) = 1.77 \times 10^{-2} \ell \frac{1}{\dot{q}} \). The experimental binder total
mass flow may be calculated using:

\[ \hat{\omega}_s (\text{Experimental}) = \rho_b A_b r \]

and with \( A_b = 5 \times 10^{-3} \text{ cm}^2 \) and \( r = 0.71 \text{ cm/sec} \), obtained from the motion pictures, \( \hat{\omega}_s (\text{Experimental}) = 4.62 \times 10^{-3} \text{ gm/sec} \). Equating the two total mass flows and solving for the theoretical binder height, \( t(\text{Theoretical}) = 6.82 \times 10^{-2} \text{ cm} \). This calculated result is in good agreement with the experimental binder height, \( t(\text{Experimental}) = 5.33 \times 10^{-2} \text{ cm} \).

The Reynolds number for this example, based on \( \delta \), is calculated to be approximately 15. For the boundary layer approximation Reynolds number should be much greater than one. Therefore this Reynolds number is rather low. However, for binder heights larger than \( \delta \) the Reynolds number will be larger and the boundary layer approximation will become better. The comparison of theoretical and experimental values shows that the approximation has some merit.
BIBLIOGRAPHY


VITA

Howard Evans Jones was born in Brownfield, Texas, on April 27, 1939. He was the first child of Mr. and Mrs. Paul H. Jones. Two sisters, Mrs. Kay Gene Payne and Mrs. Marianne Hart, completed the family. Elementary schools were attended in Seminole, Texas, and Post, Texas. Mr. Jones graduated from Post High School in May, 1957, as Valedictorian of his class. He entered Texas Technological College in Lubbock, Texas, in September, 1957, and received a Bachelor of Science in Mechanical Engineering in August, 1961. He was a member of Pi Kappa Alpha social fraternity while at Texas Tech.

General Dynamics Astronautics at Warren Air Force Base in Cheyenne, Wyoming, provided his first experience as an engineer. He worked with pneumatic and hydraulic systems associated with the Atlas Type E Missile System. Six months after joining General Dynamics Astronautics Mr. Jones resigned and accepted employment with Pratt and Whitney Aircraft at their Florida Research and Development Center in West Palm Beach, Florida, as an Experimental Engineer. While employed by Pratt and Whitney, Mr. Jones obtained a Master of Engineering in Mechanical Engineering from the University of Florida in August, 1965. This was obtained through a company sponsored program. He successfully completed the requirements and became a Registered Professional Engineer in the State of Florida in 1966.

In June, 1968, Mr. Jones received a corporation authorized leave-of-absence from Pratt and Whitney and entered the Doctoral Program in
Aerospace Engineering at the Georgia Institute of Technology. He was awarded an NDEA Title IV Fellowship and subsequently an NSF Fellowship. He was a student member of the American Institute of Aeronautics and Astronautics and was active in Student Government. He was a Representative to the Graduate Student Senate, Treasurer of the Graduate Student Senate, served on the Governing Board of the Student Center, and served as a member of the Graduate Council Curriculum Committee. In the fall of 1971, he was selected to membership in Who's Who Among Students in American Colleges and Universities.

On October 1, 1965, in Conway, South Carolina, Mr. Jones married the former Mary Elizabeth (Becky) Arrott of Jacksonville, North Carolina. They have a girl and boy, Tracy Elizabeth and Philip Howard.